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# **Advanced Chemistry**

## **Teacher Guide**

**PASCO®**

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# Contributors

## **PASCO Development Team**

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- ◆ Freda Husic, *Director of Education Solutions, Program Manager*
- ◆ Sandor Kadar, Ph.D., *Lead Author, Associate Professor, Chemistry, Salve Regina University*

## **Contributing Authors**

---

- ◆ Bill Kurnett, *High School Chemistry and AP Chemistry Teacher*
- ◆ Jessica Odobasic, *Student Associate, Salve Regina University*
- ◆ Amy Beltramini, *Student Associate, Salve Regina University*

## **Editors and Graphic Designers**

---

- ◆ Janet Miller, *Lead Editor*
- ◆ Marty Blaker, *Editor*
- ◆ Nancy Clarke, *Editor*
- ◆ Jim Collins, *Editor*
- ◆ Chuck Jaffee, *Editor*
- ◆ John Terschak, *Science Content Reviewer*
- ◆ Bill Kurnett, *Science Content Reviewer*

## **PASCO Production Team**

---

- ◆ Dan Kimberling, *Media Specialist*
- ◆ Susan Watson, *Production Specialist*

## **Student Contributors**

---

- ◆ Jeremy Osborne, *Salve Regina University*
- ◆ Mandy Letourneau, *Salve Regina University*
- ◆ Jeff DiLorenzo, *Salve Regina University*

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# Advanced Chemistry

*Experiments for AP\*, IB\*\*, and Honors Chemistry*

*Teacher Guide*

*21<sup>st</sup> Century Science*

**PASCO scientific**

10101 Foothills Blvd.  
Roseville, CA 95747-7100  
Toll Free 800-772-8700  
916-786-3800  
Fax 916-786-8905

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## Introduction

PASCO's 21st Century Science Guides focus on science through inquiry. Learning science through inquiry-targeted activities replaces the tedious and passive cookbook approach to conducting science investigations with the thrill of discovery. The science standards are perfectly clear about the significance of student inquiry in teaching. The National Science Education Standards (NSES) position student inquiry at the forefront:

Inquiry is central to science learning. When engaging in inquiry, students formulate questions and devise ways to answer them, they collect data and decide how to represent it, they organize data to generate knowledge, and they test the reliability of the knowledge they have generated. They identify their assumptions, use critical and logical thinking, and consider alternative explanations. In this way, students actively develop their understanding of science by combining scientific knowledge with reasoning and thinking skills. (NSES, 1997)

In traditional investigations, students spend most of their time carrying out low-level tasks—following procedures and collecting data. They often completely miss the purpose of an investigation, how it is designed, and how the data are related to the underlying science. By seeing a graph that summarizes the data during an investigation, students can not only learn about that particular graphical representation, but they can also pair that representation in real time with the investigation's context, inspiring questions that analyze the experimental design and results. This experience has been shown by researchers to produce dramatic increases in motivation and in understanding.

The lab activities in PASCO's 21st Century Science Guides are scaffolded so that students complete investigations successfully, learning the process of scientific inquiry as they progress, and ultimately are able to design and conduct extended inquiry investigations. In the course of conducting these activities, students develop skills that include critical thinking (posing good questions, developing experimental strategies, finding and fixing operational errors), procedural expertise (calibrating equipment, collecting data), proficiency in design and construction (assembling apparatus, safety procedures, mixing solutions), and analytical skills (graphing, modeling, statistics). Incorporating science through inquiry requires both a change in the overall science curriculum and the introduction of new tools and materials that address these skills.

Information and computer tools are essential to inquiry-targeted lab activities. The use of sensors, data analysis and graphing tools, models and simulations, and work with instruments, all support scientific inquiry and are explicitly cited in the science standards. The lab activities in PASCO's 21st Century Science Guides provide students with hands-on and minds-on learning experiences, making it possible for them to master both the scientific inquiry process and the tools that prepare them to conduct extended scientific investigations.

### **About the PASCO 21st Century Science Advanced Chemistry Guide**

AP Chemistry is a difficult course requiring students to master both lecture and laboratory material. The PASCO Advanced Chemistry Guide helps students make connections between what happens in the laboratory and the material in the lecture. The laboratory activities go beyond a structured inquiry approach and use guiding and linking questions embedded within the procedure, as well as thought-provoking analysis questions, to achieve this goal. Students discover relationships between variable quantities or make generalizations regarding data collected, which lead to the discovery of expected outcomes.

This approach teaches students the basics of conducting investigations as well as techniques for using equipment and procedures that can be used in extended investigations. To provide other

## Introduction

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inquiry possibilities for students' investigations see the suggestions in *Using these Labs with the AP and the IBO Programs* in this Introduction.

Additionally, this manual presents teacher-developed laboratory activities using 21<sup>st</sup>-century technologies to help you and your students explore topics, develop scientific inquiry skills, and prepare for state level standardized exams. Using electronic-sensor data collection, display, and analysis devices in your classroom provide several benefits. With this equipment, students

- ◆ observe phenomena that occur too quickly or are too small, occur over too long a time span, or are beyond the range of observation by unaided human senses
- ◆ repeat measurements using equipment that can be used repeatedly over the years
- ◆ collect accurate data with accurate time and/or location stamps
- ◆ rapidly collect, graphically display, and analyze data so classroom time is effectively used
- ◆ practice using equipment and interpreting data produced by equipment that is similar to the tools they might use in their adult careers and personal activities

## Conducting Successful Inquiry-Based Lab Activities

### 1. Establish the Foundation

Preparing students to conduct their own scientific inquiry activities takes time and intention. Students need a foundation in conceptual development, laboratory bench skills, using electronic data collection and display equipment, and interpreting data. The following strategies help students build the foundation for inquiry-based learning:

- ◆ Work with students to complete tutorials for equipment and software they will be using.
- ◆ Start with easy activities that use only one sensor and do not require sensor calibration.
- ◆ Demonstrate the first few activities so you model the correct use of equipment and materials.
- ◆ Work with students to complete all sections of several lab activities until they understand your expectations.
- ◆ Foster cooperative learning. Students have different skills and interests to bring into play when they cooperate as a team. Reward students' cooperative efforts.
- ◆ Create teams with defined responsibilities for members. Devise a method to track the roles each student has carried out. Make sure each student has multiple opportunities to perform each role.
  - ◆ A team leader could be responsible for the overall conduct and verbal reporting and can make sure the team has all the necessary materials and equipment.
  - ◆ A team recorder could be responsible for documenting the completion of the activity and making sure all questions are answered in writing.
  - ◆ Team technicians could be responsible for operating the electronic and laboratory equipment.
- ◆ Create opportunities for students to repeat activities that seemed beyond their grasp the first time through—perhaps with student-suggested modifications. You will see substantial

improvement as students are given increased opportunities to work with the equipment and analyze the data.

## 2. Foster Inquiry Skills

Foster the growth and development of inquiry skills. Provide multiple opportunities for students to work with the equipment, analyze data, and communicate and discuss conclusions. The following strategies support development of laboratory and data analysis skills:

- ◆ Model the more complex technical tasks, such as sensor calibration and mathematical computations.
- ◆ Provide multiple and varied opportunities for practice with hands-on activities using 21<sup>st</sup>-century tools.
- ◆ Compile and compare class data whenever possible. Discuss the sources of variation in data and the best interpretation of the data.
- ◆ Have students brainstorm related questions they would like to explore in their own investigations.

The following table lists the foundational chemistry topics and the laboratory activities in this manual that support mastery of each topic. (The activities in the Advanced Chemistry guide are numbered according to the College Board list of recommended experiments.)

*Advanced Chemistry activities by topic area*

<b>Foundational Chemistry Topics</b>	<b>Activities</b>
Introduction to PASCO Data Collection Systems	Scientific Inquiry – DataStudio <sup>®</sup> Scientific Inquiry – SPARK Science Learning Center <sup>™</sup> Scientific Inquiry – SPARKvue <sup>™</sup> Scientific Inquiry – Xplorer GLX <sup>™</sup>
Energy in Chemical Systems	Lab 13: Enthalpy of a Chemical Reaction
Acid–Base Chemistry	Lab 6: Standardizing a Solution of Sodium Hydroxide Lab 7: Acid–Base Titration Lab 11: Using Different Indicators for pH Determination Lab 19: Properties of Buffer Solutions Lab 24: Determining $K_a$ by Half-Titration of a Weak Acid Lab 30: Determination of the $K_a$ Values of Two Isomeric Multi-Protic Acids
Gas Laws	Lab 5: Molar Volume of a Gas Lab 29: Exploring Gas Laws
Interactions Between Molecules	Lab 3: Determine the Molar Mass of a Volatile Liquid Lab 4: Molecular Weight by Freezing Point Depression Lab 18: Separation by Liquid Chromatography Lab 28: Molecular Interaction in Ethanol and Acetone

## Introduction

Foundational Chemistry Topics	Activities
Kinetics and Equilibrium	Lab 10: Determine the Equilibrium Constant for a Chemical Reaction Lab 12: Determination of the Rate of the Decomposition of Hydrogen Peroxide Lab 23: Determination of a Solubility Product Lab 25: Order of Reaction Lab 31: Determining the Half-Life of an Isotope
Chemical Reactions	Lab 1: Determining the Empirical Formula of a Compound Lab 2: Determine the Percentage of Water in a Hydrate Lab 8: Oxidation–Reduction Titration Lab 9: Mole Relationships in a Chemical Reaction Lab 14a: Separation and Analysis of Cations Lab 14b: Analysis of Anions Lab 15a: Synthesis of a Coordination Compound Lab 15b: Analysis of a Coordination Compound Lab 16: Gravimetric Determination of a Precipitate Lab 20: Determination of Electrochemical Series Lab 21: Electroplating Lab 22a: Organic Synthesis I—Preparation Lab 22b: Organic Synthesis II—Analysis Lab 26: Conductometric Titration Lab 27: Identifying an Unknown Metal Lab 32: The Breathalyzer™ Test for Alcohol
Light and Matter	Lab 17a: Absorption Spectra Lab 17b: Colorimetric Analysis

### 3. Cultivate Student-Directed Inquiry

Although most of the College Board recommended laboratory activities are fairly scripted, opportunities to let students “think like scientists” can be created. After allowing students to perform the recommended labs, guide students through studies of their own design using the extended inquiry examples as your guide. Watch for those moments of inquisitiveness and interest; provide students with opportunities and guidance for student-directed inquiry. The following strategies support and guide student-directed inquiry:

- ◆ Require a written plan with procedures.
- ◆ Provide plenty of time, material, and equipment resources.
- ◆ Make sure students understand any safety precautions necessary for their procedures.
- ◆ Incorporate check points to assess progress.
- ◆ To guide the students ask such questions as: How does the sample rate impact the outcome? What do you expect to happen? What are possible sources of error? Can any of these be eliminated in the procedure?

- ◆ Students could generate their own questions. Facilitate a brainstorming session or class discussion for students to answer the questions.

#### **4. Communicate the Results of Student-Directed Inquiry**

Provide opportunities for students to communicate the results of student-directed inquiry. Strategies include:

- ◆ Formal research papers, PowerPoint® presentations, video productions, and poster presentations are ways for students to share what science they have learned.
- ◆ Student-directed inquiries related to community resources may be of interest to area news or conservation groups. Have students report on their findings in a community venue, the school website, local newspapers or other publications.

#### **The Data Collection System**

"Data collection system" refers to the data collection, display, and analysis device used to carry out the various PASCO 21st Century Science Guide activities. These include PASCO's DataStudio®, the Xplorer GLX™, SPARKvue™, and SPARK Science Learning System™.

Activities are designed so that any PASCO data collection system can be used to carry out the procedure. The appendix corresponding to the DataStudio, Xplorer GLX, SPARKvue, or SPARK Tech Tips provide the steps on how to use the data collection system (see the Using Your Data Collection System section).

#### **Getting Started**

To help you and your students become familiar with the many features of your data collection system, start with the tutorials that are available for your system. Tutorials for the SPARK Science Learning System, SPARKvue, Xplorer GLX, and DataStudio are available on the CD that comes with the equipment or you can view the tutorials on PASCO's website.

#### **Scientific Inquiry Lab Activity**

The Scientific Inquiry activity, the first in the lab guide and the first activity students should perform, serves two purposes. It introduces students to scientific inquiry, that is, the process of conducting science investigations (the scientific method). Also, each data collection system has its own custom Scientific Inquiry activity so that teachers and students are introduced easily and quickly to the commonly used features of the device. *Start with this activity to become familiar with the data collection system.*

#### **Teacher and Student Guide Contents**

All the teacher and student materials are included on the CD accompanying the Teacher Guide, but the printed version of the Student Guide: Master Handouts must be obtained separately.

#### **Lab Activity Components**

Each activity has three components: Teacher Information, Student Inquiry Worksheets, and Student Worksheets.

## Introduction

*Teacher Information* is in the Teacher Guide binder. It contains the information on selecting, planning, and implementing the lab, as well as the complete student version with answer keys. Teacher Information includes all sections of a lab activity, including objectives, procedural overview, time requirements, and materials and equipment at-a-glance.

*Student Inquiry Worksheets* begin with a driving question, providing students with a consistent scientific format that starts with formulating a question to be answered in the process of conducting a scientific investigation. Inquiry worksheets include 1) the equipment and materials list, 2) safety precautions, 3) a sequencing challenge, 4) procedures for preparing the data collection system and sensors, setting up equipment, collecting and recording data, and analyzing results, and 5) analysis, synthesis, and multiple choice questions.

*Student Worksheets* are a shortened, traditional version of the student lab, containing the driving question, background, procedural instructions of the activity, and analysis questions. It is included only in electronic format on the CD that accompanies this manual.

This table identifies the sections you will find in each of these three activity components.

TEACHER INFORMATION	STUDENT INQUIRY WORKSHEET	STUDENT WORKSHEET
Objectives	Driving Questions	Driving Questions
Procedural Overview	Background (student version)	Background (Student version)
Time Requirement		
Materials and Equipment	Materials and Equipment	Materials and Equipment
Concepts Students Should Already Know		
Related Labs in This Guide		
Using Your Data Collection System		
Background		
Pre-Lab Discussion and Activity		
Lab Preparation		
Safety	Safety	Safety
Sequencing Challenge	Sequencing Challenge	
Procedure With Inquiry	Procedure (includes conceptual questions)	Procedure (no conceptual questions)
Sample Data		
Data Analysis	Data Analysis	Data Analysis
Analysis Questions	Analysis Questions	Analysis Questions
Synthesis Questions	Synthesis Questions	
Multiple Choice Questions	Multiple Choice Questions	
Extended Inquiry Suggestions		

## Electronic Materials

The CD accompanying this manual contains the following:

- ◆ Complete Teacher Guide and Student Guide (with Student Inquiry Worksheets) in PDF format (Acrobat™ 5.0 compatible)
- ◆ Student Inquiry Worksheets and Student Worksheets of the laboratory activities in an editable format (MS Word™ 97–2003 format). PASCO provides editable files of the student lab activities so that teachers can customize activities to their needs.
- ◆ The manual appendices in PDF format, including Tech Tips for using DataStudio™, Xplorer GLX™, SPARK™, SPARKvue, and individual sensor technologies

## Overview of Lab Activity Components

The first page of each lab activity presents information to assist the teacher in choosing and planning for the appropriate activities to include in lesson plans. These sections include 1) Objectives, 2) Procedural Overview, 3) Time Requirement, and 4) Materials and Equipment list.

**Objectives.** This section contains an overview of the subject matter students will be investigating in this activity.

**Note:** Student versions of the lab activities include the primary question or questions, the *driving questions*, being investigated, instead of the Objectives.

**Procedural Overview.** This section includes the general laboratory or investigation procedures students conduct.

**Time Requirement.** Three timeframes are defined: the length of time needed 1) for preparation, 2) for the pre-lab discussion and activity, and 3) for the lab activity. If there is no lab preparation needed, ten minutes is designated. This takes into account the time required for gathering the materials, making copies of the procedure, and any other normal preparations.

**Materials and Equipment.** This section lists all materials and equipment needed to carry out the activity procedure. If items in this list need to be created using additional materials, those are indicated as a footnote, and instructions for preparing them are in the Lab Preparation section.

**What Students Should Already Know.** This section details the concepts students should have developed before doing the activity. Use this section to gauge when to include this activity in lesson plans, in assessing requirements for prior learning, and as an outline for a review or discussion before starting the lab activity.

**Related Labs in This Guide.** Activities listed in this section are noted as either prerequisites to an activity or as conceptually related to other labs in the guide.

**Background.** The background information in the student activity may be the same information as in the teacher version, or it may be shorter or simplified. For the teacher, this section contains information that might be useful for assessing student responses. The Background is limited to information that will adequately frame the activity in the context of related curriculum materials. For broader and deeper information on a topic, refer to textbooks or other reference materials.

**Pre-Lab Activities.** For the upcoming lab activity, pre-lab activities accomplish some or all of the following:

- ◆ Engage student attention
- ◆ Access prior knowledge
- ◆ Identify misconceptions
- ◆ Model procedures for mathematical computations required in the activity
- ◆ Generate student questions

This section includes pre-lab homework questions that help prepare students to optimally benefit from performing the lab activity.

**Lab Preparation.** Here you will find instructions to prepare for the lab, such as:

## Introduction

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- ◆ Finding equipment or materials not listed in the Materials and Equipment list. These are usually items used in a pre-lab demonstration.
- ◆ Making solutions used in the activity and ideas for substituting materials
- ◆ Setting up stations in the classroom or preparing for a field trip

**Safety.** This section lists the pertinent safety procedures for this lab. The Normal Safety Procedures section of this guide lists standard safety procedures that should always be followed. The teacher's version of the Safety section may include additional safety considerations the teacher should be aware of.

**Using Your Data Collection System.** This section lists the data collection system technical procedures used in the activity. The actual instructions for them (referred to as "Tech Tips") are in the appendix that corresponds to the PASCO data collection system being used for the activity.

The Tech Tip number at the end of a technical step is used to locate that Tech Tip in the appropriate appendix. That number also appears by the corresponding instruction in the Procedure. Students can refer to that Tech Tip if they are not familiar with that use of the equipment.

We suggest you provide a copy of the Tech Tip appendix to each group of students, along with the user guides for the sensors used in this manual (needed for calibration procedures). Alternatively, the set of Tech Tips and sensor user guides could also be put in three-ring binders kept in the classroom.

**Sequencing Challenge.** This student activity encourages students to think about the experiment they are about to perform, giving them a holistic overview. The main operations they will be asked to carry out are summarized and scrambled. Students are asked to put the tasks in the sequence they need to be carried out, helping them form a mental model of the activity.

**Procedure with Inquiry.** This section directs the hands-on portion of the activity. Tasks are listed with check boxes to help students track their progress. Questions embedded in the procedure ask students to predict the results of the activity and to analyze and evaluate particular procedures, as well as the experimental design.

Each step that requires a technical procedure using the data collection system has associated instructions (Tech Tips). Students who need technical help to complete the process can consult the Tech Tips from the appendix that contains the instructions for the data collection system. Consider creating a technical notebook containing the Tech Tips and user guides for the sensors students use throughout the year (see also the Using Your Data Collection System section).

The teacher's version includes the answers to the embedded questions. The Student Inquiry Worksheet contains the embedded questions as described above. The Student Worksheet follows a traditional format for the procedure with the embedded questions removed.

**Data Analysis.** Students are encouraged to analyze their data in various ways: such as completing a data table, sketching a graph of the dependent variables versus time as it appears on the equipment display, or identifying key parts of the data plots. The Teacher Information contains sample data in graphs or tables to display expected patterns. However, student data may vary and are useful for discussions of variation in scientific data.

**Analysis Questions.** These questions help students to understand the collected data. Students make comparisons, summaries, computations, and conclusions based on their data, and may be asked to evaluate the design of the experiment to identify the independent, dependent, and controlled variables.

**Synthesis Questions.** These questions are designed to integrate information not covered in the lab. This requires students to develop a deeper understanding of concepts and tests whether students can transfer the knowledge learned in the lab to other situations. Some questions require students to consult available resources, such as textbooks, reference books, resources on the Internet, and local experts.

**Multiple Choice Questions.** These questions reinforce key information acquired from the activity or information related to the topic that goes beyond the activity. Although not identical to those found on standardized exams, the questions are similar to ones included in previous exams.

**Extended Inquiry Suggestions.** These suggestions are natural extensions of the activity and can be used for further student inquiry. They include ideas for further experimentation and hands-on exploration, classroom debates, field trips, or research papers.

## **Advanced Curriculum Support**

### ***Using These Labs for Advanced and Honors Classes***

The lab activities in this lab guide were designed to be used in Advanced Placement Program\* and International Baccalaureate Organization Program science classes. At this level, concepts are covered in greater depth, and investigations are more complex and have higher expectations for accuracy and detail than introductory science lab activities. These lab activities are flexible so that teachers can customize the activities and continually reduce the amount of support and scaffolding in the activities as the school year progresses. The lab activities in their current format are suitable for both advanced and honors level science courses.

### ***College Board\* Advanced Placement (AP\*) Program***

Through strategic preparation and vertical planning, Pre-AP\* and AP teachers can work together to prepare students for the rigors of Advanced Placement classes and beyond. (Refer to the College Board publication, “The AP Vertical Teams Guide for Science,” to help you create an AP Science Vertical Team in your school or district).

Use these laboratory exercises to help students understand the difficult concepts found in the course outline and the skills necessary to undertake scientific investigations. These skills include developing hypotheses, designing and implementing controlled experiments, analyzing data, thinking critically about the data to draw conclusions, and communicating these results to the scientific community in data tables and graphs. Such scientific processes require a high level of thinking, as well as an understanding of laboratory techniques and equipment.

Virtually every lab activity guides students to gather, analyze, and evaluate their own data, and to explain the trends observed in the data. However, data analysis alone is not enough. The Pre-AP and AP student must explain the data in relation to the topic of study. These lab activities support students to master this challenge by helping them acquire math skills, gain metric literacy, and become proficient in data analysis. The assessment questions help students gain problem-solving and reasoning skills required for success on the AP Exam.

Note: This lab guide includes all 22 required AP Chemistry and 10 Advanced Chemistry labs, providing a wealth of information that can easily be adapted to the Pre-AP and AP Chemistry classrooms. AP students need to complete all 22 AP Chemistry labs. Additional Advanced Chemistry lab activities can be used to enhance specific topics.

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### **International Baccalaureate Organization (IBO\*\*) Diploma Program**

The International Baccalaureate Organization (IBO) uses a specific science curriculum model that includes both theory and practical investigative work. While this lab guide was not produced by the IBO and does not include references to the internal assessment rubrics, it does provide a wealth of information that can be adapted easily to the IB classroom.

By the end of the IB Diploma Program students are expected to have completed a specified number of practical investigative hours and are assessed using the specified internal assessment criteria. Students should be able to design a lab based on an original idea, carry out the procedure, draw conclusions, and evaluate their own results. These scientific processes require an understanding of laboratory techniques and equipment as well as a high level of thinking.

### **Using these Labs with the AP and the IBO Programs**

Use this lab guide to teach students the skills and concepts to be successful in Pre-AP and AP Chemistry and to meet IB requirements.

- ◆ **Use complete labs:** In the beginning complete the entire lab activity, using it as a model of what an investigation needs to include. This will teach students manipulative skills, such as proper lab technique, use of equipment, safety procedures and considerations, and following instructions. Features such as the Sequencing Challenge and embedded questions in the procedure help students understand why the lab directs them to perform certain steps. They can then apply these procedures in the labs they design on their own later in the course.

After students complete the lab, use an earlier AP free-response question to show students how the lab might be assessed on the AP exam. Use a student's data to answer the question and show students how the rubric is used to grade the question. Ask students to determine the independent, dependent, and controlled variables in the experiment. If rate of change is an important concept in the lab, be sure that students understand the importance of rate, its relationship to slope, and how to calculate rate.

For IB students, pick one part of the internal assessments rubrics to go over with the students. For example, review the design of the experiment and have students explain what the independent, dependent, and controlled variables are in the experiment. Ask students to design a similar experiment, but change the independent variable.

- ◆ **Delete certain sections:** As students become familiar with the skills and processes needed to design their own labs, start deleting certain sections of the labs and have students complete those parts on their own. For example, when teaching students to write their own procedures have the students complete one lab as it is in the lab guide. In the next lab, keep the Sequencing Challenge, but have students write a more elaborate procedure. Finally, remove both the Sequencing Challenge and the Procedure sections and have students write the entire procedure.

Encourage students to make their own data tables. Leave the procedure, but remove the data tables and require the students to create them on their own. In another lab, leave the driving question and procedure, but remove the analysis questions and have students write their own analysis, conclusion, and evaluation.

- ◆ **Use only the driving question:** As students progress through their understanding of the structure of an experiment, provide them with just the driving question and let them do the rest. Some

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of the driving questions are too specific (they give the students the independent variable), so revise them appropriately.

- ◆ **Extended inquiry:** After students complete an activity in the lab guide, use the extended inquiry suggestions to have the students design their own procedure, or the data collection and processing, or both.

### **About Correlations to Science Standards**

Appendix E lists correlations between the lab activities and various educational standards, including:

- ◆ Specific United States National Science Education Standards addressed by the performance, discussion, and analysis of each laboratory activity.
- ◆ Correlations to AP Chemistry Topic Outline

Additional correlations of activities to other science standards, including State Science Standards, are available at [www.pasco.com](http://www.pasco.com).

### **Global Number Formats and Standard Units**

#### ***Using Standard Units (SI Units; Metric System)***

Throughout this guide, the International System of Units (SI) or metric units is used unless specific measurements, such as air pressure, are conventionally expressed otherwise. In some instances, it may be necessary to alter the units used to adapt the material to conventions typically used and widely understood by the students.

### **Reference**

National Research Council. *National Science Education Standards*. (1997). Washington, DC: National Academy Press.



# **Normal Laboratory Safety Procedures**

## **Overview**

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PASCO is concerned with your safety and because of that, we are providing a few guidelines and precautions to use when exploring the labs in our Chemistry guide. This is a list of general guidelines only; it is by no means all-inclusive or exhaustive. Of course, common sense and standard laboratory safety practices should be followed.

Please keep in mind that the safety regulations of the institution, local, and state safety regulations always have to be observed and they supersede safety regulations in this guide.

Since handling and disposal procedures vary, our safety precautions and disposal comments are generic. Depending on your lab, instruct students on proper disposal methods. Each of the lab activities also has a Safety section for procedures necessary for that activity.

## **General Lab Safety Procedures and Precautions**

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- ◆ Follow all standard laboratory procedures.
- ◆ Absolutely no food, drink, and chewing gum is allowed in the lab.
- ◆ Keep water away from electrical outlets.
- ◆ Wear eye protection (splash-proof goggles), lab apron, and protective gloves.
- ◆ Do not touch your face with gloved hands. If you need to sneeze or scratch, take off your gloves, wash your hands, and then take care of the situation. Do not leave the lab with gloves on.
- ◆ Wash your hands after handling chemicals, glassware, and equipment.
- ◆ Know the safety features of your lab such as eye-wash stations, fire extinguisher, first-aid equipment or emergency phone use.
- ◆ Insure that loose hair and clothing is secure when in the lab.
- ◆ Handle glassware with care.
- ◆ Insure you have adequate clear space around your lab equipment before starting an activity.
- ◆ Do not wear open toe shoes or short pants in the laboratory.
- ◆ Allow heated objects and liquids to return to room temperature before moving.
- ◆ Never run or joke around in the laboratory.
- ◆ Do not perform unauthorized experiments.
- ◆ Students should use a buddy system in case of trouble.
- ◆ Keep the work area neat and free from any unnecessary objects.

## **Water Related Safety Precautions and Procedures**

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- ◆ Keep water away from electrical outlets.
- ◆ Keep water away from all electronic equipment.

## **Chemical Related Safety Precautions and Procedures**

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- ◆ Consult the manufacturer's Material Safety Data Sheets (MSDS) for instructions on handling, storage, and disposing of chemicals. Your teacher should provide the MSDS-es of the chemicals that you are using. Keep these instructions available in case of accidents.
- ◆ Many chemicals are hazardous to the environment and should not be disposed of down the drain. Always follow your teacher's instructions for disposing of chemicals.
- ◆ Sodium hydroxide, hydrochloric acid, sulfuric acid, and acetic acid are corrosive irritants. Avoid contact with your eyes and wash your hands after handling. In case of skin exposure, wash it off with plenty of water.
- ◆ Always add acids and bases to water, not the other way around, as the solutions may boil vigorously.
- ◆ Diluting acids and bases creates heat; be extra careful when handling freshly prepared solutions and glassware, as they may be very hot.
- ◆ Handle concentrated acids and bases in a fume hood; the fumes are caustic and toxic.
- ◆ Wear eye protection, lab apron, and protective gloves when handling acids. Splash-proof goggles are recommended. Either latex or nitrile gloves are suitable. Use nitrile gloves if you have latex allergy.
- ◆ Read labels on all chemicals and pay particular attention to hazard icons and safety warnings.
- ◆ When handling any bacterial species, follow aseptic techniques.
- ◆ Wash your hands before and after a laboratory session.
- ◆ If any solution comes in contact with skin or eyes, rinse immediately with a copious amount of running water for a minimum of 15 minutes.
- ◆ Follow the teacher's instructions for disposing of chemicals.
- ◆ Check the label to verify it is the correct substance before using it.
- ◆ Never point the open end of a test tube containing a substance at yourself or others.
- ◆ Use a wafting motion when smelling chemicals.
- ◆ Do not return unused chemicals to their original container.
- ◆ Keep flammable chemicals from open flame.

## **Dangerous or Harmful Substance Related Lab Safety Precautions**

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- ◆ When handling any bacterial species, follow aseptic techniques.
- ◆ Always flame inoculating loops and spreaders before setting them down on the lab bench.
- ◆ Pipetting suspension cultures can create an aerosol. Keep your nose and mouth away from the tip of the pipet to avoid inhaling any aerosol
- ◆ Use caution when working with acids.
- ◆ Use appropriate caution with the matches, burning splint and foods, and other hot materials.
- ◆ Be careful using a knife or scalpel.

## **Other Safety Precautions**

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- ◆ If water is boiled for an experiment involving heat, make sure it is never left unattended. Remember, too, that the hot plate will stay hot well after it is unplugged or turned off.
- ◆ Any injury must be reported immediately to the instructor; an accident report has to be completed by the student or a witness.
- ◆ If you are suffering from any allergy, illness, or are taking any medication, you must inform the instructor. This information could be very important in an emergency.
- ◆ Try to avoid wearing contact lenses. If a solution spills in your eye, the presence of a contact lens makes first aid difficult and can result in permanent damage. Also, organic solvents tend to dissolve in soft contact lenses, causing eye irritation.

## **Additional Resources**

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- ◆ Flinn Scientific
- ◆ The Laboratory Safety Institute (LSI)
- ◆ National Science Education Leadership Association (NSELA)/Safe Science Series



## Master Materials and Equipment List

Italicized entries indicate items not available from PASCO. The quantity indicated is per student or group. NOTE: These activities also require protective gear for each student (for example, safety goggles, gloves, apron, or lab coat).

Teachers can conduct some lab activities with sensors and probes other than those listed here. For assistance with substituting compatible sensors and probes for a lab activity, contact PASCO Teacher Support (800-772-8700 inside the United States or <http://www.pasco.com/support>).

Act	Title	Materials and Equipment	Qty
1	<b>Scientific Inquiry</b> Use a fast response temperature sensor in the design of a simple experiment as students attempt to slow the cooling rate of the water by adding insulation to the cup.	Data Collection System PASPORT Fast Response Temperature Probe <i>9-12 oz. cup</i> <i>Hot water</i> <i>Insulating devices readily available in the laboratory (polystyrene, foil, plastic wrap, cloth, wool, packing peanuts)</i>	1 1 1 16 oz A variety
2	<b>Lab 1: Determining the Empirical Formula of a Compound</b> Use a crucible and Bunsen burner to react a chemical with air in order to determine the stoichiometric composition of an ionic compound.	<i>Crucible with lid</i> Ring stand <i>Bunsen burner</i> Balance <i>Crucible tongs</i> <i>Wash bottle with deionized water</i> <i>Clay triangle</i> <i>Paper clip</i> <i>Magnesium powder</i>	1 1 1 1 per class 1 1 1 1 0.5 g
3	<b>Lab 2: Determine the Percentage of Water in a Hydrate</b> Use a crucible and Bunsen burner to determine the water content of a hydrated salt.	<i>Crucible with lid</i> <i>Crucible tongs</i> <i>Bunsen burner</i> Ring stand <i>Clay triangle</i> Balance <i>Wash bottle with deionized water</i> <i>Copper sulfate, CuSO<sub>4</sub>, hydrated</i>	1 1 1 1 1 1 per class 1 4.5 g
4	<b>Lab 3: Determine the Molar Mass of a Volatile Liquid</b> Use a stainless steel temperature sensor to determine the molar mass of an unknown volatile liquid at the boiling temperature of water and atmospheric pressure.	Data Collection System PASPORT Stainless Steel Temperature Sensor PASPORT Absolute Pressure Sensor Quick-release connector* Tubing connector* Tubing, 1- to 2-cm* <i>Beaker, 400-mL</i> <i>Erlenmeyer flask, 125-mL</i> <i>Graduated cylinder, 100-mL</i> Hot plate with magnetic stirrer and stir bar Balance Ring stand <i>Clamp</i> <i>Unknown volatile liquid (use acetone)</i> <i>Aluminum foil, about 4-cm by 4-cm</i> <i>Paper towel, sheets</i> <i>Dropper</i> <i>Water</i>	1 1 1 1 1 2 1 1 1 per class 1 2 8 mL 1 2 or 3 1 600 mL





Act	Title	Materials and Equipment	Qty
	<b>Chemical Reaction</b> Use a colorimeter to determine the equilibrium constant for a chemical reaction.	PASPORT Sensor Extension Cable <i>Beaker, 50-mL</i> <i>Test tube, 15-mL</i> <i>Test tube rack</i> Graduated pipet, 10-mL <i>Pipet bulb</i> <i>0.01 M Iron (Fe<sup>3+</sup>)</i> <i>0.00300 M Potassium thiocyanate (KSCN)</i> <i>Kimwipes®</i> <i>Deionized water</i> <i>Marker</i>	1 2 5 1 2 1 20 mL 20 mL 1 40 mL 1
12	<b>Lab 11: Using Different Indicators for pH Determination</b> Use a drop counter and pH sensor to determine the CO <sub>2</sub> content of a beverage by performing titrations with multiple acid-base indicators.	Data Collection System PASPORT High Accuracy Drop Counter PASPORT pH Sensor Micro stir bar Clamp, right-angle Clamp, buret Buret, 50-mL <i>Beaker, 25-mL</i> <i>Beaker, 250-mL</i> <i>Erlenmeyer flask, 250-mL</i> <i>Graduated cylinder, 100-mL</i> <i>Phenolphthalein</i> <i>Methyl orange</i> Magnetic stirrer and stir bar Ring stand <i>Commercial soda drink</i> <i>Kimwipes®</i> <i>4.00 M HCl solution</i> <i>1 M NaOH solution</i> <i>Wash bottle with deionized water</i> <i>Funnel</i> <i>Balloon (fits on Erlenmeyer flask; holds 100 mL)</i> Buffers, pH 4 and pH 10 <i>Cotton swab or tissue</i>	1 1 1 1 1 1 1 2 2 1 1 5 drops 5 drops 1 1 1 can 1 100 mL 100 mL 1 1 1 10 mL 1
13	<b>Lab 12: Determination of the Rate of the Decomposition of Hydrogen Peroxide</b> Use an absolute pressure sensor and stainless steel temperature sensor to determine the rate constant of a chemical reaction.	Data Collection System PASPORT Absolute Pressure Sensor PASPORT Stainless Steel Temperature Sensor PASPORT Sensor Extension Cable Quick-release connector* Tubing connector* Tubing, 1- to 2-cm* <i>Beaker, 100-mL</i> <i>Erlenmeyer flask, 250-mL</i> Graduated pipet, 25-mL <i>Pipet bulb</i> <i>Stopper with two holes for the Erlenmeyer flask</i> <i>Beaker, 50-mL</i> <i>Glycerin</i> <i>0.1000 M Potassium iodine (KI)</i>	1 1 1 1 1 1 1 3 1 3 3 1 1 several drops 60 mL

## Master Materials and Equipment List

Act	Title	Materials and Equipment	Qty
		<i>3% Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>)</i>	40 mL
		<i>Deionized water</i>	100 mL
		<i>Electrical tape, 60 in. (optional)</i>	1
14	<b>Lab 13: Enthalpy of a Chemical Reaction</b> Use a stainless steel temperature sensor to derive the enthalpy change of a reaction.	Data Collection System	1
		PASPORT Stainless Steel Temperature Sensor	1
		<i>Polystyrene cup, 8 oz.</i>	1
		Clamp, utility	1
		<i>Beaker, 250-mL</i>	1
		<i>Graduated cylinder, 50-mL or 100-mL</i>	1
		Ring stand	1
		<i>2.00 M Sodium hydroxide (NaOH)</i>	50 mL
		<i>2.00 M Hydrochloric acid (HCl)</i>	50 mL
		<i>2.00 M Ammonium chloride (NH<sub>4</sub>Cl)</i>	50 mL
		<i>2.00 M Ammonia (NH<sub>3</sub>)</i>	50 mL
15	<b>Lab 14a: Separation and Analysis of Cations</b> Use chemical reactions and chemical properties to identify the cations present in a mixture by systematically reacting the unknown with various reagents.	<i>Test tube, 10-mL</i>	10
		<i>Test tube rack</i>	1
		Pipet, graduated, 10-mL	1
		<i>Pipet bulb</i>	1
		Pipet, plastic, 1-mL	7
		<i>Centrifuge</i>	1
		<i>Beaker, 250-mL</i>	1
		<i>Evaporating dish</i>	1
		<i>Stirring rod</i>	1
		<i>Hot plate</i>	1
		<i>Litmus paper</i>	10
		<i>pH paper</i>	1 roll
		<i>6 M Sodium hydroxide (NaOH)</i>	20 mL
		<i>6 M Ammonia (NH<sub>3</sub>)</i>	20 mL
		<i>0.1 M Potassium chromate (K<sub>2</sub>CrO<sub>4</sub>)</i>	20 mL
		<i>1% Aluminon dye</i>	2 mL
		<i>6 M Hydrochloric acid (HCl)</i>	20 mL
		<i>Dimethylglyoxime (DMG) reagent</i>	5 drops
		<i>0.2 M Potassium ferrocyanide (K<sub>4</sub>[Fe(CN)<sub>6</sub>])</i>	2 mL
		<i>3 M Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>)</i>	3 mL
		<i>3 % Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>)</i>	2 mL
		<i>Unknown cation solution (use AlCl<sub>3</sub>, NiCl<sub>2</sub>, Pb(NO<sub>3</sub>)<sub>2</sub>, AgNO<sub>3</sub>, MnSO<sub>4</sub>, (NH<sub>4</sub>)<sub>2</sub>Fe(SO<sub>4</sub>)<sub>2</sub>)</i>	20 mL
		<i>Deionized water</i>	5 mL
		<i>Marking pen</i>	1

Act	Title	Materials and Equipment	Qty
16	<b>Lab 14b: Analysis of Anions</b> Use chemical reactions and chemical properties to analyze solutions of known anions, using the results to analyze a solution of unknown anions.	<i>Test tube, 10-mL</i> <i>Test tube rack</i> <i>Pipets, 1 mL, disposable</i> <i>Stirring rods</i> <i>Litmus paper</i> <i>0.2 M Sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>)</i> <i>0.2 M Monopotassium phosphate (KH<sub>2</sub>PO<sub>4</sub>)</i> <i>0.2 M Sodium nitrate (NaNO<sub>3</sub>)</i> <i>0.2 M Sodium chloride (NaCl)</i> <i>Unknown anion solution (use Na<sub>2</sub>SO<sub>4</sub>, KH<sub>2</sub>PO<sub>4</sub>, NaNO<sub>3</sub>, NaCl)</i> <i>0.2 M Barium nitrate (Ba(NO<sub>3</sub>)<sub>2</sub>)</i> <i>Saturated iron(II) sulfate (FeSO<sub>4</sub>)</i> <i>0.1 M Silver nitrate (AgNO<sub>3</sub>)</i> <i>6 M Nitric acid (HNO<sub>3</sub>)</i> <i>5 M Ammonia (NH<sub>3</sub>)</i> <i>3 M Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>)</i> <i>Concentrated H<sub>2</sub>SO<sub>4</sub></i> <i>Distilled water</i> <i>Centrifuge</i> <i>Marking pen</i>	13 1 13 5 15 10 mL 5 mL 5 mL 5 mL 20 mL 5 mL 2 mL 5 mL 5 mL 5 mL 5 mL 2 mL 10 mL 1 1
17	<b>Lab 15a: Synthesis of a Coordination Compound</b> Use a series of reactions to synthesize a coordination compound, potassium aluminum sulfate dodecahydrate (alum), and calculate the theoretical and percent yields.	<i>Balance</i> <i>Hot plate</i> <i>Fume hood</i> <i>Beaker, 400-mL</i> <i>Beaker, 250-mL</i> <i>Beaker, 100-mL</i> <i>Graduated cylinder, 50-mL</i> <i>Büchner funnel</i> <i>Büchner filter flask</i> <i>Stirring rod, glass</i> <i>Watch glass</i> <i>Scissors</i> <i>Beaker tongs</i> <i>Filter paper</i> <i>Wire gauze</i> <i>3 M Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>)</i> <i>3 M Potassium hydroxide (KOH)</i> <i>50% Ethanol</i> <i>100% Ethanol</i> <i>Acetone (C<sub>3</sub>H<sub>6</sub>O)</i> <i>Aluminum foil</i> <i>Distilled water for rinsing equipment</i> <i>Ice</i>	1 per class 1 1 1 2 1 1 1 1 1 1 1 1 1 1 1 3 1 35 mL 25 mL 50 mL 50 mL 50 mL 1.1 g 1 400 mL

**Master Materials and Equipment List**

Act	Title	Materials and Equipment	Qty
18	<b>Lab 15b: Analysis of a Coordination Compound</b> Use a stainless steel temperature sensor to help confirm the identity of a sample of alum synthesized in Lab 15a by conducting both qualitative and quantitative analyses.	Data Collection System	1
		PASPORT Stainless Steel Temperature Sensor	1
		Ring stand with ring	1
		<i>Clay triangle</i>	1
		Clamp, buret	1
		Clamp, utility	1
		<i>Crucible with lid</i>	1
		<i>Tongs</i>	1
		<i>Test tubes, 10 mL</i>	2
		<i>Beaker, 250-mL</i>	1
		<i>Capillary tube</i>	1
		<i>Stirring rod</i>	1
		<i>Watch glass, 100-mm</i>	2
		Balance	1 per class
		<i>Centrifuge</i>	1 per class
		<i>Wire with a loop on the end, 4 in.</i>	1
		Hot plate	1
		<i>Bunsen burner</i>	1
		<i>Striker</i>	1
		<i>0.2 M Barium chloride (BaCl<sub>2</sub>)</i>	1 mL
		<i>6 M Sodium hydroxide (NaOH)</i>	5 mL
		<i>6 M Hydrochloric acid (HCl)</i>	5 mL
		<i>Borax</i>	0.5 g
<i>Alum from previous experiment</i>	3 g		
<i>Rubber band</i>	1		
<i>Water</i>	200 mL		
<i>Distilled water</i>	10 mL		







## Master Materials and Equipment List

Act	Title	Materials and Equipment	Qty
26	<b>Lab 22a: Organic Synthesis I—Preparation</b> Use a stainless steel temperature sensor to synthesize an organic compound (aspirin).	Data Collection System	1
		PASPORT Stainless Steel Temperature Sensor	1
		Ring stand	1
		Clamp	2
		<i>Erlenmeyer flask, 125-mL</i>	1
		<i>Graduated cylinder, 10-mL</i>	1
		<i>Beaker, 100-mL</i>	1
		<i>Beaker, 400-mL</i>	1
		Hot plate	1
		<i>Büchner filter flask</i>	1
		<i>Büchner funnel</i>	1
		<i>Filter paper</i>	1
		<i>Salicylic acid (C<sub>7</sub>H<sub>6</sub>O<sub>3</sub>)</i>	2 g
		<i>Acetic anhydride (C<sub>4</sub>H<sub>6</sub>O<sub>3</sub>)</i>	4 mL
		<i>Concentrated phosphoric acid (H<sub>3</sub>PO<sub>4</sub>)</i>	1 mL
		<i>Wash bottle with distilled water</i>	1
		<i>Eye dropper</i>	1
		<i>Rubber policeman</i>	1
		<i>Ice cold distilled water</i>	50 mL
		<i>Ice for ice bath</i>	300 mL
	<i>Forceps</i>	1	
27	<b>Lab 22b: Organic Synthesis II—Analysis</b> Use a stainless steel temperature sensor, pH sensor, and drop counter to perform qualitative and quantitative analytical methods, including melting point determination and titration, to analyze the purity of the aspirin synthesized in Lab 22a.	Data Collection System	1
		PASPORT Stainless Steel Temperature Sensor	1
		PASPORT pH Sensor	1
		PASPORT High Accuracy Drop Counter	1
		Micro stir bar	1
		Ring stand	1
		Clamp, utility	1
		Clamp, right-angle	1
		Clamp, buret	1
		<i>Beaker, 150-mL</i>	2
		<i>Beaker, 100-mL</i>	1
		<i>Beaker, 25-mL</i>	2
		<i>Test tubes, 15-mL</i>	3
		<i>Melting point capillary tube</i>	1
		Buret, 50-mL	1
		<i>Graduated cylinder, 100-mL</i>	1
		Magnetic stirrer and stir bar	1
		Hot plate with magnetic stirrer and stir bar	1
		<i>Mortar and pestle</i>	1
		<i>Product from Organic Synthesis I activity</i>	1
		<i>Aspirin tablet</i>	1
		<i>Ethanol</i>	15 mL
		<i>0.1 M Sodium hydroxide (NaOH)</i>	75 mL
		<i>1% Iron chloride (FeCl<sub>3</sub>)</i>	2 mL
		<i>Mineral oil</i>	150 mL
		<i>Buffers, pH 4 and pH 10</i>	10 mL
		<i>Water, distilled</i>	100 mL
<i>Rubber band, small</i>	1		
<i>Wash bottle with deionized water</i>	1		









**Calibration materials**

If you want to calibrate various sensors, you will need the following:

**pH Sensor**

Item	Quantity	Where Used
Buffer solution, pH 4	25 mL	7, 8, 12, 23, 27, 28, 29, 35
Buffer solution, pH 10	25 mL	
Beaker, small	3	
Wash bottle with deionized or distilled water	1	

**Colorimeter**

Item	Quantity	Where Used
Cuvette (included with colorimeter)	1	11, 21, 30, 37
Distilled water	7 mL	

## Activity by PASCO Sensors

This list shows the sensors and other PASCO equipment used in the lab activities.

Items Available from PASCO	Qty	Activity Where Used
Data Collection System	1	1, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 18, 19, 20, 21, 23, 24, 25, 26, 27, 28, 29, 30, 31, 32, 33, 34, 35, 36, 37
PASPORT Alpha Beta Gamma Radiation Sensor	1	36
PASPORT Absolute Pressure Sensor <sup>1</sup>	1	4, 6, 13, 32, 33, 34
PASPORT Colorimeter	1	11, 21, 30, 37
PASPORT Conductivity Sensor	1	10, 31
PASPORT High Accuracy Drop Counter	1	7, 8, 9, 12, 27, 28, 29, 31, 35
PASPORT Fast Response Temperature Probe	1	1
PASPORT Oxidation-Reduction Potential Electrode	1	9
PASPORT pH Sensor <sup>1</sup>	1	7, 8, 12, 23, 27, 28, 29, 35
PASPORT Sensor Extension Cable	1	6, 11, 13, 20, 21, 30, 32, 33, 34, 37
PASPORT Stainless Steel Temperature Sensor <sup>1</sup>	1	4, 5, 6, 13, 14, 18, 19, 26, 27, 32, 33, 34
PASPORT Voltage Sensor <sup>1</sup>	1	24
PASPOST Voltage-Current Sensor	1	25
Amadeus Spectrometer System	1	20
Isotope Generator Kit (Barium-137m)	1	36

<sup>1</sup>This sensor is part of the PASPORT Chemistry Sensor, a MultiMeasure Sensor™



## Activity and Filename Cross Reference

### Filename Identification

Each activity has three files associated with it: the teacher file, the student inquiry worksheet, and the traditional student worksheet. Each filename is prefaced with "AC" (for Advanced Chemistry). Filenames can end with "T.doc", "(I) S.doc", or "(TR) S.doc".

- ◆ "T.doc" at the end of the filename indicates the teacher document. It contains supplementary information to help teachers prepare and implement the activity, and includes answers and sample data.
- ◆ "(I) S.doc" indicates the student inquiry worksheet.
- ◆ "(TR) S.doc" indicates a traditional student worksheet.

The table below refers only to the filename of the teacher document ("T.doc"). Except for using "(I) S.doc" or "(TR) S.doc," the names are the same.

Lab activity names and filenames

Activity Name	Filename
Scientific Inquiry – SPARK Science Learning System™	Advanced Scientific Methods SPARK SLS
Scientific Inquiry – SPARKvue™	Advanced Scientific Inquiry SPARKvue
Scientific Inquiry – Xplorer GLX®	Advanced Scientific Methods GLX
Scientific Inquiry – DataStudio®	Advanced Scientific Inquiry DataStudio
Lab 1: Determining the Empirical Formula of a Compound	AC Lab 1 Determine Empirical Formula
Lab 2: Determine the Percentage of Water in a Hydrate	AC Lab 2 Determine Percent Water
Lab 3: Determine the Molar Mass of a Volatile Liquid	AC Lab 3 Determine Molar Mass Volatile Liquid
Lab 4: Molecular Weight by Freezing Point Depression	AC Lab 4 Molecular Weight Freezing Point
Lab 5: Molar Volume of a Gas	AC Lab 5 Molar Volume of a Gas
Lab 6: Standardizing a Solution of Sodium Hydroxide	AC Lab 6 Standardizing NaOH Solution
Lab 7: Acid–Base Titration	AC Lab 7 Acid Base Titration
Lab 8: Oxidation–Reduction Titration	AC Lab 8 Oxidation Reduction Titration

## Advanced Environmental Science

Activity Name	Filename
Lab 9: Mole Relationships in a Chemical Reaction	AC Lab 9 Mole Relationships Chemical Rxn
Lab 10: Determine the Equilibrium Constant for a Chemical Reaction	AC Lab 10 Determine Equilibrium Constant
Lab 11: Using Different Indicators for pH Determination	AC Lab 11 Indicators for pH Determination
Lab 12: Determination of the Rate of the Decomposition of Hydrogen Peroxide	AC Lab 12 Decomposition Rate
Lab 13: Enthalpy of a Chemical Reaction	AC Lab 13 Enthalpy of a Chemical Reaction
Lab 14a: Separation and Analysis of Cations	AC Lab 14a Separation and Analysis of Cations
Lab 14b: Analysis of Anions	AC Lab 14b Analysis of Anions
Lab 15a: Synthesis of a Coordination Compound	AC Lab 15a Synth of Coord Comp
Lab 15b: Analysis of a Coordination Compound	AC Lab 15b Analysis of Coord Comp
Lab 16: Gravimetric Determination of a Precipitate	AC Lab 16 Gravimetric Determination
Lab 17a: Absorption Spectra	AC Lab 17a Absorption Spectra
Lab 17b: Colorimetric Analysis	AC Lab 17b Colorimetric Analysis
Lab 18: Separation by Liquid Chromotography	AC Lab 18 Liquid Chromotography
Lab 19: Properties of Buffer Solutions	AC Lab 19 Properties of Buffer Solutions
Lab 20: Determination of Electrochemical Series	AC Lab 20 Determine Electrochemical Series
Lab 21: Electroplating	AC Lab 21 Electroplating
Lab 22a: Organic Synthesis I—Preparation	AC Lab 22a Organic Synthesis I
Lab 22b: Organic Synthesis II—Analysis	AC Lab 22b Organic Synthesis II
Lab 23: Determination of a Solubility Product	AC Lab 23 Determine Solubility Product
Lab 24: Determining $K_a$ by Half-Titration of a Weak Acid	AC Lab 24 Determine $K_a$ by Half Titration
Lab 25: Order of Reaction	AC Lab 25 Order of Reaction
Lab 26: Conductometric Titration	AC Lab 26 Conductometric Titration

<b>Activity Name</b>	<b>Filename</b>
Lab 27: Identifying an Unknown Metal	AC Lab 27 Identifying an Unknown Metal
Lab 28: Molecular Interaction in Ethanol and Acetone	AC Lab 28 Molecular Interaction
Lab 29: Exploring Gas Laws	AC Lab 29 Exploring Gas Laws
Lab 30: Determination of the $K_a$ Values of Two Isomeric Multi-Protic Acids	AC Lab 30 Determine $K_a$ Two Isomer Acids
Lab 31: Determining the Half-Life of an Isotope	AC Lab 31 Determine Half Life of Isotope
Lab 32: The Breathalyzer™ Test for Alcohol	AC Lab 32 The Breathalyzer(tm) Test



# Scientific Inquiry – SPARK Science Learning System™

## Objectives

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This lab is designed to help students familiarize themselves with the SPARK Science Learning System™ (SPARK) while engaging in scientific investigations. By completing these exercises students learn to:

- ◆ Engage in the process of scientific inquiry by making predictions, collecting and analyzing data, and explaining observations
- ◆ Use the most popular and effective SPARK tools

## Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Setting up the SPARK Science Learning System (SPARK) to collect data in different types of experiments
- ◆ Using the manual and periodic sampling modes of SPARK
- ◆ Using the fast-response temperature probe with SPARK to collect temperatures of various body parts in a table display
- ◆ Using the fast-response temperature probe and the graph display to continuously record and display the temperature of hot water that is left to cool in an insulated and non-insulated cup at room temperature for 5 minutes
- ◆ Designing a way to insulate a cup so it keeps hot water warm for as long as possible

## Time Requirement

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- |                                   |            |
|-----------------------------------|------------|
| ◆ Preparation time                | 5 minutes  |
| ◆ Pre-lab discussion and activity | 5 minutes  |
| ◆ Lab activity                    | 40 minutes |

## Materials and Equipment

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### *For each student or group:*

- |                                   |   |
|-----------------------------------|---|
| ◆ SPARK Science Learning System™  | ◆ Materials available in the laboratory to design a |
| ◆ Fast-response temperature probe | cup that keeps water hot <sup>2</sup>               |
| ◆ Cup <sup>1</sup> , 9- to 12-oz  | ◆ Hot water, 16 oz                                  |

<sup>1</sup>Use a non-insulated plastic or paper cup.

<sup>2</sup>For example: polystyrene, cloth, foil, plastic wrap, wool, water baths, or packing peanuts.

## **Background**

---

### **The Scientific Method and Scientific Inquiry**

Scientific inquiry is a general term for the numerous ways in which scientists learn about the natural world to produce scientific knowledge. The knowledge produced is supported by evidence collected during the inquiry process.

Traditionally, the term scientific method has been used when teaching students how scientists learn about the natural world. However, this term has recently been criticized because it creates the misconception that all scientists follow the same steps (processes) in the same order all the time. In reality, the many methods used to investigate nature in the various sciences are better represented by a broader term, scientific inquiry.

Although the sequence and choice of steps used varies from scientist to scientist and from one investigation to the next, a common set of processes is used by scientists to learn about the natural world. Making observations; asking questions; making predictions or posing hypotheses; planning investigations; controlling variables; using systematic, reliable, repeatable, and externally validated techniques; analyzing the data collected; and interpreting and communicating results are among the major processes that are involved in scientific inquiry.

Each lab in this manual is designed to guide students through a sequence of scientific inquiry processes that may be used to learn about the world around them. Students learn about scientific inquiry by engaging in inquiry processes, and they develop the skills needed to answer their own questions through scientific inquiry. At the same time, students gain content knowledge by actively engaging in scientific inquiry as opposed to being told about or reading about science.

Scientists in the 21st century increasingly rely on technology to enhance data collection and analysis. Technology enables scientists to collect large amounts of data accurately and to display, analyze, and interpret their data more quickly.

### **Using the SPARK Science Learning System**

This investigation includes detailed steps on how to set up the SPARK Science Learning System (SPARK) to collect data in different types of experiments. In the first part of this investigation, students find the temperature of four different body parts. To do this, the student will need to place the temperature probe on the body part and allow the temperature to stabilize. When ready, the student will trigger SPARK to record the data point. SPARK allows for this type of data collection in manual sampling mode. Manual sampling mode allows users to see the data value as it changes over time and to record a data point when they are ready.

In the second part of this investigation, students measure temperature values each second as a cup of hot water cools. SPARK allows for a measurement to be automatically recorded at a specified rate. This type of data collection is called periodic sampling (this is the default mode). Taking many data points each second (high sampling rates) is needed when changes are expected to occur very quickly. Lower sampling rates are used when changes occur slowly or data is being taken for an extended period of time. The default sample rate for each PASCO sensor is appropriate for the most typical applications of that sensor, reducing the need for the user to change the sample rate before each experiment.

This investigation also guides students in two different ways to display data (table and graph) and how to start and stop data collection, open and close tool palettes, perform simple analyses (statistics), and save data.

This combination of methods and tools can be a powerful stimulation for students to develop their own scientific inquiries arising from their natural curiosity about the world around them.

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Lead a discussion with your students about what science is and what processes scientists use to learn about the world around them. Lead the discussion with questions such as “What is science?” and “What do scientists do?” Students will probably start out with specifics about what different scientists do. Let the ideas emerge, and see if you can find a common thread within the ideas. Ask students questions about different types of technologies scientists use and how these technologies help them in the different processes.

**Teacher Tip:** Accept all answers and write ideas on the board or overhead projector to remain displayed during the activity.

Ideally, you can lead your students to the conclusion that science is “asking questions, making predictions (constructing hypotheses), conducting experiments, collecting data (gathering evidence), analyzing the data, and drawing conclusions.” Let your students know that this is exactly what they will be doing in this laboratory exercise and throughout the year in your class.

## Lab Preparation

These are the materials and equipment to set up prior to the lab.

1. Make cups and insulating material available to each student group. Use what you have on hand as possible insulating materials. Some suggestions include: polystyrene cups, cloth, foil, plastic wrap, wool, water baths, or packing peanuts.
2. Make sure that a source of hot water with a constant temperature ( $\sim 50\text{ }^{\circ}\text{C}$ ) is available for your students throughout the activity.

## Safety

Add these important safety precautions to your normal laboratory procedures:

- ◆ Do not use the fast-response temperature probe in water that is  $70\text{ }^{\circ}\text{C}$  or hotter.
- ◆ Be careful when working with hot water.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

3	1	5	2	4
Next, carry out a controlled experiment to determine whether body temperature is the same for different parts of your body.	Turn on the SPARK Science Learning System, and connect the fast-response temperature probe.	Analyze the effectiveness of the cup you designed to slow the rate at which warm water cools.	First, determine how the temperature of your thumb compares to your predicted body temperature.	Use SPARK in a different mode of sampling to determine the temperature change of hot water as it cools in a plain cup versus a student-designed cup.

## Procedure with Inquiry

---

After you complete a step (or answer a question), place a check mark in the box (☐) next to that step.

### Part 1 – Manual sampling of body temperature

#### Set Up

1. ☐ What is the average internal temperature of the human body? If necessary, discuss this with your classmates or consult a textbook and write the answer in the space below.

\_\_\_\_\_ 37.0 \_\_\_\_\_ °C

2. ☐ Press and hold the power button on the bottom of the SPARK Science Learning System (SPARK) to turn it on.
3. ☐ Connect the fast-response temperature probe to the temperature port on the top of SPARK.

*Result:* After SPARK has finished booting up, the Home screen appears displaying the current temperature reading.

4. ☐ Write the room temperature in the space below.

\_\_\_\_\_ 23.4 \_\_\_\_\_ °C

5. ☐ What do you think the temperature of your thumb will be?

\_\_\_\_\_ 37.0 \_\_\_\_\_ °C

6. ☐ Place the end of the fast-response temperature probe on your thumb, and hold it in place with your forefinger. When the temperature reading stabilizes, write the temperature in the space below.

\_\_\_\_\_ 33.9 \_\_\_\_\_ °C

7. ☐ How did the temperature of your thumb compare to the value for average internal body temperature?

The temperature of my thumb was lower than normal internal body temperature.

8.  Why do you think the temperature of your thumb did not match the value for average internal body temperature? Below is a list of ideas that other students have generated. Add at least one additional explanation to the bottom of the list.
- A. Body temperature depends on the age of the person.
  - B. Body temperature is not constant, but changes based on the weather.
  - C. Body temperature is based on gender.
  - D. Body temperature changes with distance from the body core.
  - E. Body temperature depends on whether or not the body part is covered by clothing.

9.  This investigation will explore "student idea D". You will determine the temperature of body parts that get incrementally closer to the core of the body (thumb, hand, elbow, and armpit).

**Note:** Your teacher may allow you time later to design an experiment to test the other ideas.

10.  Predict how the temperature of a body part will be affected by its distance from the core of the body.

The temperature of body parts farthest from the core will have the lowest temperature, and the temperature will increase as it gets closer to the core body temperature.

11.  Create a table to display temperature.

**a.** Touch **BUILD**.

*Result:* The "Build your new page" screen opens.

**b.** In the measurements list on the left-hand side of the screen, touch **Temperature** to select it.

*Result:* The selected measurement is highlighted.

**c.** With **Temperature** highlighted, touch the **Table** button (.

*Result:* A table appears in the preview section of the "Build your new page" screen (in the upper right-hand corner of the screen).

**d.** Touch **OK**.

*Result:* A page is created with a table prepared to display temperature.

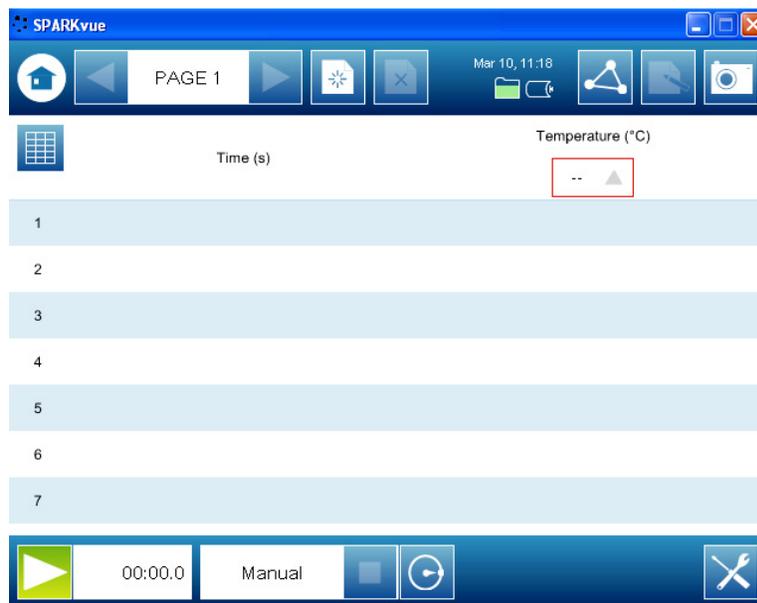
**Note:** SPARK automatically includes time as a column in the table as well, but it is not needed in this activity and can be ignored.

12. □ Put SPARK into manual sampling mode.

**Note:** SPARK is automatically set up to record temperature data from the fast-response temperature probe two times each second (periodic: 2 Hz). In this experiment, we only want to record one data point for each body part, and we need to tell SPARK when we are ready for the temperature to be recorded. This can be done by putting SPARK into manual mode, which will monitor and display the measurement but will not record any data points until the **Keep** button (  ) is pressed.

- a. Touch the **Sampling Options** button (  ) to open the Sampling Options screen.
- b. Touch **Manual**; then touch **OK**.

*Result:* SPARK is now in manual sampling mode. SPARK displays a table that looks like this:



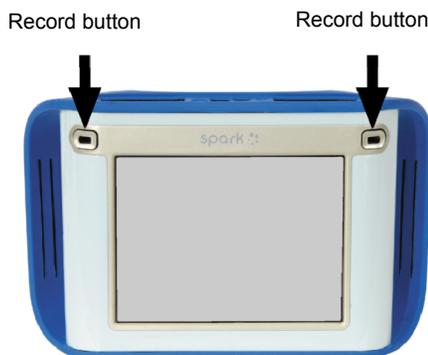
### Collect Data

13. □ To begin monitoring temperature, touch the **Start** button (  ).

*Result:* The Start button is replaced by the **Keep** button (  ), and blue lights flash indicating that the data run is in progress.

- 14.**  Place the temperature probe on your thumb, and hold it in position with your forefinger. Allow the temperature to stabilize, and then touch the **Keep** button (  ) to record the data point.

**Note:** Alternatively, you can press one of the flashing record buttons to record a data point.



- 15.**  Write the temperature for your thumb in Table 1 in the Data Analysis section of this worksheet.
- 16.**  Repeat data collection for the palm of your hand, crook of the elbow, and your armpit. In each case, hold the temperature probe in place using the tip of your forefinger. Touch the **Keep** button (  ) to record these temperatures on SPARK, and write your data in Table 1 in the Data Analysis section of this worksheet.
- 17.**  Touch the **Stop** button (  ) to complete data collection.
- 18.**  Save your work:
- Touch the **Sharing** button (  ) to open the Sharing screen.
  - Touch **SAVE FILE AS**.
  - Touch the **Name** box, enter a descriptive file name, and touch **OK**.
  - Touch **SAVE**.
  - Touch **DONE** to return to your table.
- 19.**  Complete the tasks for Part 1 of the Data Analysis section of this worksheet.

## Part 2 – Periodic sampling of water cooling

### Set Up – Run 1: Control

20. □ Predict what will happen to the temperature of warm water (~50 °C) that sits in a cup at room temperature for 5 minutes.

The temperature of the water will decrease.

21. □ To determine whether your prediction is correct you will run the experiment. To do this, start by setting up a graph display on SPARK.

- a. Touch the Home button () to return to the Home screen.

**Note:** If any changes have been made since last saving the file, SPARK will prompt you to save your previous work.

- b. On the Home screen, touch **BUILD**.

*Result:* The "Build your new page" screen opens.

- c. In the measurements list, touch **Temperature** to select it.

*Result:* The selected measurement is highlighted.

- d. With **Temperature** highlighted, touch the **Graph** button ()

*Result:* A graph appears in the preview section of the "Build your new page" screen.

- e. Touch **OK**.

*Result:* A page is created with a graph prepared to display temperature versus time.

22. □ Set the sampling rate on SPARK to record 1 data point each second (periodic: 1 seconds).

**Note:** The term "periodic" is used to describe the sampling mode that automatically records data, and the term "sampling rate" is used to describe how often the data will be recorded (once each second in this experiment). Taking many data points each second (high sampling rates) is needed when changes are expected to occur very quickly. Lower sampling rates are used when changes occur slowly or data is being taken for an extended period of time.

- a. Touch the **Sampling Options** button () to open the Sampling Options screen.

- b. Touch the **Sample Rate Unit** box and select **seconds**.

*Result:* The sample rate defaults to 1 second between samples.

- c. Touch **OK**.

23. □ What is sampling rate? What does it mean to have a sampling rate of 1 sample per second?

Sampling rate is the rate at which SPARK records the data you are studying. If the sampling rate is 1 per second, then SPARK is gathering data once every second.

24. □ Fill a cup two-thirds full of hot water (~50 °C).

### Collect Data – Run 1: Control

25. □ Place the tip of the fast-response temperature probe into the water. Hold the temperature probe in place or tape it to the cup so that it does not fall out of the cup during data collection.

26. □ Touch the **Start** button ().

*Result:* SPARK begins recording data.

**Note:** Alternatively, you can press one of the record buttons above the touch screen to start data recording. The blue lights will flash to indicate that data collection is in progress.

27. □ Adjust the scale of the graph to clearly see any changes in temperature:

a. Touch the **Tools** button () to open the tool palette.

b. Touch the **Scale-to-fit** button ().

c. Touch the **Tools** button () to close the tool palette.

28. □ Continue to collect data for 5 minutes (300 seconds).

29. □ When 5 minutes (300 seconds) have passed, touch the **Stop** button ().

**Note:** Alternatively, you can press one of the record buttons to stop data collection.

30. □ Label the data run:

a. Touch the **Tools** button to open the tool palette ().

b. Touch the **Select** button ().

*Result:* The Select button turns orange.

c. Touch a point on the plotted data run.

d. Touch **OK** (   ).

e. Touch the **Annotation** button ().

*Result:* The on-screen keyboard appears.

f. Type "Control" and touch **OK**.

- g.** Touch the orange **Select** button () to turn off the selection.
- h.** Touch the **Tools** button () to close the tool palette.

**31.**  Complete the tasks in the Part 2 – Control section of the Data Analysis section of this worksheet.

### **Set Up – Run 2: Student-designed cup**

**32.**  Using the materials provided by your teacher and the knowledge gained in the previous data run, modify the cup you used in the first trial so that it will keep the water as warm as possible when it is left at room temperature for 5 minutes.

**33.**  Describe the materials you used to modify the cup.

I wrapped the cup in cotton cloth.

**34.**  How will you know if the cup you design holds heat better than the cup by itself?

I will perform the same experiment that I did with the normal cup and compare the results. For this example, if my cup holds heat better, then the change in temperature will be less than 4.3 °C.

**35.**  Fill your specially designed cup two-thirds full of hot water (~50 °C).

### **Collect Data – Run 2: Student-designed cup**

**36.**  Place the fast-response temperature probe into the water. Hold the temperature probe in place or tape it to the cup so it does not fall out of the cup during data collection.

**37.**  Touch the **Start** button () to begin data collection.

**38.**  Does the modification to the cup seem to be keeping the water warmer? Explain how you know.

The temperature of the water seems to be dropping more slowly than the control run.

**39.**  After 5 minutes (300 seconds) have passed, touch the **Stop** button ()

**40.**  Label the data run with a word or phrase that identifies the change to the cup.

**41.**  Complete the tasks for the Part 2 – Student-designed cup section of the Data Analysis section of this worksheet.

42.  Save your work:

- a. Touch the **Sharing** button () to open the Sharing screen.
- b. Touch **SAVE FILE AS**.
- c. Touch the **Name** box, enter a file name, and touch **OK**.
- d. Touch **SAVE**.
- e. Touch **DONE**.

## Data Analysis

### Part 1 – Manual sampling of body temperature

Table 1: Manual sampling of body temperature

Data Point	Body Part	Temperature (°C)
1	Thumb	28.7
2	Hand	30.7
3	Crook of elbow	34.4
4	Armpit	35.2

1.  Do you see any trends in the data that you collected? Explain any trends you observed in the space below.

Yes, the body temperature increases as you get closer to the body core.

2.  Does your prediction about body temperature match the data that you collected? Explain below.

Yes, my predictions were correct. Recorded body temperature was higher at the armpit than in the hand or thumb. It could be that the hands and thumbs are more exposed to ambient temperature; therefore, they are cooler than body temperature. Also, warm blood, traveling from the core to the extremities loses heat to the environment; therefore, hands are colder than the body core.

### Part 2 – Periodic sampling of water cooling

#### Run 1: Control

3.  Was your prediction about what will happen to the temperature of warm water (~50 °C) that sits in a paper cup at room temperature for 5 minutes supported by this experiment?

Yes, my prediction was supported. I predicted that the temperature of the water would decrease, and it did decrease.

4.  Find the maximum temperature and the minimum temperature of the water.

a. Touch the **Tools** button () to open the tool palette.

b. Touch the **Statistics** button ().

*Result:* The Statistics screen opens.

c. Touch **Minimum** and then touch **Maximum** to select both of them.

*Result:* Selected statistics are highlighted.

d. Touch **OK**.

*Result:* The minimum and maximum values of the data run appear on the graph.

e. Touch the **Tools** button () to close the tool palette.

5.  Record the maximum and minimum temperatures in Table 2.

6.  Calculate the change in temperature of the water in the cup during the 5 minutes, and record it in Table 2.

**Note:** Change in temperature = maximum temperature – minimum temperature.

Change in temperature =  $49.4\text{ }^{\circ}\text{C} - 45.1\text{ }^{\circ}\text{C} = 4.3\text{ }^{\circ}\text{C}$ .

7.  Explain why this temperature change was observed.

The temperature of the warm water decreased over time because the temperature of the room was cooler. Heat moves from hot objects to cold objects, so the heat from the warm water was slowly being transferred to the air.

### **Run 2: Student-designed cup**

8.  Record a description of your design for cup insulation in Table 2.

9.  Find the maximum temperature and minimum temperature of the water, and record it in Table 2.

10. □ Calculate the change in temperature of the water in the cup during the 5 minutes, and record it in Table 2.

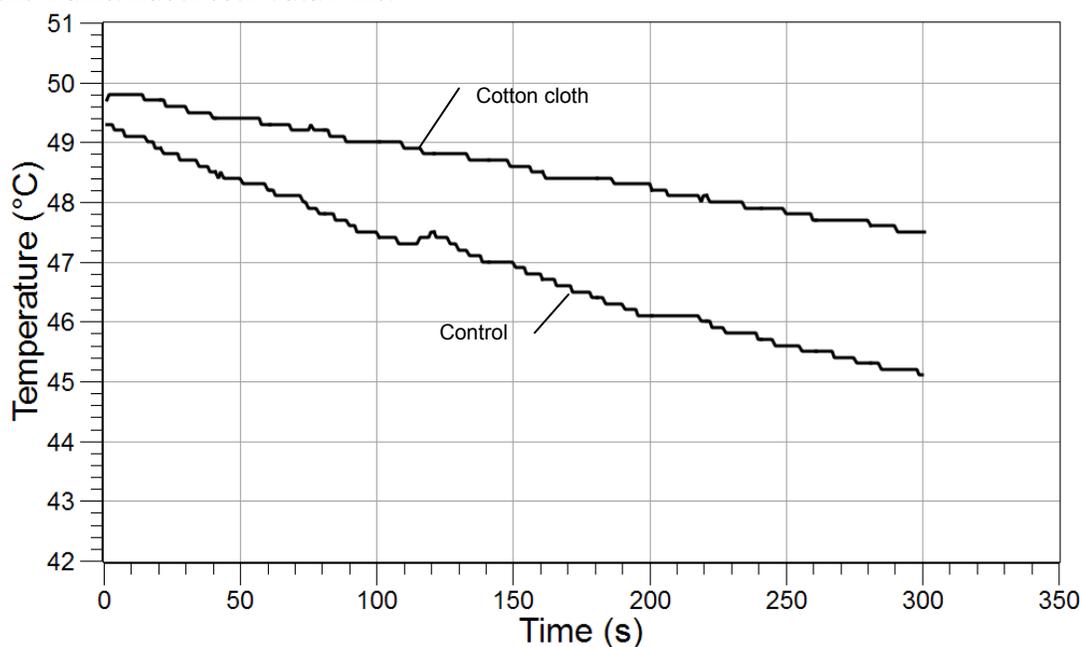
**Note:** Change in temperature = maximum temperature – minimum temperature.

Change in temperature =  $49.8\text{ }^{\circ}\text{C} - 47.5\text{ }^{\circ}\text{C} = 2.3\text{ }^{\circ}\text{C}$ .

Table 2: Effects of student cup modifications on change in temperature of water

Type of Cup	Describe the Cup Insulation Design	Maximum Temperature (°C)	Minimum Temperature (°C)	Change in Temperature (°C)
Plain cup		49.4	45.1	4.3
Student-designed cup	Wrapped the cup in cotton cloth	49.8	47.5	2.3

11. □ Sketch a graph of both data runs showing the Temperature on the y-axis and Time on the x-axis. Label both data runs.



## Analysis Questions

**1. How did your specially designed cup maintain the initial heat of the hot water compared to the plain cup? What evidence do you have to support your claim?**

The cup surrounded by cotton was more effective at maintaining the heat of the warm water than the plain cup. After sitting at room temperature for 5 minutes, the temperature of the water in the plain cup decreased 4.3 °C, whereas the temperature of the water in the cup wrapped in cotton decreased by only 2.3 °C.

**2. Can you be sure that your modification of the cup was the only reason for the change in temperature? Name two probable sources of error for this experiment.**

No, I cannot be sure that the insulation was the only reason for change in temperature. Some possible sources of error include the volume of water in the cup, which was not quantified, and the initial temperature of the water, which was not constant.

**3. How did SPARK technology improve the data collection and analysis you performed in the periodic sampling of water cooling part of the lab?**

SPARK technology was able to collect an accurate temperature value every second, which would be very difficult using a normal thermometer. SPARK technology also created the graph immediately so that the data could be analyzed immediately.

## Synthesis Questions

Use available resources to help you answer the following questions.

**1. Imagine that you just designed the most cost-effective insulated cup in the world! Companies everywhere will want to buy your design. Do you feel comfortable selling this design today? If not, what further steps would you take to ensure confidence in your design?**

After one test, you could not be confident in the design of your cup. Extensive testing should be done to ensure that your results are accurate and repeatable. Other scientists should test your hypothesis to ensure that the design works in other labs under other environmental conditions.

**2. Independent variables are those for which conditions are set by the experimenter. Dependent variables are those for which values depend upon the values of the independent variables. Identify the independent variable and dependent variable used in each part of the lab.**

	Independent Variable	Dependent Variable
Part 1: Body Temperature	body part	temperature
Part 2: Water Cooling	type of cup (plain cup versus student-designed cup)	temperature change

## Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

- 1. Which of the following is not a part of the inquiry process?**
  - A. Making observations
  - B. Posing hypotheses
  - C. Analyzing the data collected
  - D. Making sure your results support your hypothesis**
  
- 2. Why is it necessary to use a consistent technique while collecting data?**
  - A. To control the variables**
  - B. To change as many variables as you can
  - C. To make sure your results support your hypothesis
  - D. None of the above
  
- 3. How would you keep a cup of ice water cold all day?**
  - A. Leave the cup in a dark place
  - B. Make sure the cup does not have a lid
  - C. Wrap the cup in layers of newspaper**
  - D. Stir the ice water frequently

## Extended Inquiry Suggestions

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Instruct students to compare their data with that of other groups to determine which group designed the most effective insulation for the cup. Be sure to compare the changes in temperature across groups, not just minimum temperatures.

Have the students design their own experiment to research.

Let students design and conduct an experiment that explores the effects of activity on body temperature.



## Scientific Inquiry – SPARKvue™

### Objectives

This lab is designed to help students familiarize themselves with SPARKvue™ while engaging in scientific investigations. By completing these exercises students learn to:

- ◆ Engage in the process of scientific inquiry by making predictions, collecting and analyzing data, and explaining observations
- ◆ Use the most popular and effective SPARKvue tools

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Setting up SPARKvue to collect data in different types of experiments
- ◆ Using the manual and periodic sampling modes of SPARKvue
- ◆ Using the fast-response temperature probe with SPARKvue to collect temperatures of various body parts in a table display
- ◆ Using the fast-response temperature probe and the graph display to continuously record and display the temperature of hot water that is left to cool in an insulated and non-insulated cup at room temperature for 5 minutes
- ◆ Designing a way to insulate a cup so it keeps hot water warm for as long as possible

### Time Requirement

◆ Preparation time	5 minutes
◆ Pre-lab discussion and activity	5 minutes
◆ Lab activity	40 minutes

### Materials and Equipment

#### *For each student or group:*

- ◆ Computer with SPARKvue software
- ◆ PASPORT sensor interface<sup>1</sup>
- ◆ PASPORT temperature sensor
- ◆ Fast-response temperature probe
- ◆ Cup<sup>2</sup>, 9- to 12-oz
- ◆ Materials available in the laboratory to design a cup that keeps water hot<sup>3</sup>
- ◆ Hot water, 16 oz

<sup>1</sup>Use a PASPORT USB Link, SPARKLink, PASPORT PowerLink, PASPORT AirLink SI, PASPORT Xplorer, or PASPORT Xplorer GLX

<sup>2</sup>Use a non-insulated plastic or paper cup.

<sup>3</sup>For example: polystyrene, cloth, foil, plastic wrap, wool, water baths, or packing peanuts

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Scientists in the 21st century increasingly rely on technology to enhance data collection and analysis. Technology enables scientists to collect large amounts of data accurately and to display, analyze, and interpret their data more quickly.

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This investigation includes detailed steps on how to set up SPARKvue to collect data in different types of experiments. In the first part of this investigation, students find the temperature of four different body parts. To do this, the student will need to place the temperature probe on the body part and allow the temperature to stabilize. When ready, the student will trigger SPARKvue to record the data point. SPARKvue allows for this type of data collection in manual sampling mode. Manual sampling mode allows users to see the data value as it changes over time and to record a data point when they are ready.

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This combination of methods and tools can be a powerful stimulation for students to develop their own scientific inquiries arising from their natural curiosity about the world around them.

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These are the materials and equipment to set up prior to the lab.

1. Make cups and insulating material available to each student group. Use what you have on hand as possible insulating materials. Some suggestions include: polystyrene cups, cloth, foil, plastic wrap, wool, water baths, or packing peanuts.
2. Make sure that a source of hot water with a constant temperature (~50 °C) is available for your students throughout the activity.

## Safety

---

Add these important safety precautions to your normal laboratory procedures:

- ◆ Do not use the fast-response temperature probe in water that is 70 °C or hotter.
- ◆ Be careful when working with hot water.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

3	1	5	2	4
Next, carry out a controlled experiment to determine whether body temperature is the same for different parts of your body.	Set up SPARKvue, and connect the temperature sensor and fast-response temperature probe to the computer using a sensor interface.	Analyze the effectiveness of the cup you designed to slow the rate at which warm water cools.	First, determine how the temperature of your thumb compares to your predicted body temperature.	Use SPARKvue in a different mode of sampling to determine the temperature change of hot water as it cools in a plain cup versus a student-designed cup.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

### Part 1 – Manual sampling of body temperature

#### Set Up

1.  What is the average internal temperature of the human body? If necessary, discuss this with your classmates or consult a textbook and write the answer in the space below.

\_\_\_\_\_ 37.0 \_\_\_\_\_ °C

2.  With the computer running, double-click the SPARKvue icon to launch SPARKvue.
3.  Connect the fast response temperature probe to the PASPORT temperature sensor, the PASPORT temperature sensor to the PASPORT interface, and the PASPORT interface to the computer.

*Result:* The Home screen appears displaying the current temperature reading.

4.  Write the room temperature in the space below.

\_\_\_\_\_ 23.4 \_\_\_\_\_ °C

5.  What do you think the temperature of your thumb will be?

\_\_\_\_\_ 37.0 \_\_\_\_\_ °C

6.  Place the end of the fast-response temperature probe on your thumb, and hold it in place with your forefinger. When the temperature reading stabilizes, write the temperature in the space below.

\_\_\_\_\_ 33.9 \_\_\_\_\_ °C

7.  How did the temperature of your thumb compare to the value for average internal body temperature?

The temperature of my thumb was lower than normal internal body temperature.

8.  Why do you think the temperature of your thumb did not match the value for average internal body temperature? Below is a list of ideas that other students have generated. Add at least one additional explanation to the bottom of the list.
- A. Body temperature depends on the age of the person.
  - B. Body temperature is not constant, but changes based on the weather.
  - C. Body temperature is based on gender.
  - D. Body temperature changes with distance from the body core.
  - E. Body temperature depends on whether or not the body part is covered by clothing.

9.  This investigation will explore "student idea D". You will determine the temperature of body parts that get incrementally closer to the core of the body (thumb, hand, elbow, and armpit).

**Note:** Your teacher may allow you time later to design an experiment to test the other ideas.

10.  Predict how the temperature of a body part will be affected by its distance from the core of the body.

The temperature of body parts farthest from the core will have the lowest temperature, and the temperature will increase as it gets closer to the core body temperature.

11.  Create a table to display temperature.

- a. Click **BUILD**.

*Result:* The "Build your new page" screen opens.

- b. In the measurements list on the left-hand side of the screen, click **Temperature** to select it.

*Result:* The selected measurement is highlighted.

- c. With **Temperature** highlighted, click the **Table** button (.

*Result:* A table appears in the preview section of the "Build your new page" screen (in the upper right-hand corner of the screen).

- d. Click **OK**.

*Result:* A page is created with a table prepared to display temperature.

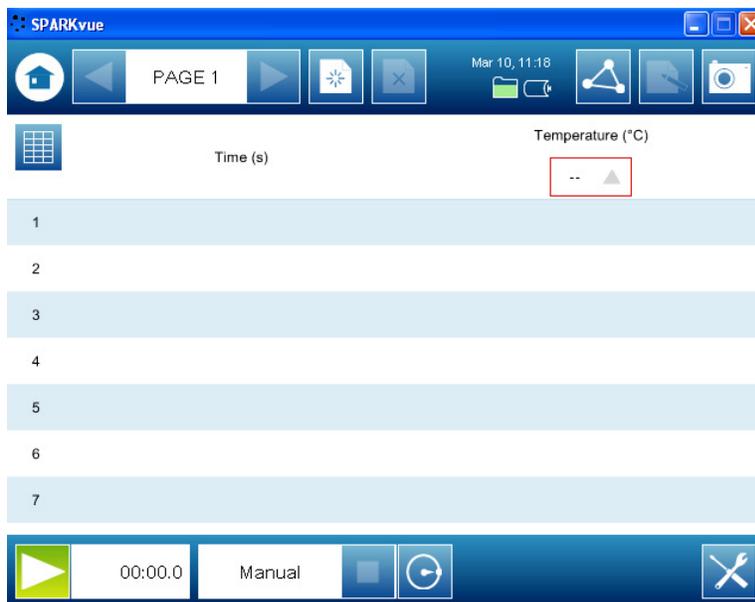
**Note:** SPARKvue automatically includes time as a column in the table as well, but it is not needed in this activity and can be ignored.

- 12. □ Put SPARKvue into manual sampling mode.

**Note:** SPARKvue is automatically set up to record temperature data from the fast-response temperature sensor two times each second (periodic: 2 Hz). In this experiment, we only want to record one data point for each body part, and we need to tell SPARKvue when we are ready for the temperature to be recorded. This can be done by putting SPARKvue into manual mode, which will monitor and display the measurement but will not record any data points until the **Keep** button (  ) is clicked.

- a. Click the **Sampling Options** button (  ) to open the Sampling Options screen.
- b. Click **Manual**; then click **OK**.

*Result:* SPARKvue is now in manual sampling mode. SPARKvue displays a table that looks like this:



### Collect Data

- 13. □ To begin monitoring temperature, click the **Start** button (  ).

*Result:* The Start button is replaced by the **Keep** button (  ), and blue lights flash indicating that the data run is in progress.

- 14.**  Place the temperature probe on your thumb, and hold it in position with your forefinger. Allow the temperature to stabilize, and then click the **Keep** button () to record the data point.
- 15.**  Write the temperature for your thumb in Table 1 in the Data Analysis section of this worksheet.
- 16.**  Repeat data collection for the palm of your hand, crook of the elbow, and your armpit. In each case, hold the temperature probe in place using the tip of your forefinger. Click the **Keep** button () to record these temperatures on SPARKvue, and write your data in Table 1 in the Data Analysis section of this worksheet.
- 17.**  Click the **Stop** button () to complete data collection.
- 18.**  Save your work:
- Click the **Sharing** button () to open the Sharing screen.
  - Click **SAVE FILE AS**.
- Result:* The Save As window opens.
- Navigate to a folder where you would like to save the file.
  - Enter a filename.
  - Click **SAVE**.
  - Click **DONE** to close the sharing screen.
- 19.**  Complete the tasks for Part 1 of the Data Analysis section of this worksheet.

## Part 2 – Periodic sampling of water cooling

### Set Up – Run 1: Control

- 20.**  Predict what will happen to the temperature of warm water (~50 °C) that sits in a cup at room temperature for 5 minutes.

The temperature of the water will decrease.

- 21.**  To determine whether your prediction is correct you will run the experiment. To do this, start by setting up a graph display on SPARKvue.
- Click the Home button () to return to the Home screen.

**Note:** If any changes have been made since last saving the file, SPARKvue will prompt you to save your previous work.

- b.** On the Home screen, click **BUILD**.

*Result:* The "Build your new page" screen opens.

- c.** In the measurements list, click **Temperature** to select it.

*Result:* The selected measurement is highlighted.

- d.** With **Temperature** highlighted, click the **Graph** button ().

*Result:* A graph appears in the preview section of the "Build your new page" screen.

- e.** Click **OK**.

*Result:* A page is created with a graph prepared to display temperature versus time.

- 22.**  Set the sampling rate on SPARKvue to record 1 data point each second (periodic: 1 seconds).

**Note:** The term "periodic" is used to describe the sampling mode that automatically records data, and the term "sampling rate" is used to describe how often the data will be recorded (once each second in this experiment). Taking many data points each second (high sampling rates) is needed when changes are expected to occur very quickly. Lower sampling rates are used when changes occur slowly or data is being taken for an extended period of time.

- a.** Click the **Sampling Options** button () to open the Sampling Options screen.

- b.** Click the **Sample Rate Unit** box and select **seconds**.

*Result:* The sample rate defaults to 1 second between samples.

- c.** Click **OK**.

- 23.**  What is sampling rate? What does it mean to have a sampling rate of 1 sample per second?

Sampling rate is the rate at which SPARKvue records the data you are studying. If the sampling rate is 1 per second, then SPARKvue is gathering data once every second.

- 24.**  Fill a cup two-thirds full of hot water (~50 °C).

### **Collect Data – Run 1: Control**

- 25.**  Place the tip of the fast-response temperature probe into the water. Hold the temperature probe in place or tape it to the cup so that it does not fall out of the cup during data collection.

- 26.**  Click the **Start** button (.

*Result:* SPARKvue begins recording data.

- 27.**  Adjust the scale of the graph to clearly see any changes in temperature:
- Click the **Tools** button () to open the tool palette.
  - Click the **Scale-to-fit** button ()
  - Click the **Tools** button () to close the tool palette.
- 28.**  Continue to collect data for 5 minutes (300 seconds).
- 29.**  When 5 minutes (300 seconds) have passed, click the **Stop** button ()

**Note:** Alternatively, you can press one of the record buttons to stop data collection.

- 30.**  Label the data run:
- Click the **Tools** button to open the tool palette ()
  - Click the **Select** button ()
- Result:* The Select button turns orange.
- Click a point on the plotted data run.
  - Click **OK** (  ).
  - Click the **Annotation** button ()
- Result:* The on-screen keyboard appears.
- Type "Control" and click **OK**.
  - Click the orange **Select** button () to turn off the selection.
  - Click the **Tools** button () to close the tool palette.

- 31.**  Complete the tasks in the Part 2 – Control section of the Data Analysis section of this worksheet.

### **Set Up – Run 2: Student-designed cup**

- 32.**  Using the materials provided by your teacher and the knowledge gained in the previous data run, modify the cup you used in the first trial so that it will keep the water as warm as possible when it is left at room temperature for 5 minutes.
- 33.**  Describe the materials you used to modify the cup.

I wrapped the cup in cotton cloth.

34. □ How will you know if the cup you design holds heat better than the cup by itself?

I will perform the same experiment that I did with the normal cup and compare the results. For this example, if my cup holds heat better, then the change in temperature will be less than 4.3 °C.

35. □ Fill your specially designed cup two-thirds full of hot water (~50 °C).

### **Collect Data – Run 2: Student-designed cup**

36. □ Place the fast-response temperature probe into the water. Hold the temperature probe in place or tape it to the cup so it does not fall out of the cup during data collection.

37. □ Click the **Start** button () to begin data collection.

38. □ Does the modification to the cup seem to be keeping the water warmer? Explain how you know.

The temperature of the water seems to be dropping more slowly than the control run.

39. □ After 5 minutes (300 seconds) have passed, click the **Stop** button ()

40. □ Label the data run with a word or phrase that identifies the change to the cup.

41. □ Complete the tasks for the Part 2 – Student-designed cup section of the Data Analysis section of this worksheet.

42. □ Save your work:

- a. Click the **Sharing** button () to open the Sharing screen.

- b. Click **SAVE FILE AS**.

*Result:* The Save As window opens.

- c. Navigate to a folder where you would like to save the file.

- d. Enter a filename.

- e. Click **SAVE**.

- f. Click **DONE**.

## Data Analysis

### Part 1 – Manual sampling of body temperature

Table 1: Manual sampling of body temperature

Data Point	Body Part	Temperature (°C)
1	Thumb	28.7
2	Hand	30.7
3	Crook of elbow	34.4
4	Armpit	35.2

1.  Do you see any trends in the data that you collected? Explain any trends you observed in the space below.

Yes, the body temperature increases as you get closer to the body core.

2.  Does your prediction about body temperature match the data that you collected? Explain below.

Yes, my predictions were correct. Recorded body temperature was higher at the armpit than in the hand or thumb. It could be that the hands and thumbs are more exposed to ambient temperature; therefore, they are cooler than body temperature. Also, warm blood, traveling from the core to the extremities loses heat to the environment; therefore, hands are colder than the body core.

### Part 2 – Periodic sampling of water cooling

#### Run 1: Control

3.  Was your prediction about what will happen to the temperature of warm water (~50 °C) that sits in a paper cup at room temperature for 5 minutes supported by this experiment?

Yes, my prediction was supported. I predicted that the temperature of the water would decrease, and it did decrease.

4.  Find the maximum temperature and the minimum temperature of the water.

a. Click the **Tools** button () to open the tool palette.

b. Click the **Statistics** button ()

*Result:* The Statistics screen opens.

c. Click **Minimum** and then click **Maximum** to select both of them.

*Result:* Selected statistics are highlighted.

d. Click **OK**.

*Result:* The minimum and maximum values of the data run appear on the graph.

e. Click the **Tools** button () to close the tool palette.

5.  Record the maximum and minimum temperatures in Table 2.

6.  Calculate the change in temperature of the water in the cup during the 5 minutes, and record it in Table 2.

**Note:** Change in temperature = maximum temperature – minimum temperature.

Change in temperature = 49.4 °C – 45.1 °C = 4.3 °C.

7.  Explain why this temperature change was observed.

The temperature of the warm water decreased over time because the temperature of the room was cooler. Heat moves from hot objects to cold objects, so the heat from the warm water was slowly being transferred to the air.

**Run 2: Student-designed cup**

8.  Record a description of your design for cup insulation in Table 2.

9.  Find the maximum temperature and minimum temperature of the water, and record it in Table 2.

10.  Calculate the change in temperature of the water in the cup during the 5 minutes, and record it in Table 2.

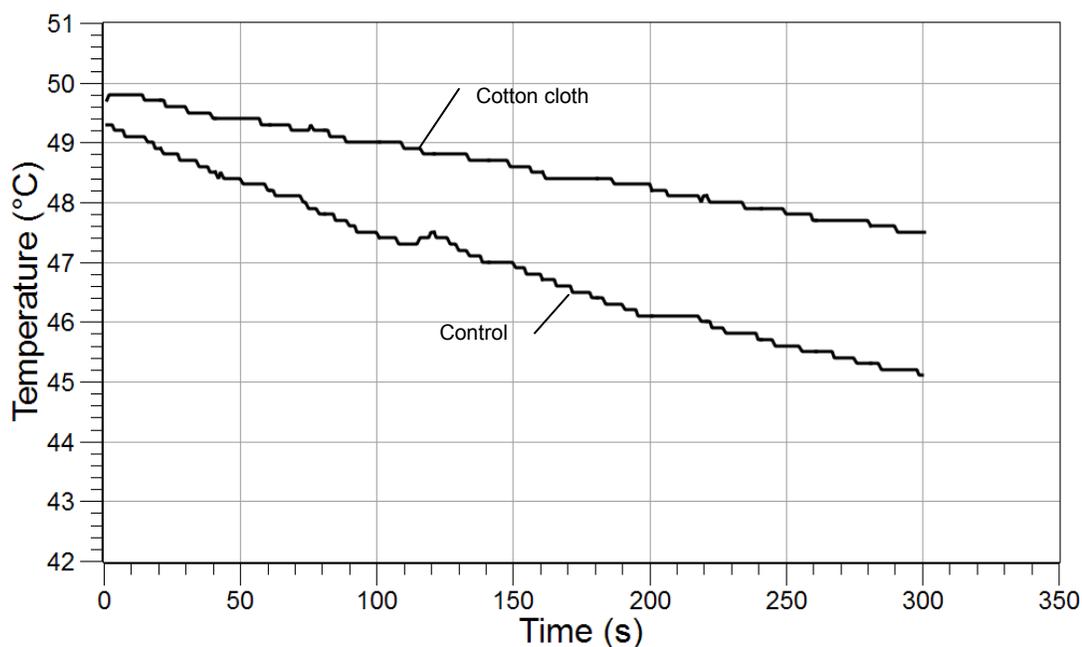
**Note:** Change in temperature = maximum temperature – minimum temperature.

Change in temperature = 49.8 °C – 47.5 °C = 2.3 °C.

Table 2: Effects of student cup modifications on change in temperature of water

Type of Cup	Describe the Cup Insulation Design	Maximum Temperature (°C)	Minimum Temperature (°C)	Change in Temperature (°C)
Plain cup		49.4	45.1	4.3
Student-designed cup	Wrapped the cup in cotton cloth	49.8	47.5	2.3

11. □ Sketch a graph of both data runs showing the Temperature on the y-axis and Time on the x-axis. Label both data runs.



### Analysis Questions

- 1. How did your specially designed cup maintain the initial heat of the hot water compared to the plain cup? What evidence do you have to support your claim?**

The cup surrounded by cotton was more effective at maintaining the heat of the warm water than the plain cup. After sitting at room temperature for 5 minutes, the temperature of the water in the plain cup decreased 4.3 °C, whereas the temperature of the water in the cup wrapped in cotton decreased by only 2.3 °C.

- 2. Can you be sure that your modification of the cup was the only reason for the change in temperature? Name two probable sources of error for this experiment.**

No, I cannot be sure that the insulation was the only reason for change in temperature. Some possible sources of error include the volume of water in the cup, which was not quantified, and the initial temperature of the water, which was not constant.

- 3. How did SPARKvue and PASPORT technology improve the data collection and analysis you performed in the periodic sampling of water cooling part of the lab?**

SPARKvue and PASPORT technology was able to collect an accurate temperature value every second, which would be very difficult using a normal thermometer. SPARKvue technology also created the graph immediately so that the data could be analyzed immediately.

## Synthesis Questions

Use available resources to help you answer the following questions.

**1. Imagine that you just designed the most cost-effective insulated cup in the world! Companies everywhere will want to buy your design. Do you feel comfortable selling this design today? If not, what further steps would you take to ensure confidence in your design?**

After one test, you could not be confident in the design of your cup. Extensive testing should be done to ensure that your results are accurate and repeatable. Other scientists should test your hypothesis to ensure that the design works in other labs under other environmental conditions.

**2. Independent variables are those for which conditions are set by the experimenter. Dependent variables are those for which values depend upon the values of the independent variables. Identify the independent variable and dependent variable used in each part of the lab.**

	Independent Variable	Dependent Variable
Part 1: Body Temperature	body part	temperature
Part 2: Water Cooling	type of cup (plain cup versus student-designed cup)	temperature change

## Multiple Choice Questions

Select the best answer or completion to each of the questions or incomplete statements below.

**1. Which of the following is not a part of the inquiry process?**

- A.** Making observations
- B.** Posing hypotheses
- C.** Analyzing the data collected
- D.** Making sure your results support your hypothesis

**2. Why is it necessary to use a consistent technique while collecting data?**

- A.** To control the variables
- B.** To change as many variables as you can
- C.** To make sure your results support your hypothesis
- D.** None of the above

**3. How would you keep a cup of ice water cold all day?**

- A.** Leave the cup in a dark place
- B.** Make sure the cup does not have a lid
- C.** Wrap the cup in layers of newspaper
- D.** Stir the ice water frequently

### **Extended Inquiry Suggestions**

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Instruct students to compare their data with that of other groups to determine which group designed the most effective insulation for the cup. Be sure to compare the changes in temperature across groups, not just minimum temperatures.

Have the students design their own experiment to research.

Let students design and conduct an experiment that explores the effects of activity on body temperature



## Scientific Inquiry – Xplorer GLX®

### Objectives

This lab is designed to help students familiarize themselves with the Xplorer GLX® (GLX) while engaging in scientific investigations. By completing these exercises students learn to:

- ◆ Engage in the process of scientific inquiry by making predictions, collecting and analyzing data, and explaining observations
- ◆ Use the most popular and effective GLX tools

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Setting up the Xplorer GLX (GLX) to collect data in different types of experiments
- ◆ Using the manual and periodic sampling modes of GLX
- ◆ Using the fast-response temperature probe with GLX to collect temperatures of various body parts in a table display
- ◆ Using the fast-response temperature probe and the graph display to continuously record and display the temperature of hot water that is left to cool in an insulated and non-insulated cup at room temperature for 5 minutes
- ◆ Designing a way to insulate a cup so it keeps hot water warm for as long as possible

### Time Requirement

◆ Preparation time	5 minutes
◆ Pre-lab discussion and activity	5 minutes
◆ Lab activity	40 minutes

### Materials and Equipment

#### *For each student or group:*

- ◆ Xplorer GLX
- ◆ Fast-response temperature probe
- ◆ Cup<sup>1</sup>, 9- to 12-oz
- ◆ Materials available in the laboratory to design a cup that keeps water hot<sup>2</sup>
- ◆ Hot water, 16 oz

<sup>1</sup>Use a non-insulated plastic or paper cup.

<sup>2</sup>For example: polystyrene, cloth, foil, plastic wrap, wool, water baths, or packing peanuts

## **Background**

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### **The Scientific Method and Scientific Inquiry**

Scientific inquiry is a general term for the numerous ways in which scientists learn about the natural world to produce scientific knowledge. The knowledge produced is supported by evidence collected during the inquiry process.

Traditionally, the term scientific method has been used when teaching students how scientists learn about the natural world. However, this term has recently been criticized because it creates the misconception that all scientists follow the same steps (processes) in the same order all the time. In reality, the many methods used to investigate nature in the various sciences are better represented by a broader term, scientific inquiry.

Although the sequence and choice of steps used varies from scientist to scientist and from one investigation to the next, a common set of processes is used by scientists to learn about the natural world. Making observations; asking questions; making predictions or posing hypotheses; planning investigations; controlling variables; using systematic, reliable, repeatable, and externally validated techniques; analyzing the data collected; and interpreting and communicating results are among the major processes that are involved in scientific inquiry.

Each lab in this manual is designed to guide students through a sequence of scientific inquiry processes that may be used to learn about the world around them. Students learn about scientific inquiry by engaging in inquiry processes, and they develop the skills needed to answer their own questions through scientific inquiry. At the same time, students gain content knowledge by actively engaging in scientific inquiry as opposed to being told about or reading about science.

Scientists in the 21st century increasingly rely on technology to enhance data collection and analysis. Technology enables scientists to collect large amounts of data accurately and to display, analyze, and interpret their data more quickly.

### **Using the Xplorer GLX**

This investigation includes detailed steps on how to set up the Xplorer GLX (GLX) to collect data in different types of experiments. In the first part of this investigation, students find the temperature of four different body parts. To do this, the student will need to place the temperature probe on the body part and allow the temperature to stabilize. When ready, the student will trigger the GLX to record the data point. The GLX allows for this type of data collection in manual sampling mode. Manual sampling mode allows users to see the data value as it changes over time and to record a data point when they are ready.

In the second part of this investigation, students measure temperature values each second as a cup of hot water cools. The GLX allows for a measurement to be automatically recorded at a specified rate. This type of data collection is called periodic sampling (this is the default mode). Taking many data points each second (high sampling rates) is needed when changes are expected to occur very quickly. Lower sampling rates are used when changes occur slowly or data is being taken for an extended period of time. The default sample rate for each PASCO sensor is appropriate for the most typical applications of that sensor, reducing the need for the user to change the sample rate before each experiment.

This investigation also guides students in two different ways to display data (table and graph) and how to start and stop data collection, open and close tool palettes, perform simple analyses (statistics), and save data.

This combination of methods and tools can be a powerful stimulation for students to develop their own scientific inquiries arising from their natural curiosity about the world around them.

## Pre-Lab Discussion

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Lead a discussion with your students about what science is and what processes scientists use to learn about the world around them. Lead the discussion with questions such as “What is science?” and “What do scientists do?” Students will probably start out with specifics about what different scientists do. Let the ideas emerge, and see if you can find a common thread within the ideas. Ask students questions about different types of technologies scientists use and how these technologies help them in the different processes.

**Teacher Tip:** Accept all answers and write ideas on the board or overhead projector to remain displayed during the activity.

Ideally, you can lead your students to the conclusion that science is “asking questions, making predictions (constructing hypotheses), conducting experiments, collecting data (gathering evidence), analyzing the data, and drawing conclusions.” Let your students know that this is exactly what they will be doing in this laboratory exercise and throughout the year in your class.

## Lab Preparation

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These are the materials and equipment to set up prior to the lab.

1. Make cups and insulating material available to each student group. Use what you have on hand as possible insulating materials. Some suggestions include: polystyrene cups, cloth, foil, plastic wrap, wool, water baths, or packing peanuts.
2. Make sure that a source of hot water with a constant temperature (~50 °C) is available for your students throughout the activity.

## Safety

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Add these important safety precautions to your normal laboratory procedures:

- ◆ Do not use the fast-response temperature probe in water that is 70 °C or hotter.
- ◆ Be careful when working with hot water.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

3	1	5	2	4
Next, carry out a controlled experiment to determine whether body temperature is the same for different parts of your body.	Turn on the Xplorer GLX, and connect the fast-response temperature probe.	Analyze the effectiveness of the cup you designed to slow the rate at which warm water cools.	First, determine how the temperature of your thumb compares to your predicted body temperature.	Use the GLX in a different mode of sampling to determine the temperature change of hot water as it cools in a plain cup versus a student-designed cup.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

### Part 1 – Manual sampling of body temperature

#### Set Up

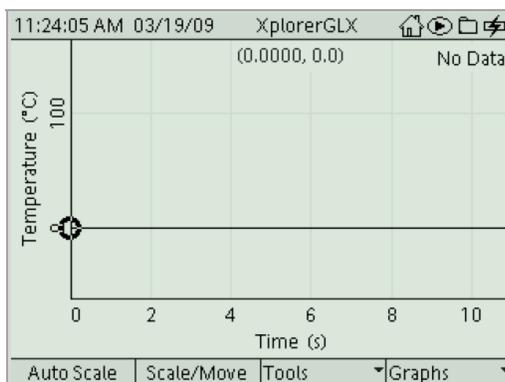
1.  What is the average internal temperature of the human body? If necessary, discuss this with your classmates or consult a textbook and write the answer in the space below.

\_\_\_\_\_ 37.0 \_\_\_\_\_ °C

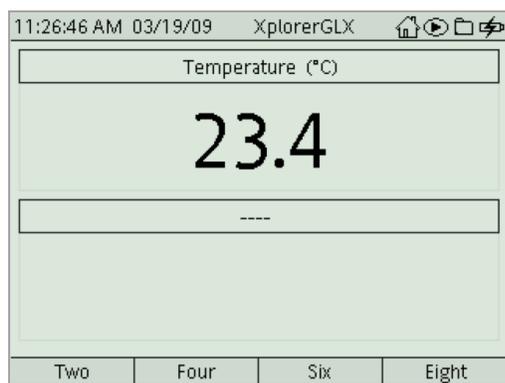
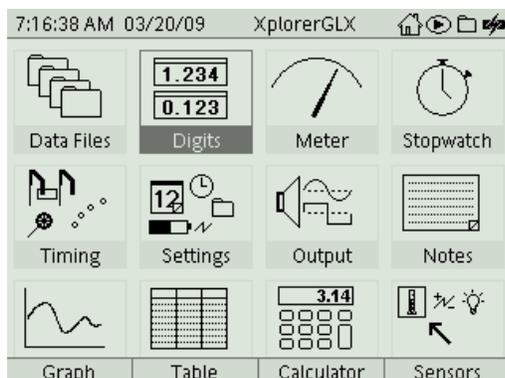
2.  Press and hold the power button (  ) on the bottom right, of the Xplorer GLX to turn it on.

3.  Connect the fast-response temperature probe to the temperature port on the side of the GLX.

*Result:* A graph display will automatically be created, displaying temperature vs. time.



4.  Press the **Home** button (  ). From the home screen, use the arrow keys to select **Digits** and press  .



5.  Write the room temperature in the space below.

23.4 °C

6.  What do you think the temperature of your thumb will be?

37.0 °C

7.  Place the end of the fast-response temperature probe on your thumb, and hold it in place with your forefinger. When the temperature reading stabilizes, write the temperature in the space below.

33.9 °C

8.  How did the temperature of your thumb compare to the value for average internal body temperature?

The temperature of my thumb was lower than normal internal body temperature.

9.  Why do you think the temperature of your thumb did not match the value for average internal body temperature? Below is a list of ideas that other students have generated. Add at least one additional explanation to the bottom of the list.

- A. Body temperature depends on the age of the person.
- B. Body temperature is not constant, but changes based on the weather.
- C. Body temperature is based on gender.
- D. Body temperature changes with distance from the body core.
- E. Body temperature depends on whether or not the body part is covered by clothing.

10.  This investigation will explore "student idea D". You will determine the temperature of body parts that get incrementally closer to the core of the body (thumb, hand, elbow, and armpit).

**Note:** Your teacher may allow you time later to design an experiment to test the other ideas.

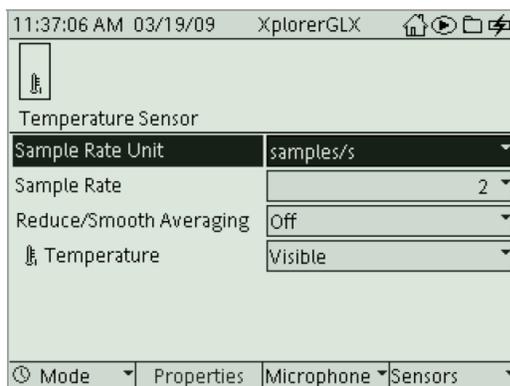
11.  Predict how the temperature of a body part will be affected by its distance from the core of the body.

The temperature of body parts farthest from the core will have the lowest temperature and the temperature will increase as it gets closer to the core body temperature.

12. □ Put the GLX into manual sampling mode.

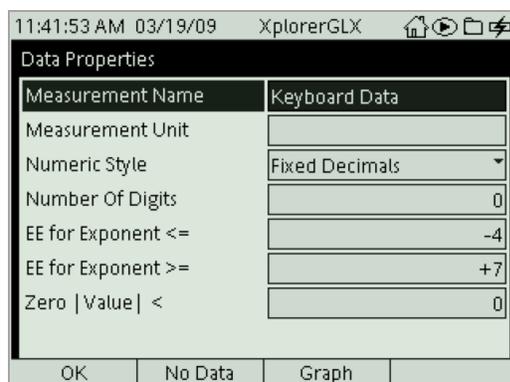
**Note:** The GLX is automatically set up to record temperature data from the fast-response temperature probe two times each second (continuous: 2 Hz). In this experiment, we only want to record one data point for each body part, and we need to tell the GLX when we are ready for the temperature to be recorded. This can be done by putting GLX into manual mode, which will display the measurement but will not record any data points until the **Flag** button (  ) is pressed.

- a. Press the **Home** button (  ). Then press **F4** Sensors.



- b. Press **F1** Mode. Then highlight **Manual**, and press  .

*Result:* A Data Properties display will be visible.



**Note:** This dialog is prompting you to give the GLX information that you would want to have associated with the data that will be collected by the sensor. In this case, the temperature measured by the sensor should be associated with the body location being measured.

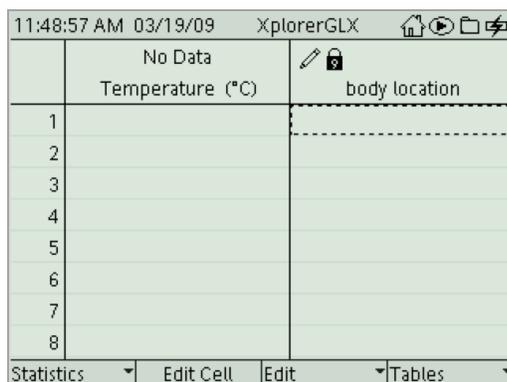
To enter this, highlight **Measurement Name** and press  . Use the alphanumeric keypad to enter “body location”. When finished, press  , then **F1** OK.

*Result:* The GLX is in manual sampling mode.

13. □ Create a table to display temperature.

- a. Press  to return to the home screen.
- b. Press  Table.

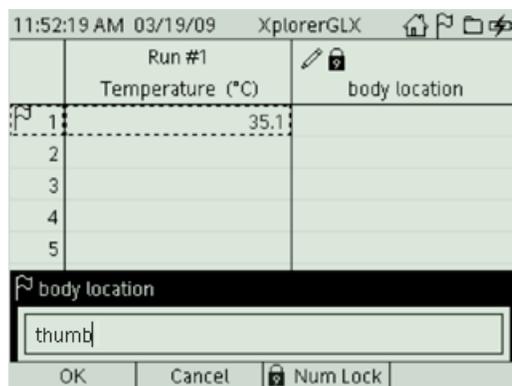
*Result:* The GLX displays a table that has temperature in the first column, and “body location” as the heading of the second column



	No Data Temperature (°C)	 body location
1		
2		
3		
4		
5		
6		
7		
8		

**Collect Data**

- 14. □ To begin monitoring temperature, press .
- 15. □ Place the temperature probe on your thumb, and hold it in position with your forefinger. Allow the temperature to stabilize, and then press  to record the data point.
- 16. □ You will be prompted to type in the “body location”. Press  to turn off the Number Lock feature, and then use the alphanumeric keypad to type “thumb”.



	Run #1 Temperature (°C)	 body location
1	35.1	
2		
3		
4		
5		

 body location

thumb

OK    Cancel     Num Lock

- a. Confirm that this column of data will be handled as text when prompted.
- 17. □ Write the temperature for your thumb in Table 1 in the Data Analysis section of this worksheet.

18. □ Repeat data collection for the palm of your hand, crook of the elbow, and your armpit. In each case, hold the temperature probe in place using the tip of your forefinger.

Press  to record these temperatures on the GLX, and write your data in Table 1 in the Data Analysis section of this worksheet.

19. □ Press  to complete data collection.

20. □ Save your work:

a. Press .

b. Highlight **Data Files** and press .



c. Press  Files and select **Save As** and press .

d. Use the alphanumeric keypad to type the name you would like to save the file as.

e. Press , then press  Save.

21. □ Complete the tasks for Part 1 of the Data Analysis section of this worksheet.

## Part 2 – Continuous sampling of water cooling

### Set Up – Run 1: Control

22. □ Create a new file by pressing  Files, select **New File**, and press .

23. □ Set the sampling rate on the GLX to record 1 data point each second (continuous: 1 seconds).

**Note:** The term "continuous" is used to describe the sampling mode that automatically records data, and the term "sampling rate" is used to describe how often the data will be recorded (once each second in this experiment). Taking many data points each second (high sampling rates) is needed when changes are expected to occur very quickly. Lower sampling rates are used when changes occur slowly or data is being taken for an extended period of time.

- a. Press , and then  Sensors.

*Result:* The display will show that the sample rate defaults to 2 second between samples.

- b. Highlight **Sampling Rate** using the arrow keys.

- c. Press  to bring up a list of sampling rates.

- d. Use the arrow keys to select 1, then press .

24. □ What is sampling rate? What does it mean to have a sampling rate of 1 sample per second?

Sampling rate is the rate at which the GLX records the data you are studying. If the sampling rate is 1 per second, then the GLX is gathering data once every second.

25. □ Predict what will happen to the temperature of warm water (~50 °C) that sits in a cup at room temperature for 5 minutes.

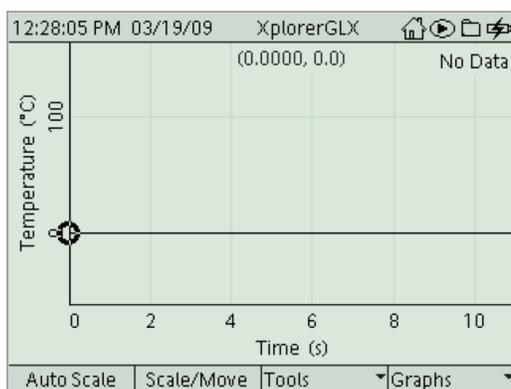
The temperature of the water will decrease.

26. □ To determine whether your prediction is correct you will run the experiment. To do this, start by setting up a graph display on the GLX.

- a. Press  to return to the Home screen.

- b. On the Home screen, press  Graph.

*Result:* A page is created with a graph prepared to display temperature versus time.



27. □ Fill a cup two-thirds full of hot water (~50 °C).

**Collect Data – Run 1: Control**

- 28.**  Place the tip of the fast-response temperature probe into the water. Hold the temperature probe in place or tape it to the cup so that it does not fall out of the cup during data collection.

- 29.**  Press  to begin data collection.

- 30.**  Press  AutoScale.

*Result:* The scale of the graph adjusts to clearly show any changes in temperature.

- 31.**  Continue to collect data for 5 minutes (300 seconds).

- 32.**  When 5 minutes (300 seconds) have passed, press  to complete data collection.

- 33.**  Rename the data run:

- a.** Press  and use the arrows to highlight “Run #1”.
- b.** Press  again, use the arrow keys to highlight “Rename Run”, and press  once more.
- c.** Use the alphanumeric keypad to type “control”.
- d.** Press  OK.

- 34.**  Complete the tasks in the Part 2 – Control section of the Data Analysis section of this worksheet.

**Set Up – Run 2: Student-designed cup**

- 35.**  Using the materials provided by your teacher and the knowledge gained in the previous data run, modify the cup you used in the first trial so that it will keep the water as warm as possible when it is left at room temperature for 5 minutes.

- 36.**  Describe the materials you used to modify the cup.

I wrapped the cup in cotton cloth.

- 37.**  How will you know if the cup you design holds heat better than the cup by itself?

I will perform the same experiment that I did with the normal cup and compare the results. For this example, if my cup holds heat better, then the change in temperature will be less than 4.3 °C.

- 38.**  Fill your specially designed cup two-thirds full of hot water (~50 °C).

**Collect Data – Run 2: Student-designed cup**

39.  Place the fast-response temperature probe into the water. Hold the temperature probe in place or tape it to the cup so it does not fall out of the cup during data collection.

40.  Press  to begin data collection.

41.  Does the modification to the cup seem to be keeping the water warmer? Explain how you know.

The temperature of the water seems to be dropping more slowly than the control run.

42.  After 5 minutes (300 seconds) have passed, press  to complete data collection.

43.  Rename the data run to indicate what changes have been made to the cup.

44.  Complete the tasks for the Part 2 – Student-designed cup section of the Data Analysis section of this worksheet.

45.  Save your work. You can refer to the steps at the end of Part 1 if you need a reminder.

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**Data Analysis**

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**Part 1 – Manual sampling of body temperature**

Table 1: Manual sampling of body temperature

Data Point	Body Part	Temperature (°C)
1	Thumb	28.7
2	Hand	30.7
3	Crook of elbow	34.4
4	Armpit	35.2

1.  Do you see any trends in the data that you collected? Explain any trends you observed in the space below.

Yes, the body temperature increases as you get closer to the body core.

2.  Does your prediction about body temperature match the data that you collected? Explain below.

Yes, my predictions were correct. Recorded body temperature was higher at the armpit than in the hand or thumb. It could be that the hands and thumbs are more exposed to ambient temperature; therefore they are cooler than body temperature. Also, warm blood, traveling from the core to the extremities loses heat to the environment; therefore, hands are colder than the body core.

## Part 2 – Periodic sampling of water cooling

### Run 1: Control

3.  Was your prediction about what will happen to the temperature of warm water (~50 °C) that sits in a paper cup at room temperature for 5 minutes supported by this experiment?

Yes, my prediction was supported. I predicted that the temperature of the water would decrease, and it did decrease.

4.  Find the maximum temperature and the minimum temperature of the water.
- Press **F3** Tools.
  - Use the arrow keys to highlight **Statistics** and press .
  - Use the arrows to make sure all of the data points in the run are selected within the dotted line box.

*Result:* The maximum and minimum values are displayed at the bottom of the graph screen.

5.  Record the maximum and minimum temperatures in Table 2.
6.  Calculate the change in temperature of the water in the cup during the 5 minutes, and record it in Table 2.

**Note:** Change in temperature = maximum temperature – minimum temperature.

Change in temperature = 49.4 °C – 45.1 °C = 4.3 °C.

7.  Explain why this temperature change was observed.

The temperature of the warm water decreased over time because the temperature of the room was cooler. Heat moves from hot objects to cold objects, so the heat from the warm water was slowly being transferred to the air.

### Run 2: Student-designed cup

8.  Record a description of your design for cup insulation in Table 2.
9.  Find the maximum temperature and minimum temperature of the water, and record it in Table 2.

10. □ Calculate the change in temperature of the water in the cup during the 5 minutes, and record it in Table 2.

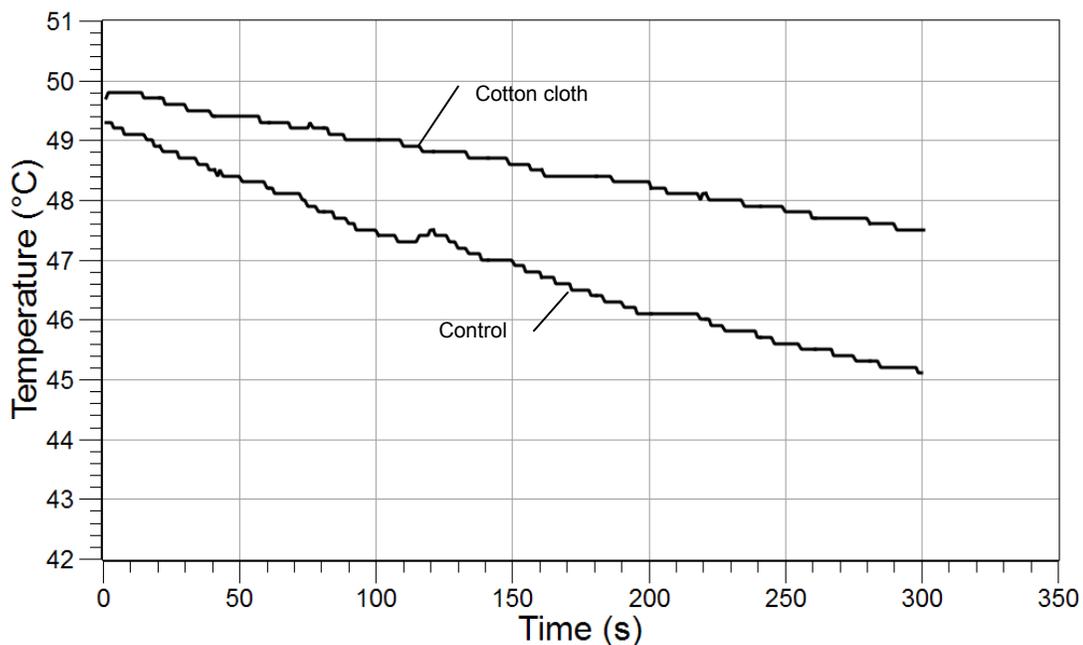
**Note:** Change in temperature = maximum temperature – minimum temperature.

Change in temperature =  $49.8\text{ }^{\circ}\text{C} - 47.5\text{ }^{\circ}\text{C} = 2.3\text{ }^{\circ}\text{C}$ .

Table 2: Effects of student cup modifications on change in temperature of water

Type of Cup	Describe the Cup Insulation Design	Maximum Temperature (°C)	Minimum Temperature (°C)	Change in Temperature (°C)
Plain cup		49.4	45.1	4.3
Student-designed cup	Wrapped the cup in cotton cloth	49.8	47.5	2.3

11. □ Sketch a graph of both data runs showing the Temperature on the y-axis and Time on the x-axis. Label both data runs.



## Analysis Questions

**1. How did your specially designed cup maintain the initial heat of the hot water compared to the plain cup? What evidence do you have to support your claim?**

The cup surrounded by cotton was more effective at maintaining the heat of the warm water than the plain cup. After sitting at room temperature for 5 minutes, the temperature of the water in the plain cup decreased 4.3 °C, whereas the temperature of the water in the cup wrapped in cotton decreased by only 2.3 °C.

**2. Can you be sure that your modification of the cup was the only reason for the change in temperature? Name two probable sources of error for this experiment.**

No, I cannot be sure that the insulation was the only reason for change in temperature. Some possible sources of error include the volume of water in the cup, which was not quantified, and the initial temperature of the water, which was not constant.

**3. How did the GLX technology improve the data collection and analysis you performed in the periodic sampling of water cooling part of the lab?**

GLX technology was able to collect an accurate temperature value every second, which would be very difficult using a normal thermometer. GLX technology also created the graph immediately so that the data could be analyzed immediately.

## Synthesis Questions

Use available resources to help you answer the following questions.

**1. Imagine that you just designed the most cost-effective insulated cup in the world! Companies everywhere will want to buy your design. Do you feel comfortable selling this design today? If not, what further steps would you take to ensure confidence in your design?**

After one test, you could not be confident in the design of your cup. Extensive testing should be done to ensure that your results are accurate and repeatable. Other scientists should test your hypothesis to ensure that the design works in other labs under other environmental conditions.

**2. Independent variables are those for which conditions are set by the experimenter. Dependent variables are those for which values depend upon the values of the independent variables. Identify the independent variable and dependent variable used in each part of the lab.**

	Independent Variable	Dependent Variable
Part 1: Body Temperature	body part	temperature
Part 2: Water Cooling	type of cup (plain cup versus student-designed cup)	temperature change

## Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

- 1. Which of the following is not a part of the inquiry process?**
  - A. Making observations
  - B. Posing hypotheses
  - C. Analyzing the data collected
  - D. Making sure your results support your hypothesis**
  
- 2. Why is it necessary to use a consistent technique while collecting data?**
  - A. To control the variables**
  - B. To change as many variables as you can
  - C. To make sure your results support your hypothesis
  - D. None of the above
  
- 3. How would you keep a cup of ice water cold all day?**
  - A. Leave the cup in a dark place
  - B. Make sure the cup does not have a lid
  - C. Wrap the cup in layers of newspaper**
  - D. Stir the ice water frequently

## Extended Inquiry Suggestions

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Instruct students to compare their data with that of other groups to determine which group designed the most effective insulation for the cup. Be sure to compare the changes in temperature across groups, not just minimum temperatures.

Have the students design their own experiment to research.

Let students design and conduct an experiment that explores the effects of activity on body temperature

## Scientific Inquiry – DataStudio®

### Objectives

This lab is designed to help students familiarize themselves with DataStudio® software while engaging in scientific investigations. By completing these exercises students learn to:

- ◆ Engage in the process of scientific inquiry by making predictions, collecting and analyzing data, and explaining observations
- ◆ Use the most popular and effective DataStudio tools

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Setting up the DataStudio to collect data in different types of experiments
- ◆ Using the manual and periodic sampling modes of DataStudio
- ◆ Using the fast-response temperature probe with DataStudio to collect temperatures of various body parts in a table display
- ◆ Using the fast-response temperature probe and the graph display to continuously record and display the temperature of hot water that is left to cool in an insulated and non-insulated cup at room temperature for 5 minutes
- ◆ Designing a way to insulate a cup so it keeps hot water warm for as long as possible

### Time Requirement

◆ Preparation time	5 minutes
◆ Pre-lab discussion and activity	5 minutes
◆ Lab activity	40 minutes

### Materials and Equipment

#### *For each student or group:*

- ◆ Computer with DataStudio software
- ◆ PASPORT sensor interface<sup>1</sup>
- ◆ PASPORT temperature sensor
- ◆ Fast response temperature probe
- ◆ Cup<sup>2</sup>, 9- to 12-oz
- ◆ Materials available in the laboratory to design a cup that keeps water hot<sup>3</sup>
- ◆ Hot water, 16 oz

<sup>1</sup>Use a PASPORT USB Link, SPARKLink, PASPORT PowerLink, PASPORT AirLink SI, PASPORT Xplorer, or PASPORT Xplorer GLX

<sup>2</sup>Use a non-insulated plastic or paper cup.

<sup>3</sup>For example: polystyrene, cloth, foil, plastic wrap, wool, water baths, or packing peanuts

## **Background**

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### **The Scientific Method and Scientific Inquiry**

Scientific inquiry is a general term for the numerous ways in which scientists learn about the natural world to produce scientific knowledge. The knowledge produced is supported by evidence collected during the inquiry process.

Traditionally, the term scientific method has been used when teaching students how scientists learn about the natural world. However, this term has recently been criticized because it creates the misconception that all scientists follow the same steps (processes) in the same order all the time. In reality, the many methods used to investigate nature in the various sciences are better represented by a broader term, scientific inquiry.

Although the sequence and choice of steps used varies from scientist to scientist and from one investigation to the next, a common set of processes is used by scientists to learn about the natural world. Making observations; asking questions; making predictions or posing hypotheses; planning investigations; controlling variables; using systematic, reliable, repeatable, and externally validated techniques; analyzing the data collected; and interpreting and communicating results are among the major processes that are involved in scientific inquiry.

Each lab in this manual is designed to guide students through a sequence of scientific inquiry processes that may be used to learn about the world around them. Students learn about scientific inquiry by engaging in inquiry processes, and they develop the skills needed to answer their own questions through scientific inquiry. At the same time, students gain content knowledge by actively engaging in scientific inquiry as opposed to being told about or reading about science.

Scientists in the 21st century increasingly rely on technology to enhance data collection and analysis. Technology enables scientists to collect large amounts of data accurately and to display, analyze, and interpret their data more quickly.

### **Using DataStudio**

This investigation includes detailed steps on how to set up the DataStudio to collect data in different types of experiments. In the first part of this investigation, students find the temperature of four different body parts. To do this, the student will need to place the temperature probe on the body part and allow the temperature to stabilize. When ready, the student will trigger DataStudio to record the data point. DataStudio allows for this type of data collection in manual sampling mode. Manual sampling mode allows users to see the data value as it changes over time and to record a data point when they are ready.

In the second part of this investigation, students measure temperature values each second as a cup of hot water cools. DataStudio allows for a measurement to be automatically recorded at a specified rate. This type of data collection is called periodic sampling (this is the default mode). Taking many data points each second (high sampling rates) is needed when changes are expected to occur very quickly. Lower sampling rates are used when changes occur slowly or data is being taken for an extended period of time. The default sample rate for each PASCO sensor is appropriate for the most typical applications of that sensor, reducing the need for the user to change the sample rate before each experiment.

This investigation also guides students in two different ways to display data (table and graph) and how to start and stop data collection, open and close tool palettes, perform simple analyses (statistics), and save data.

This combination of methods and tools can be a powerful stimulation for students to develop their own scientific inquiries arising from their natural curiosity about the world around them.

## Pre-Lab Discussion

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Lead a discussion with your students about what science is and what processes scientists use to learn about the world around them. Lead the discussion with questions such as “What is science?” and “What do scientists do?” Students will probably start out with specifics about what different scientists do. Let the ideas emerge, and see if you can find a common thread within the ideas. Ask students questions about different types of technologies scientists use and how these technologies help them in the different processes.

**Teacher Tip:** Accept all answers and write ideas on the board or overhead projector to remain displayed during the activity.

Ideally, you can lead your students to the conclusion that science is “asking questions, making predictions (constructing hypotheses), conducting experiments, collecting data (gathering evidence), analyzing the data, and drawing conclusions.” Let your students know that this is exactly what they will be doing in this laboratory exercise and throughout the year in your class.

## Lab Preparation

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These are the materials and equipment to set up prior to the lab.

1. Make cups and insulating material available to each student group. Use what you have on hand as possible insulating materials. Some suggestions include: polystyrene cups, cloth, foil, plastic wrap, wool, water baths, or packing peanuts.
2. Make sure that a source of hot water with a constant temperature (~50 °C) is available for your students throughout the activity.

## Safety

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Add these important safety precautions to your normal laboratory procedures:

- ◆ Do not use the fast-response temperature probe in water that is 70 °C or hotter.
- ◆ Be careful when working with hot water.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

3	1	5	2	4
Next, carry out a controlled experiment to determine whether body temperature is the same for different parts of your body.	Set up DataStudio, and connect the temperature sensor and fast-response temperature probe to the computer using a sensor interface.	Analyze the effectiveness of the cup you designed to slow the rate at which warm water cools.	First, determine how the temperature of your thumb compares to your predicted body temperature.	Use DataStudio in a different mode of sampling to determine the temperature change of hot water as it cools in a plain cup versus a student-designed cup.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

### Part 1 – Manual sampling of body temperature

#### Set Up

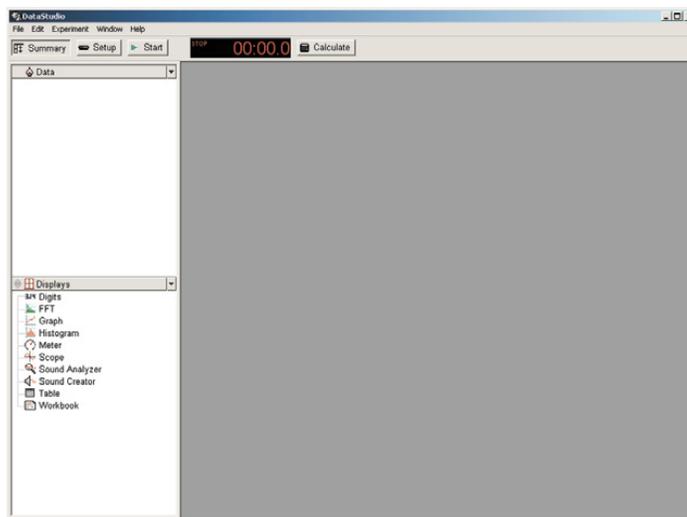
1.  What is the average internal temperature of the human body? If necessary, discuss this with your classmates or consult a textbook and write the answer in the space below.

\_\_\_\_\_ 37.0 \_\_\_\_\_ °C

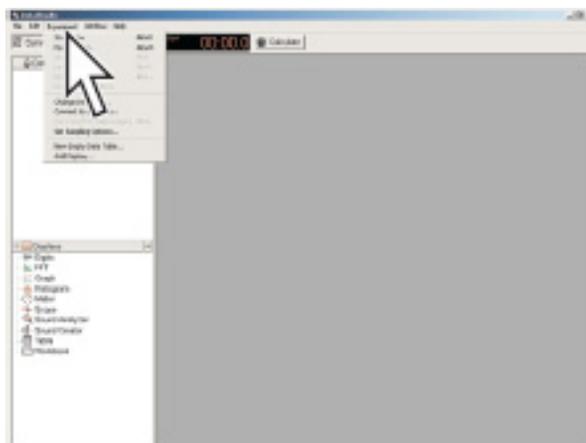
2.  Connect the fast response temperature probe to the PASPORT temperature sensor, the PASPORT temperature sensor to the PASPORT interface, and the PASPORT interface to the computer to launch Data Studio.
- a. If DataStudio is already running, Open the **File** menu and select **New Activity**.
  - b. If the Welcome to DataStudio window appears, select **Create Experiment**.
  - c. If Data Studio does not launch automatically, double-click the DataStudio program icon.

**Note:** Each sensor will automatically bring up a default display in DataStudio when it is connected the first time. For this lab, you can either choose to close the display or ignore it.

3.  Set up DataStudio to monitor temperature data.
- a. If the Summary bar is not on the screen, click **Summary** ( Summary) so that your screen looks like this:



- b. In the summary bar, double-click **Digits**.
- c. If the Please Choose a Data Source window appears, click the **Temperature** measurement.
4.  Begin monitoring live data by clicking **Monitor Data** in the **Experiment** menu.



5.  Write the room temperature in the space below.

\_\_\_\_\_ 23.4 \_\_\_\_\_ °C

6.  What do you think the temperature of your thumb will be?

\_\_\_\_\_ 37.0 \_\_\_\_\_ °C

7.  Place the end of the fast-response temperature probe on your thumb, and hold it in place with your forefinger. When the temperature reading stabilizes, write the temperature in the space below.

\_\_\_\_\_ 33.9 \_\_\_\_\_ °C

8.  Click **Stop** () to stop monitoring data.
9.  How did the temperature of your thumb compare to the value for average internal body temperature?

The temperature of my thumb was lower than normal internal body temperature.

10.  Why do you think the temperature of your thumb did not match the value for average internal body temperature? Below is a list of ideas that other students have generated. Add at least one additional explanation to the bottom of the list.
- A. Body temperature depends on the age of the person.
  - B. Body temperature is not constant, but changes based on the weather.
  - C. Body temperature is based on gender.
  - D. Body temperature changes with distance from the body core.
  - E. Body temperature depends on whether or not the body part is covered by clothing.
11.  This investigation will explore "student idea D". You will determine the temperature of body parts that get incrementally closer to the core of the body (thumb, hand, elbow, and armpit).

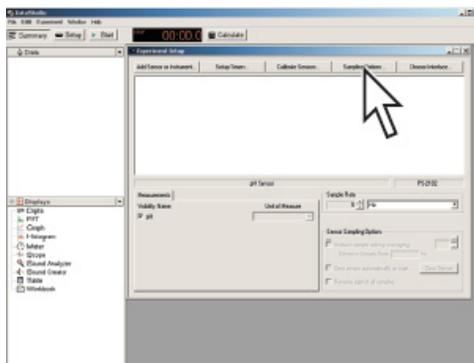
**Note:** Your teacher may allow you time later to design an experiment to test the other ideas.

12.  Predict how the temperature of a body part will be affected by its distance from the core of the body.

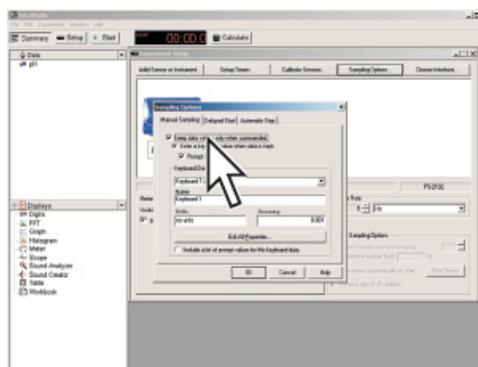
The temperature of body parts farthest from the core will have the lowest temperature and the temperature will increase as it gets closer to the core body temperature.



- b.** In the Experiment Setup window, click **Sampling Options**.



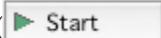
- c.** Select **Keep data values only when commanded**.



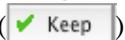
*Result:* This puts DataStudio into manual sampling mode.

- d.** Clear the check box labeled **Enter a keyboard value when data is kept**.  
**e.** Click **OK** to close the Sampling Options Window.  
**f.** Close the Setup Window.

### Collect Data

- 15.**  To begin monitoring temperature, click the **Start** button (  ).

The start button is replaced by the **Keep** button (  ).

- 16.**  Place the temperature probe on your thumb, and hold it in position with your forefinger. Allow the temperature to stabilize, and then click the **Keep** button (  ) to record the data point.

- 17.**  Write the temperature for your thumb in Table 1 in the Data Analysis section of this worksheet.

- 18.**  Repeat data collection for the palm of your hand, crook of the elbow, and your armpit. In each case, hold the temperature probe in place using the tip of your forefinger. Click the **Keep** button to record these temperatures in the DataStudio table and write your data in Table 1 in the Data Analysis section of this worksheet.

19.  Click the **Stop** button (  ) to complete data collection.
20.  Save your work:
- Open the **File** menu and select **Save Activity As**.
  - Select a folder.
  - Type a filename and click **Save**.
21.  Complete the tasks for Part 1 of the Data Analysis section of this worksheet.

## Part 2 – Periodic sampling of water cooling

### Set Up – Run 1: Control

22.  Open the **File** menu and select **New Activity**.
23.  Predict what will happen to the temperature of warm water ( $\sim 50\text{ }^{\circ}\text{C}$ ) that sits in a cup at room temperature for 5 minutes.

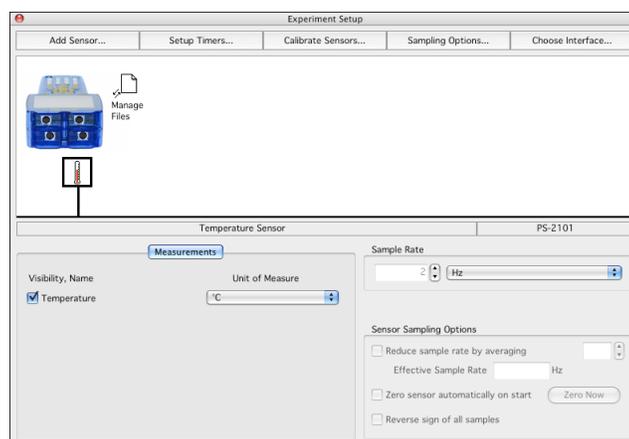
The temperature of the water will decrease.

24.  To determine whether your prediction is correct you will run the experiment. To do this, start by setting up a graph display in DataStudio. If a graph of Temperature versus Time is already present you can continue to the next step
- If the summary bar is not visible, click **Summary**.
  - In the summary bar, double-click **Graph**.
  - If the Please Choose a Data Source window appears, click the measurement that you would like to see on the y-axis (temperature); then click **OK**.
25.  Set the sampling rate in DataStudio to record 1 data point each second.

**Note:** The term "periodic" is used to describe the sampling mode that automatically records data, and the term "sampling rate" is used to describe how often the data will be recorded (once each second in this experiment). Taking many data points each second (high sampling rates) is needed when changes are expected to occur very quickly. Lower sampling rates are used when changes occur slowly or data is being taken for an extended period of time. The default mode of DataStudio is Periodic Sampling.

- Click **Setup**.

- b.** In the Experiment Setup window, click the icon of the sensor that you wish to change.



- c.** Under **Sample Rate**, select the units (seconds) and select the value **1**.  
**d.** Close the Setup window.

- 26.** □ What is sampling rate? What does it mean to have a sampling rate of 1 sample per second?

Sampling rate is the rate at which DataStudio records the data you are studying. If the sampling rate is 1 per second, then DataStudio is gathering data once every second.

- 27.** □ Fill a cup two-thirds full of hot water (~50 °C).

**Collect Data – Run 1: Control**

- 28.** □ Place the tip of the fast-response temperature probe into the water. Hold the temperature probe in place or tape it to the cup so that it does not fall out of the cup during data collection.

- 29.** □ Click the **Start** button (  ) to begin recording data.

- 30.** □ Adjust the scale of the graph to clearly see any changes in temperature:

- a.** Click the **Scale to Fit** button (  ) in the toolbar of the graph to automatically rescale the graph to fit all the data.

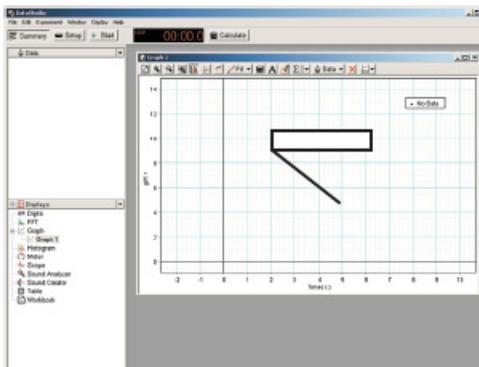
- 31.** □ Continue to collect data for 5 minutes (300 seconds).

- 32.** □ When 5 minutes (300 seconds) have passed, touch the **Stop** button (  ).

33.  Label the data run:

- In the toolbar of the graph, click the **Note** button (  ).
- Click where you want the note to point. This will bring up the Annotation Properties window.
- Type "Control" and click **OK**.

*Result:* The note appears on the graph.



- Drag the note to position it on the graph.

34.  Complete the tasks in the Part 2 – Control section of the Data Analysis section of this worksheet.

### ***Set Up – Run 2: Student-designed cup***

35.  Using the materials provided by your teacher and the knowledge gained in the previous data run, modify the cup you used in the first trial so that it will keep the water as warm as possible when it is left at room temperature for 5 minutes.

36.  Describe the materials you used to modify the cup.

I wrapped the cup in cotton cloth.

37.  How will you know if the cup you design holds heat better than the cup by itself?

I will perform the same experiment that I did with the normal cup and compare the results. For this example, if my cup holds heat better, then the change in temperature will be less than 4.3 °C.

38.  Fill your specially designed cup two-thirds full of hot water (~50 °C).

### ***Collect Data – Run 2: Student-designed cup***

39.  Place the fast-response temperature probe into the water. Hold the temperature probe in place or tape it to the cup so it does not fall out of the cup during data collection.

40.  Click the **Start** button (  ) to begin data collection.

41.  Does the modification to the cup seem to be keeping the water warmer? Explain how you know.

The temperature of the water seems to be dropping more slowly than the control run.

42.  After 5 minutes (300 seconds) have passed, click the **Stop** button (  ).

43.  Label the data run with a word or phrase that identifies the change to the cup.

44.  Complete the tasks for the Part 2 – Student-designed cup section of the Data Analysis section of this worksheet.

45.  Save your work:
- Open the **File** menu and select **Save Activity As**.
  - Select a folder.
  - Type a filename and click **Save**.

## Data Analysis

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### Part 1 – Manual sampling of body temperature

Table 1: Manual sampling of body temperature

Data Point	Body Part	Temperature (°C)
1	Thumb	28.7
2	Hand	30.7
3	Crook of elbow	34.4
4	Armpit	35.2

1.  Do you see any trends in the data that you collected? Explain any trends you observed in the space below.

Yes, the body temperature increases as you get closer to the body core.

2.  Does your prediction about body temperature match the data that you collected? Explain below.

Yes, my predictions were correct. Recorded body temperature was higher at the armpit than in the hand or thumb. It could be that the hands and thumbs are more exposed to ambient temperature; therefore they are cooler than body temperature. Also, warm blood, traveling from the core to the extremities loses heat to the environment; therefore, hands are colder than the body core.

**Part 2 – Periodic sampling of water cooling****Run 1: Control**

3.  Was your prediction about what will happen to the temperature of warm water (~50 °C) that sits in a paper cup at room temperature for 5 minutes supported by this experiment?

Yes, my prediction was supported. I predicted that the temperature of the water would decrease, and it did decrease.

4.  Find the maximum temperature and the minimum temperature of the water.

a. In the toolbar of the graph, click the **Statistics** button (.

5.  Record the maximum and minimum temperatures in Table 2.

6.  Calculate the change in temperature of the water in the cup during the 5 minutes, and record it in Table 2.

**Note:** Change in temperature = maximum temperature – minimum temperature.

Change in temperature = 49.4 °C – 45.1 °C = 4.3 °C.

7.  Explain why this temperature change was observed.

The temperature of the warm water decreased over time because the temperature of the room was cooler. Heat moves from hot objects to cold objects, so the heat from the warm water was slowly being transferred to the air.

**Run 2: Student-designed cup**

8.  Record a description of your design for cup insulation in Table 2.

9.  Find the maximum temperature and minimum temperature of the water, and record it in Table 2.

10. □ Calculate the change in temperature of the water in the cup during the 5 minutes, and record it in Table 2.

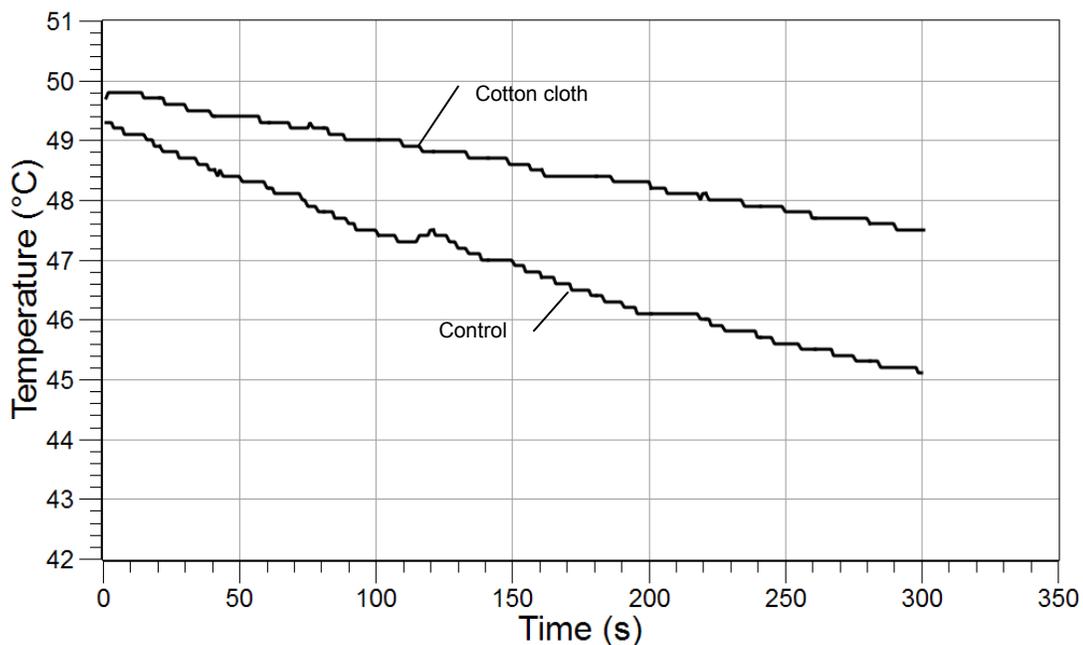
**Note:** Change in temperature = maximum temperature – minimum temperature.

Change in temperature =  $49.8\text{ }^{\circ}\text{C} - 47.5\text{ }^{\circ}\text{C} = 2.3\text{ }^{\circ}\text{C}$ .

Table 2: Effects of student cup modifications on change in temperature of water

Type of Cup	Describe the Cup Insulation Design	Maximum Temperature (°C)	Minimum Temperature (°C)	Change in Temperature (°C)
Plain cup		49.4	45.1	4.3
Student-designed cup	Wrapped the cup in cotton cloth	49.8	47.5	2.3

11. □ Sketch a graph of both data runs showing the Temperature on the y-axis and Time on the x-axis. Label both data runs.



## Analysis Questions

**1. How did your specially designed cup maintain the initial heat of the hot water compared to the plain cup? What evidence do you have to support your claim?**

The cup surrounded by cotton was more effective at maintaining the heat of the warm water than the plain cup. After sitting at room temperature for 5 minutes, the temperature of the water in the plain cup decreased 4.3 °C, whereas the temperature of the water in the cup wrapped in cotton decreased by only 2.3 °C.

**2. Can you be sure that your modification of the cup was the only reason for the change in temperature? Name two probable sources of error for this experiment.**

No, I cannot be sure that the insulation was the only reason for change in temperature. Some possible sources of error include the volume of water in the cup, which was not quantified, and the initial temperature of the water, which was not constant.

**3. How did DataStudio and PASPORT technology improve the data collection and analysis you performed in the periodic sampling of water cooling part of the lab?**

DataStudio and PASPORT technology was able to collect an accurate temperature value every second, which would be very difficult using a normal thermometer. DataStudio technology also created the graph immediately so that the data could be analyzed immediately.

## Synthesis Questions

Use available resources to help you answer the following questions.

**1. Imagine that you just designed the most cost-effective insulated cup in the world! Companies everywhere will want to buy your design. Do you feel comfortable selling this design today? If not, what further steps would you take to ensure confidence in your design?**

After one test, you could not be confident in the design of your cup. Extensive testing should be done to ensure that your results are accurate and repeatable. Other scientists should test your hypothesis to ensure that the design works in other labs under other environmental conditions.

**2. Independent variables are those for which conditions are set by the experimenter. Dependent variables are those for which values depend upon the values of the independent variables. Identify the independent variable and dependent variable used in each part of the lab.**

	Independent Variable	Dependent Variable
Part 1: Body Temperature	body part	temperature
Part 2: Water Cooling	type of cup (plain cup versus student-designed cup)	temperature change

## Multiple Choice Questions

---

Select the best answer or completion to each of the questions or incomplete statements below.

- 1. Which of the following is not a part of the inquiry process?**
  - A. Making observations
  - B. Posing hypotheses
  - C. Analyzing the data collected
  - D. Making sure your results support your hypothesis**
  
- 2. Why is it necessary to use a consistent technique while collecting data?**
  - A. To control the variables**
  - B. To change as many variables as you can
  - C. To make sure your results support your hypothesis
  - D. None of the above
  
- 3. How would you keep a cup of ice water cold all day?**
  - A. Leave the cup in a dark place
  - B. Make sure the cup does not have a lid
  - C. Wrap the cup in layers of newspaper**
  - D. Stir the ice water frequently

## Extended Inquiry Suggestions

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Instruct students to compare their data with that of other groups to determine which group designed the most effective insulation for the cup. Be sure to compare the changes in temperature across groups, not just minimum temperatures.

Have the students design their own experiment to research.

Let students design and conduct an experiment that explores the effects of activity on body temperature.

## Lab 1: Determining the Empirical Formula of a Compound

### Objectives

---

Students determine the stoichiometric composition of an ionic compound.

### Procedural Overview

---

Students gain experience conducting the following procedures using a balance and a Bunsen burner to:

- ◆ Perform a reaction under controlled conditions—heating a metal to form the metal oxide
- ◆ Eliminate the byproduct of a reaction and convert it to a desired product
- ◆ Determine the change in mass to find the stoichiometric composition of the metal oxide

### Time Requirement

---

- |                                   |             |
|-----------------------------------|-------------|
| ◆ Preparation time                | 15 minutes  |
| ◆ Pre-lab discussion and activity | 15 minutes  |
| ◆ Lab activity                    | 180 minutes |

### Materials and Equipment

---

***For each student or group:***

- |                         |  |
|-------------------------|--|
| ◆ Crucible with lid     | ◆ Wash bottle with deionized water     |
| ◆ Ring stand            | ◆ Clay triangle                        |
| ◆ Bunsen burner         | ◆ Paper clip                           |
| ◆ Balance (1 per class) | ◆ Magnesium powder, 0.5 g <sup>1</sup> |
| ◆ Crucible tongs        |  |

<sup>1</sup>Refer to the Lab Preparation section for alternate forms of magnesium.

## **Concepts Students Should Already Know**

---

Students should be familiar with the following concepts:

- ◆ The meaning of a chemical formula
- ◆ Balancing chemical equations
- ◆ The composition of air

## **Related Labs in This Guide**

---

Labs conceptually related to this one include:

- ◆ Lab 2: Determine the Percent Water in a Hydrate
- ◆ Lab 15a: Synthesis of Coordination Compound

## **Using Your Data Collection System**

---

Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

As this lab activity does not use a data collection system, no Tech Tips (indicated by the symbol "◆" and a superscripted number following a step) are needed.

## **Background**

---

The empirical formula of a compound is the simplest whole number ratio of the elements in the compound. The stoichiometric ratio of the different types of atoms in a compound is given by that ratio of whole numbers. One mole of a substance has the number of moles of different types of atoms in the same ratio as the stoichiometric ratio.

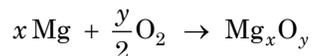
For example, the formula  $\text{H}_2\text{O}$  reflects a 2:1 stoichiometric ratio between hydrogen and oxygen atoms. Also, one mole of  $\text{H}_2\text{O}$  molecules has a 2:1 molar ratio between the total number of hydrogen and oxygen atoms. That is, there are 2 moles of hydrogen and 1 mole of oxygen atoms in 1 mole of  $\text{H}_2\text{O}$  molecule.

The empirical formula can be determined experimentally if a compound can be synthesized from its elements. This process requires three steps: determining the mass of each element in the compound; calculating the number of moles of each element in the sample; and expressing the molar ratio of each element as the smallest whole number.

## Pre-Lab Activity

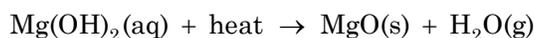
### Setting the stage for the activity

Molecular oxygen is very reactive, whether in pure form or in a mixture such as air. The most abundant component of air, nitrogen, is relatively unreactive. An element reacting with oxygen forms an oxide. For example, magnesium and oxygen form magnesium oxide:



The reaction of nitrogen with an element forms a nitride. Oxygen has a greater reactivity than nitrogen, therefore the oxide is more likely to form.

In this experiment, you will burn magnesium (Mg) in air to form magnesium oxide and magnesium nitride. The magnesium nitride will be converted to magnesium hydroxide and ammonia by adding water. Upon heating, the magnesium hydroxide will be converted to magnesium oxide with the release of water vapor.



### Example calculation to try

Chemical analysis of a solid found it contains 1.76 g of aluminum and 1.57 g of oxygen. The simplest whole number ratio was found to be 2:3. Therefore the empirical formula is  $\text{Al}_2\text{O}_3$  for aluminum oxide.

$$\frac{1.76 \text{ g Al}}{26.98 \frac{\text{g}}{\text{mol}} \text{ Al}} = 0.0652 \text{ mol Al}$$

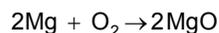
$$\frac{1.57 \text{ g O}}{16.00 \frac{\text{g}}{\text{mol}} \text{ O}} = 0.0981 \text{ mol O}$$

The molar ratio between the Al and O is

$$\begin{aligned} \left( \frac{0.0652 \text{ mol Al}}{0.0652 \text{ mol Al}} \right) : \left( \frac{0.0981 \text{ mol O}}{0.0652 \text{ mol Al}} \right) &= 1.00 \text{ mol Al} : 1.50 \text{ mol O} \\ &= 2.00 \text{ mol Al} : 3.00 \text{ mol O} \end{aligned}$$

Therefore, the empirical formula is  $\text{Al}_2\text{O}_3$ .

**1.** Based on the information in the Background section, propose a chemical equation for the reaction between Mg and  $\text{O}_2$ . (Hint: Refer to Equation 1 between Mg and  $\text{N}_2$ ).



## Lab 1: Determining the Empirical Formula of a Compound

---

### 2. What stoichiometric ratio do you expect between the Mg and O? Why?

Mg:O = 1:1

Equation 1 suggests that nitrogen has a  $-3$  charge and magnesium has a  $+2$  charge in its compounds. Also, knowing that oxygen has a  $-2$  charge in oxides would suggest a 1:1 ratio between Mg and O.

### 3. The $N_2$ molecule is extremely stable, yet it appears to react with Mg in this experiment. Propose an explanation for the reaction between Mg and $N_2$ . (Hint: Think about the amount of energy required to break the bonds in a stable molecule.)

The Mg burns at an extremely high temperature, releasing sufficient energy to break up the  $N_2$  molecule.

## Lab Preparation

---

These are the materials and equipment to set up prior to the lab:

1. Magnesium powder is best because it oxidizes most readily. Magnesium chips will also work. Magnesium ribbon is acceptable but is difficult to oxidize. If ribbon is used, loosely wind the ribbon around itself into a spiral to improve oxidation. With magnesium ribbon, you may need sandpaper or steel wool to clean it.
2. Explain to students how the Bunsen burner works and how the air flow can be controlled.

## Safety

---

Add these important safety precautions to your normal laboratory procedures:

- ◆ Be very careful when using the Bunsen burner.
- ◆ Never place a hot crucible or other hot objects on a balance pan.
- ◆ Do not move the hot crucible from the clay triangle until it has cooled.
- ◆ Make sure there is nothing flammable around the setup.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

6	1	3	4
Determine the empirical formula based on the number of moles of magnesium and oxygen.	Make sure the crucible is clean, has no defects, and there are no contaminants.	React the magnesium with air by heating the crucible and exposing the Mg to air.	Convert the magnesium nitride to magnesium oxide.
	5	2	
	Measure the mass of the crucible containing the reacted magnesium.	Measure the mass of the clean crucible without and then with the magnesium sample.	

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** This activity does not use a data collection system, so there are no Tech Tips (indicated by the symbol "T" and a superscripted number following a step).

- Obtain a crucible and lid, and inspect the crucible for cracks, nicks, and other defects.

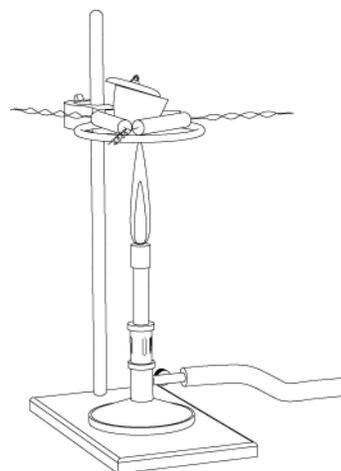
**Note:** Replace a defective crucible.

- Measure and record the mass of the crucible and lid in Table 1.
- You will be heating the crucible before the experiment is performed. Why is it necessary to heat the crucible before performing the experiment?

Heating eliminates error from such things as contamination or moisture that may be present in the crucible. These could change the mass of the crucible.

## Lab 1: Determining the Empirical Formula of a Compound

4.  Prepare the crucible as follows:
- Place the crucible and lid on the clay triangle over the Bunsen burner.
  - Heat the crucible with a gentle flame for 5 minutes by moving the burner around the bottom of the crucible.
  - After the bottom of the crucible has become red-hot, increase the flame by allowing more air into the burner.
  - Continue moving the burner around the bottom of the crucible.
  - Heat the crucible for 10 to 12 minutes.
  - Allow the crucible to cool to room temperature.



**Note:** For the rest of the experiment handle the crucible and lid using only crucible tongs. Also, do not set the crucible on the lab bench or it may crack or become contaminated.

5.  Why do you have to use tongs to hold the lid?

Touching the lid with bare hands contaminates the lid and changes its mass, introducing error in the mass measurement. Also, the crucible may still be hot and cause injury.

6.  After the crucible has cooled, measure and record the mass of the “fired” crucible together with its lid in Table 1.

Table 1: Mass of the empty crucible measured to the nearest milligram

Measurement	Trial 1	Trial 2
Crucible and lid before heating (g)	28.233	30.213
Crucible and lid after the first heating (g)	28.230	30.211
Crucible and lid after the second heating (g)	28.231	30.210
Crucible and lid after the third heating (g)		
Crucible and lid after the fourth heating (g)		

7.  Repeat the steps above for heating, cooling, and measuring the mass of the crucible and lid until you have two readings for the mass that are within 10 mg of each other.
8.  Copy the last measurement in Table 1 into Table 3.

### Collect Data

9.  Obtain between 0.17 and 0.23 g of magnesium powder or chips. (Magnesium ribbon is acceptable, although it does not burn as easily.)

10.  Add the magnesium sample to the crucible.

**Note:** If magnesium ribbon is used, you may need to clean it first with sandpaper or steel wool until it is bright and shiny. Also, wind the magnesium ribbon into a loose spiral so that it will fit into the crucible and oxidize.

11.  Measure the mass of the crucible, lid, and magnesium to the nearest milligram and record it in Table 3.

12.  Return the crucible, with the sample and lid, to the clay triangle over the Bunsen burner.

13.  To react all of the metal:

a. Gently heat the sample for 2 to 3 minutes by moving the burner around the bottom of the crucible.

b. Increase the heat and continue heating until the sample starts to glow.

**Note:** Remember that the top third of the flame is the hottest.

c. Continue moving the burner around the bottom of the crucible.

d. Manage the burning so the magnesium glows for about 3 minutes:

Do not remove the lid. Using the crucible tongs, slightly lift the lid for a few seconds to admit air into the crucible.

**Note:** Quickly cover the crucible with the lid if the metal bursts into flame. When done correctly, the content of the crucible will begin to glow.

e. Continue heating for about 3 more minutes, carefully lifting the lid to observe the contents of the crucible.

f. When the bright glow of the contents of the crucible turns to a pale glow, stop the heating.

g. Allow the crucible to cool to room temperature with the lid in place.

h. Using the tongs to lift the lid, break the crust over the contents of the crucible with a straightened paper clip.

i. Hold the lid with the tongs and mix the contents carefully so that any unreacted metal comes to the surface.

**Note:** Make sure no residue remains on the paper clip.

14.  Why do you need to bring the unreacted metal to the surface?

Bringing the unreacted metal to the surface allows it to be exposed to the air, and therefore to oxygen, for the next time the sample is heated.

15.  Repeat all parts of the step, “To react all of the metal,” until no unreacted metal is visible.

16.  When the sample has cooled and no metal is present, use the squeeze bottle to add three drops of deionized water.

## Lab 1: Determining the Empirical Formula of a Compound

17.  By wafting the air towards your nose over the crucible, try to identify and describe the smell of ammonia.

Ammonia has a distinct pungent odor that irritates the sense of smell.

18.  Heat the crucible and sample again. It takes a considerable amount of time and heat to decompose  $\text{Mg}(\text{OH})_2$  to  $\text{MgO}$  and  $\text{H}_2\text{O}$ .
- With the lid slightly open to allow water vapor to escape, begin heating the crucible and sample over a gentle flame for about 3 minutes.
  - Gradually increase the heat, being careful not to let the crucible heat up too fast such that water spatters out.
  - Continue at high heat for about 15 to 20 minutes, carefully lifting the lid to observe the contents of the crucible.
  - When the sample has a pale glow, stop the heating.
  - Allow the crucible to cool to room temperature with the lid in place.
19.  After the crucible and sample have cooled, measure and record the mass of the crucible, lid, and metal oxide, to the nearest milligram, in Table 2.

Table 2: Mass of the crucible and magnesium oxide, measured to the nearest milligram

Measurement	Trial 1	Trial 2
Crucible, lid, and MgO after the first heating (g)	28.527	30.520
Crucible, lid, and MgO after the second heating (g)	28.526	30.521
Crucible, lid, and MgO after the third heating (g)		
Crucible, lid, and MgO after the fourth heating (g)		

20.  Reheat the crucible and sample at high heat for 5 minutes. Allow the crucible and sample to cool, then measure and record the mass in Table 2. Repeat this step until you obtain two consecutive readings within 10 mg of each other.
21.  Copy the last measurement in Table 2 into Table 3.
22.  Dispose of the metal oxide in the appropriate container.
23.  Beginning with preparing the crucible, repeat the entire procedure with a new sample of magnesium (between 0.17 and 0.23 g). Record the results for Trial 2 in the specified tables and column.
24.  Wash the crucible with soap, and then rinse three times with tap water to clean the crucible of magnesium oxide residue.
25.  After rinsing three times with tap water, rinse again with deionized water.

26.  Clean up according to your teacher's instructions.

## Data Analysis

1.  Calculate the mass of magnesium and mass of magnesium oxide. Record those values in Table 3.

Table 3: Measurements and calculations of the crucible, magnesium, and magnesium oxide mass

Parameter	Trial 1	Trial 2
Mass of the crucible and lid (g)	28.231	30.210
Mass of the crucible, lid, and magnesium (g)	28.413	30.405
Mass of magnesium (g)	0.182	0.195
Mass of crucible, lid, and magnesium oxide (g)	28.526	30.521
Mass of magnesium oxide (g)	0.295	0.311
Amount of magnesium in the compound (mol)	$7.49 \times 10^{-3}$	$8.02 \times 10^{-3}$
Mass of oxygen in the compound (g)	0.113	0.116
Amount of oxygen in the compound (mol)	$7.06 \times 10^{-3}$	$7.25 \times 10^{-3}$
Simplest whole number ratio of oxygen to magnesium	1:1	1:1
Experimental empirical formula of the compound using significant figures	Mg <sub>1.00</sub> O <sub>0.94</sub>	Mg <sub>1.00</sub> O <sub>0.90</sub>
Suggested empirical formula of the compound using whole numbers	MgO	MgO

2.  What is the mass of magnesium in the magnesium oxide compound? How many moles of magnesium is this? Record your answers in Table 3.

The mass of magnesium in magnesium oxide is the mass that was originally measured, 0.182 g, for Trial 1. Magnesium has an atomic weight of 24.305 g/mol.

$$\frac{0.182 \text{ g Mg}}{24.305 \frac{\text{g}}{\text{mol}} \text{ Mg}} = 0.00749 \text{ mol Mg}$$

3.  Calculate the mass and number of moles of oxygen in the magnesium oxide. Record your answers in Table 3.

Subtracting the mass of magnesium from the mass of magnesium oxide for Trial 1:

$$0.295 \text{ g} - 0.182 \text{ g} = 0.113 \text{ g of oxygen}$$

$$\frac{0.113 \text{ g O}}{16.00 \frac{\text{g}}{\text{mol}} \text{ O}} = 0.00706 \text{ mol O}$$

## Lab 1: Determining the Empirical Formula of a Compound

---

4.  What is the molar ratio between magnesium and oxygen? Use this to write the experimental empirical formula of the compound using significant figures in Table 3.

$$\left(\frac{0.00749 \text{ mol Mg}}{0.00749 \text{ mol Mg}}\right) : \left(\frac{0.00706 \text{ mol O}}{0.00749 \text{ mol Mg}}\right) = 1.00 \text{ mol Mg} : 0.940 \text{ mol O}$$

5.  What is the empirical formula of the magnesium oxide compound, using whole numbers? Record your answer in Table 3.

MgO

### Analysis Questions

---

1. Using available reference sources, what is the accepted empirical formula of magnesium oxide?

The empirical formula appears to be MgO.

2. Compare your results to the accepted empirical formula for magnesium oxide. What are some sources of experimental error and what could be done to prevent such error?

Imperfect burning could be one reason. More thorough mixing with the paper clip between heating cycles could help the exposure of unreacted magnesium to air. Also, if an insufficient amount of water is added, not all the  $\text{Mg}_3\text{N}_2$  will decompose. Any sample stuck on the paper clip would introduce error as well. All of these sources of error would result in a lower final mass than expected, therefore lower oxygen to magnesium ratio.

### Synthesis Questions

---

Use available resources to help you answer the following questions.

1. If some unreacted magnesium metal remains in the crucible, explain how this will affect the empirical formula.

The final mass of the sample would be less if the magnesium had not reacted completely. Therefore, the difference between the final mass and the mass of magnesium would result in less oxygen and a higher magnesium to oxygen ratio.

2. If there is insufficient oxygen from the air, some magnesium nitride ( $\text{Mg}_3\text{N}_2$ ) will form. If this is not converted to magnesium oxide, will the ratio of oxygen to magnesium appear to be high or low? Explain your answer.

Since the same amount of magnesium would yield less  $\text{Mg}_3\text{N}_2$  than MgO, the amount of considered oxygen would be less, yielding higher magnesium to oxygen ratio.

3. Recalling the smell that you sensed, where you have smelled ammonia in the past?

One can smell ammonia on the human body after running or in urine as it is the byproduct of the metabolic process. Also it is an ingredient of household cleaners such as window cleaner.

---

## Multiple Choice Questions

---

Select the best answer or completion to each of the questions or incomplete statements below.

**1. What information, other than the number of moles of magnesium, was necessary to calculate the stoichiometric ratio between the magnesium and oxygen in the magnesium oxide?**

- A.** The number of moles of oxygen, calculated from the final loss of mass of the sample.
- B.** The number of moles of oxygen from the increased mass of the sample.
- C.** The number of moles of oxygen from the increased mass of the nitrogen and oxygen in the sample.
- D.** The number of moles of nitrogen from the increased mass of the nitrogen in the sample.

**2. Why did you have to add water and reheat the sample?**

- A.** Without water, Mg would not react with  $O_2$ .
- B.** Water is necessary to convert  $Mg(OH)_2$  to  $MgO$ , which is a byproduct of the reaction.
- C.** Water is necessary to convert to  $MgO$  from  $Mg_3N_2$ , which is a byproduct of the reaction.
- D.** Water is a catalyst of the reaction.
- E.** All of the above.

**3. Consider an experiment where we obtain  $Mg_{1.5}O_1$  as the empirical formula. Which of the following can be the reason for this kind of error?**

- A.** There was unreacted metal left at the end of the experiment.
- B.** There was unreacted  $Mg_3N_2$  left at the end of the experiment.
- C.** Some of the sample stuck on the paper clip used for mixing.
- D.** All of the above can be responsible for this kind of error.
- E.** None of the above can be responsible for this kind of error.

**4. Why may unreacted magnesium remain after the heating process?**

- A.** Magnesium is extremely hard to burn.
- B.** The most important reason is contamination of the elemental magnesium.
- C.** The presence of nitrogen is the reason, to the extent that a byproduct  $Mg_3N_2$  forms.
- D.** Insufficient amounts of oxygen gas result in leftover magnesium metal.

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## Extended Inquiry Suggestions

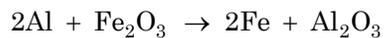
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Discuss with your students the reaction between aluminum and iron oxide. Aluminum burns less readily than magnesium. However, once it starts burning, it burns at an equally high temperature to form  $Al_2O_3$ . Therefore if aluminum is mixed with  $Fe_2O_3$  (which mix is called “thermite”) aluminum removes the oxygen from  $Fe_2O_3$  to form iron. Because of the high

## ***Lab 1: Determining the Empirical Formula of a Compound***

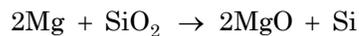
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temperature the iron melts which is why it is used to weld rails together in railroad constructions. The chemical reaction for this is:



This reaction is also known as “thermite” reaction.

Another reaction to look at is the one between magnesium and sand (silicon dioxide):



A mixture of magnesium powder and sand, when lit by a magnesium strip, burns at such a high temperature that magnesium takes the oxygen from the  $\text{SiO}_2$  to form  $\text{MgO}$  and amorphous silicon. This can be done as a demonstration experiment. Perform the experiment in a fired clay flower pot since most materials would burn through, due to the extremely high temperature.

## Lab 2: Determine the Percentage of Water in a Hydrate

### Objectives

---

Students determine the water content of a hydrated salt.

### Procedural Overview

---

Students gain experience conducting the following procedures, using a balance and a Bunsen burner:

- ◆ Carefully heat the hydrated sample in a crucible
- ◆ Take accurate measurements of the sample before and after it is thoroughly heated
- ◆ Calculate the percentage of water in the hydrate based on the measurements

### Time Requirement

---

- |                                   |             |
|-----------------------------------|-------------|
| ◆ Preparation time                | 15 minutes  |
| ◆ Pre-lab discussion and activity | 15 minutes  |
| ◆ Lab activity                    | 150 minutes |

### Materials and Equipment

---

*For each student or group:*

- |                     |   |
|---------------------|---|
| ◆ Crucible with lid | ◆ Clay triangle                                     |
| ◆ Crucible tongs    | ◆ Balance (1 per class)                             |
| ◆ Bunsen burner     | ◆ Wash bottle with deionized water                  |
| ◆ Ring stand        | ◆ Copper sulfate, $\text{CuSO}_4$ , hydrated, 4.5 g |

## **Concepts Students Should Already Know**

---

Students should be familiar with the following concepts:

- ◆ What a chemical formula means
- ◆ Balancing chemical equations

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 1: Determine the Empirical Formula of a Compound
- ◆ Lab 15a: Synthesis of Alum

## **Using Your Data Collection System**

---

Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

**Note:** There are no Tech Tips to list in this section as this activity does not use a data collection system.

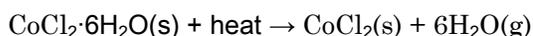
## **Background**

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Many naturally occurring or manmade salts contain water molecules bound within the crystal structure of the solid. These are called hydrated salts. The water molecules are known as “water of crystallization” or “water of hydration.”

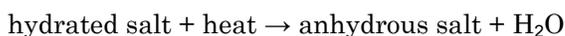
The number of moles of water will often remain in a fixed ratio to the number of moles of salt present. The formula for a hydrated salt is written as the formula of the anhydrous (without water) salt followed by a raised dot followed by the number of water molecules. For example, the formula for cobalt chloride hexahydrate is  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ .

In some cases the water is loosely bound to the salt, allowing the water to be removed by applying heat:



Some salts have their water bound so tightly that producing an anhydrous salt is nearly impossible. In the case of iron trichloride hexahydrate, the salt will decompose before all the water can be removed.

The percentage of water, by mass, in a hydrate can be determined by heating a known quantity until complete dehydration is achieved.



Dehydration results in decreased mass. The difference of the mass before and after heating makes it possible to determine the amount of water that was present in the hydrate.

Total mass of hydrated salt = mass of anhydrous salt + mass of water of hydration

The percent mass of water in the hydrated salt may be easily calculated:

$$\% \text{ water} = \frac{m_{\text{water}}}{m_{\text{hydrated salt}}} \times 100$$

Where:

$m_{\text{water}}$  = mass of water of hydration

$m_{\text{hydrated salt}}$  = total mass of hydrated salt

## Pre-Lab Activity

### Setting the stage for the activity

The hydrated salt  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}(\text{s})$  is stable at room temperature and has a different color than the dehydrated version. The hydrated crystals are blue, while the dehydrated crystals are white. This makes it easy to see when the crystals are completely dehydrated.

### Example calculation to try

You perform the same type of activity as in "Determining the Empirical Formula of a Compound." You will heat a known amount of substance and test how much the mass changes due to the heating process. From the loss of mass, you can calculate the number of water molecules that are bound to each molecule of  $\text{CuSO}_4$ . Until you determine the number of water molecules, use the formula " $\text{CuSO}_4 \cdot x\text{H}_2\text{O}(\text{s})$ ."

Analyze the following example: A 5.40 g sample of hydrated sodium sulfate,  $\text{Na}_2\text{SO}_4 \cdot x\text{H}_2\text{O}$ , was found to contain 2.34 g of anhydrous  $\text{Na}_2\text{SO}_4$ . This means that the sample had 3.06 g of water (5.40 g – 2.34 g). The water content of the sample is

$$\left( \frac{3.06 \text{ g H}_2\text{O}}{5.40 \text{ g Na}_2\text{SO}_4 \cdot x\text{H}_2\text{O}} \right) \times 100 = 56.7\% \text{ H}_2\text{O}$$

The number of water molecules bound with one molecule of  $\text{Na}_2\text{SO}_4$  can also be calculated by determining the number of moles of each and then determining the ratio of one to the other:

$$\left( \frac{2.34 \text{ g Na}_2\text{SO}_4}{142 \frac{\text{g}}{\text{mol}} \text{ Na}_2\text{SO}_4} \right) = 0.0165 \text{ mol Na}_2\text{SO}_4$$

$$\left( \frac{3.06 \text{ g H}_2\text{O}}{18.02 \frac{\text{g}}{\text{mol}} \text{ H}_2\text{O}} \right) = 0.170 \text{ mol H}_2\text{O}$$

## Lab 2: Determine the Percentage of Water in a Hydrate

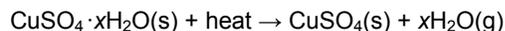
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The molar ratio between the  $\text{Na}_2\text{SO}_4$  and  $\text{H}_2\text{O}$ :

$$\left( \frac{0.0165 \text{ mol Na}_2\text{SO}_4}{0.0165 \text{ mol Na}_2\text{SO}_4} \right) : \left( \frac{0.170 \text{ mol H}_2\text{O}}{0.0165 \text{ mol Na}_2\text{SO}_4} \right) = 1.00 \text{ mol Na}_2\text{SO}_4 : 10.3 \text{ mol H}_2\text{O}$$

The stoichiometric ratio should be the nearest whole number ratio therefore, the empirical formula is  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ .

**1. Using the generic formula of  $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$ , provide an equation for the water loss of your unknown sample.**



**2. How do you know when the dehydration is complete? (Hint: Research the color of the hydrated compound and the color of the anhydrous compound.)**

The hydrated salt is blue while the anhydrous salt is white. The dehydration is complete when the substance is white.

## Lab Preparation

---

These are the materials and equipment to set up prior to the lab:

1. The hydrated  $\text{CuSO}_4$  is hygroscopic. (It attracts moisture from the air.) Try to use a freshly opened container, or make sure that the crystals are not clumped together.
2. Remind students how the Bunsen burner works and how the air flow can be controlled.

## Safety

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Add these important safety precautions to your normal laboratory procedures:

- ◆ Be very careful when using the Bunsen burner.
- ◆ Never place a hot crucible or other hot objects on a balance pan.
- ◆ Do not move the hot crucible from the clay triangle until it has cooled.
- ◆ Make sure there is nothing flammable around the setup.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

3	4	2	1	5
Heat the crucible carefully until the crystals turn white. Allow the crucible to cool. Measure the mass of the crucible.	Heat the crucible and cool it. Repeat this until the change in mass is less than 10 mg.	Measure the mass of the empty crucible and the crucible containing the $\text{CuSO}_4 \cdot x\text{H}_2\text{O}_{(s)}$ sample.	Make sure the crucible is clean and that there are no defects or contaminants.	Calculate the water content of the sample.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** This activity does not use a data collection system, so there are no Tech Tips (indicated by the symbol "♦" and a superscripted number following a step).

### Set Up

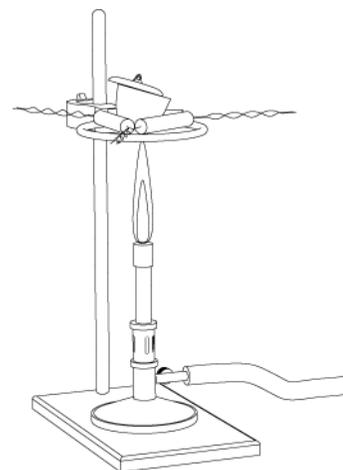
- Obtain a crucible and lid, and inspect the crucible for cracks, nicks, and other defects.

**Note:** Replace a defective crucible.

- Measure and record the mass of the crucible and lid in Table 1.
- Assuming there are contaminations in the crucible that remain even after heating, would such contaminations falsify the data? If so, why would they?

Since the contamination does not lose mass when heated, it would behave the same as the crucible itself. Therefore it can be considered part of the crucible and would not falsify the data.

- Place the crucible and lid on the clay triangle over the Bunsen burner.



**Note:** For the rest of the experiment handle the crucible and lid using only crucible tongs. Also, do not set the crucible on the lab bench or it may crack or become contaminated.

## Lab 2: Determine the Percentage of Water in a Hydrate

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5.  Why do you have to use tongs to hold the lid?

Touching the lid with bare hands would leave a residue of grease on the lid. The grease would add to the mass of the crucible. Because it would burn off, it would result in a higher mass for the evaporated water. Also, the crucible may be hot and cause injury.

### Collect Data

#### Obtain data for the empty crucible

6.  The steps for heating, cooling, and measuring the mass of the crucible are as follows:
- Heat the crucible with a gentle flame for 5 minutes by moving the burner around the bottom of the crucible.
  - After the bottom of the crucible has become red-hot, increase the flame by allowing more air into the burner.
  - Continue moving the burner around the bottom of the crucible.
  - Heat the crucible for 10 to 12 minutes.
  - Allow the crucible to cool to room temperature.
  - After the crucible has cooled, measure and record the mass of the “fired” crucible and lid in Table 1.

Table 1: Mass of the empty crucible measured to the nearest milligram

Measurement	Trial 1	Trial 2
Crucible and lid before heating (g)	27.494	29.142
Crucible and lid after the first heating (g)	27.426	29.039
Crucible and lid after the second heating (g)	27.423	29.038
Crucible and lid after the third heating (g)		
Crucible and lid after the fourth heating (g)		

7.  Repeat the steps above for heating, cooling, and measuring the mass of the crucible and lid until you have two readings for the mass that are within 10 mg of each other.
8.  Copy the last measurement into both Table 1 and Table 3.

#### Obtain data for the crucible and sample

9.  Place between 1.75 g and 2.25 g of hydrated copper sulfate in the crucible.
10.  Measure the mass of the crucible, lid, and hydrated copper sulfate to the nearest milligram and record it in Table 3.

- 11.**  Return the crucible with the sample and lid to the clay triangle over the Bunsen burner.
- 12.**  Gently heat the sample for 2 to 3 minutes by moving the burner around the bottom of the crucible.
- 13.**  Increase the heat and continue heating for 3 minutes.
- Note:** Remember that the top third of the flame is the hottest.
- 14.**  Carefully lifting the lid with the tongs, check the sample. If the sample hasn't turned white, replace the lid and continue heating for a few more minutes
- 15.**  With the lid in place, remove the heat source and allow the crucible to return to room temperature.
- 16.**  Measure the mass of the crucible, lid, and sample, and record it to the nearest milligram in Table 2.

Table 2: Mass of the crucible and copper sulfate salt after heating, measured to the nearest milligram

Measurement	Trial 1	Trial 2
Crucible, lid, and salt after the first heating (g)	27.426	29.039
Crucible, lid, and salt after the second heating (g)	27.423	29.038
Crucible, lid, and salt after the third heating (g)		
Crucible, lid, and salt after the fourth heating (g)		

- 17.**  Why should the lid remain on the crucible?
- If the anhydrous salt is exposed to open air, it will absorb moisture from the air.
- 18.**  Reheat the crucible and sample at high heat for 5 minutes. Allow the crucible and sample to cool, then measure and record the mass in Table 2 as before.
- 19.**  Continue to reheat the crucible and sample until you obtain two consecutive readings that are within 10 mg of each other.
- 20.**  Copy the last measurement into both Table 2 and Table 3.
- 21.**  What would you observe if you added a few drops of deionized water to the anhydrous sample?
- The sample would become hydrated again and would turn blue.
- 22.**  Squeeze a few drops of deionized water into the anhydrous sample to check your prediction.

## Lab 2: Determine the Percentage of Water in a Hydrate

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23.  Dispose of the sample in the appropriate container.
24.  Repeat the procedure with a new sample of hydrated copper sulfate (between 1.75 g and 2.25 g). Record the results for Trial 2 in the specified tables and column.
25.  Wash the crucible with soap. Then rinse 3 times with tap water to clean the crucible of copper sulfate residue.
26.  After rinsing three times with tap water, rinse again with deionized water.
27.  Clean up according to your teacher's instructions.

### Data Analysis

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1.  Calculate a) the mass of the hydrated salt, b) the mass of the anhydrous salt, c) the mass of the water lost, d) the percentage of water in the hydrated salt, and e) the average percentage of water in the hydrated salt. Record your answers in Table 3.

Shown for Trial 1:

a) Mass of the hydrated salt:  $29.327 \text{ g} - 27.423 \text{ g} = 1.904 \text{ g}$

b) Mass of the anhydrous salt:  $28.637 \text{ g} - 27.423 \text{ g} = 1.214 \text{ g}$

c) Mass of the water lost:  $1.904 \text{ g} - 1.214 \text{ g} = 0.690 \text{ g}$

d) Percent water in the hydrated salt:  $\left( \frac{0.690 \text{ g H}_2\text{O}}{1.904 \text{ g Cu}_2\text{SO}_4 \cdot x\text{H}_2\text{O}} \right) \times 100 = 36.2\% \text{ H}_2\text{O}$

e) Average percent water from trials 1 and 2:  $(36.2\% + 32.4\%)/2 = 34.3\%$

Table 3: Determine the percent water in the sample

Parameter	Trial 1	Trial 2
Mass of crucible, lid, and hydrated salt (g)	29.327	31.106
Mass of crucible, lid, and anhydrous salt (g)	28.637	30.436
Mass of crucible and lid (g)	27.423	29.038
Mass of hydrated salt (g)	1.904	2.068
Mass of anhydrous salt (g)	1.214	1.398
Mass of water lost (g)	0.690	0.670
Percent of water in the hydrated salt (%)	36.2	32.4
Average percent water in the hydrated salt (%)	34.3	
Number of moles of anhydrous salt (mol)	$7.61 \times 10^{-3}$	$8.76 \times 10^{-3}$
Number of moles of water lost (mol)	$3.83 \times 10^{-2}$	$3.72 \times 10^{-2}$
Ratio between salt and water	1:5.040	1:4.30
Formula of hydrated salt	$\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$	

2.  Record the formula weight (or molar mass) of anhydrous copper(II) sulfate and the formula weight of water below:

$$FW_{\text{anhydrous copper(II) sulfate}} \text{ (g/mol): } \underline{\hspace{2cm} 159.61 \hspace{2cm}}$$

$$FW_{\text{water}} \text{ (g/mol): } \underline{\hspace{2cm} 18.02 \hspace{2cm}}$$

3.  Calculate the number of moles of anhydrous salt used for each trial and record the values in Table 3.

Shown for Trial 1:

$$\left( \frac{1.214 \text{ g CuSO}_4}{159.62 \frac{\text{g}}{\text{mol}} \text{ CuSO}_4} \right) = 7.609 \times 10^{-3} \text{ mol CuSO}_4$$

4.  Calculate the number of moles of water lost after heating for each trial and record the values in Table 3.

Shown for Trial 1:

$$\left( \frac{0.6900 \text{ g H}_2\text{O}}{18.02 \frac{\text{g}}{\text{mol}} \text{ H}_2\text{O}} \right) = 3.833 \times 10^{-2} \text{ mol H}_2\text{O}$$

## Lab 2: Determine the Percentage of Water in a Hydrate

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5. □ How many water molecules are bound with one molecule of  $\text{CuSO}_4$ , that is, what is the molar ratio between  $\text{CuSO}_4$  and  $\text{H}_2\text{O}$  for each trial? Show your work and record the values in Table 3.

Shown for Trial 1:

$$\left(\frac{0.00761 \text{ mol CuSO}_4}{0.00761 \text{ mol CuSO}_4}\right) : \left(\frac{0.0383 \text{ mol H}_2\text{O}}{0.00761 \text{ mol CuSO}_4}\right) = 1.00 \text{ mol CuSO}_4 : 5.03 \text{ mol H}_2\text{O}$$

The stoichiometric ratio should be the nearest whole number ratio, therefore the formula is  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ .

### Analysis Questions

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1. If your results do not yield a molar ratio of whole numbers for the  $\text{CuSO}_4$  and  $\text{H}_2\text{O}$ , is it justified to round the numbers to the nearest whole numbers? Explain.

The molar ratio between  $\text{CuSO}_4$  and  $\text{H}_2\text{O}$  must be a ratio between whole numbers. Deviation from that is a result of experimental error, so rounding is justified.

2. Why would leaving the dehydrated sample exposed to air introduce a significant error?

Dehydrated  $\text{CuSO}_4$  is very hygroscopic. That is, it absorbs moisture from air. This increases the mass of the sample.

3. Why must the crucible cool to room temperature before taking any mass measurement?

The balance could be damaged if anything significantly higher than room temperature is placed on the balance. It also increases the chance of someone getting burned by trying to measure the mass of items that are hot. Additionally, convection currents will cause an unstable reading (the reading fluctuates).

4. If the crucible was heated too rapidly and some sample material spattered out, how would this affect the final percent water calculations? Explain.

Slower heating assures that water separates from the hydrate without losing any of the salt. If some of the salt is lost, the mass of the lost salt will be included in the mass of the water lost through heating. This will result in a larger proportion of water in the hydrated salt, leading to an erroneously high ratio of water.

5. What would you observe if you removed the lid from the crucible while the sample was being heated and held a cold watch glass over the crucible?

Water being eliminated from the sample would condense on the cold surface of the watch glass.

### Synthesis Questions

---

Use available resources to help you answer the following questions.

1. Using available reference sources, what is the empirical formula of hydrated  $\text{CuSO}_4$ ?

The empirical formula is  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ .

**2. When heated, why do  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  crystals "melt" at a much lower temperature than the ionic compound  $\text{Na}_2\text{SO}_4$  normally does (it occurs below the boiling point of water)? (Hint: There is  $\text{Na}_2\text{SO}_4$  and water present in the crystals)**

The crystals contain sufficient water to dissolve the substance,  $\text{Na}_2\text{SO}_4$ . The heating actually produces a solution and does not melt the crystals.

**3. Are the hydrated and dehydrated forms of a substance two different chemical substances? (Hint: What happened when you added water drops to the dehydrated crystals?)**

No, they are the same chemical substance. Adding water reverses the process of losing water. Since the process is reversible it is a physical change, not a chemical change.

### Multiple Choice Questions

Select the best answer or completion to each of the questions or incomplete statements below.

**1. What is the molar ratio between  $\text{CuSO}_4$  and its water content in the hydrated form?**

- A.** It cannot be calculated. Only the percent water content can.
- B.** It can be calculated from the number of moles of  $\text{CuSO}_4$  remaining after heating and from the loss of mass, which can be converted to the number of moles of water.
- C.** It depends on how high the temperature is during the heating process.
- D.** It is not a fixed ratio and varies between samples.

**2. In an analysis of a hydrated  $\text{CuSO}_4$  sample, what could the source of error be for a lower than actual water to  $\text{CuSO}_4$  ratio?**

- A.** Heating too quickly resulted in spattering and loss of some of the sample.
- B.** There was partial decomposition of the already anhydrous  $\text{CuSO}_4$ .
- C.** The sample did not completely lose its water content.
- D.** None of the above.
- E.** All of the above.

**3. How are anhydrous and hydrated  $\text{CuSO}_4$  related chemically?**

- A.** They are different compounds with different chemical and physical properties.
- B.** They are the same compound with somewhat different physical properties.
- C.** They are different but similar to other hydrated and anhydrous compounds.

## Lab 2: Determine the Percentage of Water in a Hydrate

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- 4.** For what reason might you *not* obtain a ratio of whole numbers for the molar ratio between the  $\text{CuSO}_4$  and  $\text{H}_2\text{O}$ ?
- A.** The composition of the hydrated compound is not constant and it varies between samples.
  - B.** The ratio between the  $\text{CuSO}_4$  and  $\text{H}_2\text{O}$  does not have to be a ratio between whole numbers.
  - C.** The most likely reason is experimental error.
  - D.** None of the above options can account for the deviation.

### Extended Inquiry Suggestions

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Discuss with students how  $\text{CoCl}_2$  is used as an "indicator" in desiccators:

Desiccators are closed glass containers with drying agents. When a wet sample is placed in a desiccator, the drying agent absorbs moisture and the wet sample dries. In the indicator, traces of  $\text{CoCl}_2$  start to absorb moisture when the drying agent can no longer absorb moisture. Since  $\text{CoCl}_2$  is blue in its anhydrous state and pink in its hydrated form, pink indicates that the drying agent needs to be replaced. As long as the  $\text{CoCl}_2$  remains blue, the drying agent is still capable of absorbing moisture.

## Lab 3: Determine the Molar Mass of a Volatile Liquid

### Objectives

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Students determine the molar mass of an unknown volatile liquid at the boiling temperature of water and at atmospheric pressure.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Using hot and cold water baths to evaporate and condense an unknown volatile liquid, measuring the mass of a flask with and without the condensed unknown liquid
- ◆ Obtaining the mass, pressure, temperature, and volume of the evaporated volatile liquid
- ◆ Using the Ideal Gas Law to calculate the molar mass of the volatile liquid

### Time Requirement

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◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

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#### *For each student or group:*

- |   |  |
|---|--|
| ◆ Data collection system                      | ◆ Ring stand                                 |
| ◆ Stainless steel temperature sensor          | ◆ Clamp (2) <sup>1</sup>                     |
| ◆ Pressure sensor                             | ◆ Unknown volatile liquid, 8 mL <sup>2</sup> |
| ◆ Beaker (2), 400-mL                          | ◆ Aluminum foil, about 4-cm by 4-cm          |
| ◆ Erlenmeyer flask, 125-mL                    | ◆ Paper towel (2-3 sheets)                   |
| ◆ Graduated cylinder, 100-mL                  | ◆ Dropper                                    |
| ◆ Hotplate with magnetic stirrer and stir bar | ◆ Water, 600 mL                              |
| ◆ Balance (1 per class)                       |  |

<sup>1</sup> To hold the Erlenmeyer flask and temperature sensor

<sup>2</sup> The unknown volatile liquid is acetone. Refer to the Lab Preparation section for details or for alternative liquids.

## **Concepts Students Should Already Know**

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Students should be familiar with the following concepts:

- ◆ Ideal Gas Law
- ◆ Properties of gases and liquids
- ◆ Relationship between phase changes and heat

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 5: Molar Volume of a Gas
- ◆ Lab 27: Identifying an Unknown Metal
- ◆ Lab 29: Exploring Gas Laws

## **Using Your Data Collection System**

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting multiple sensors to your data collection system ◆<sup>(2.2)</sup>
- ◆ Changing the units of a measurement ◆<sup>(5.3)</sup>
- ◆ Monitor live data without recording ◆<sup>(6.1)</sup>
- ◆ Displaying data in a digits display ◆<sup>(7.3.1)</sup>
- ◆ Adding a variable to a digits display ◆<sup>(7.3.2)</sup>
- ◆ Saving your experiment ◆<sup>(11.1)</sup>

## **Background**

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The Dumas method of molar mass determination involves measuring the mass of the condensed vapor of a volatile liquid. This is done under controlled conditions, with known volume, pressure, and temperature. A small sample of liquid with a low boiling point is added to an Erlenmeyer flask with a pre-measured mass. The Erlenmeyer flask is submerged in a hot water bath. As the sample evaporates, air is flushed from the flask. When the entire sample has evaporated, the flask contains only the vapor of the unknown substance. At that point, the flask is cooled, and the vapor condenses. Its mass may be determined by re-measuring the mass of the flask.

The Ideal Gas Law is given by the equation:

$$pV = nRT$$

where:

$p$  = the pressure of the gas (Pa)

$V$  = the volume of the gas sample ( $\text{m}^3$ )

$n$  = the number of moles of gas present (mol)

$T$  = the temperature of the sample (K)

$R$  = the Ideal Gas Law constant, which is  $8.314 \text{ J mol}^{-1} \text{ K}^{-1}$

This relationship describes the behavior of gases very well at ordinary pressures and moderate temperatures.

The number of moles of a substance is equal to the mass of the substance divided by the mass of a mole of that substance:

$$n = \frac{m}{FW}$$

where:

$m$  = the mass of the sample (g)

$FW$  = the molar mass, or formula weight, of the substance (g/mol)

Substituting this relationship into the Ideal Gas Law:

$$pV = \frac{mRT}{FW}$$

and rearranging to isolate the molar mass yields

$$FW = \frac{mRT}{pV}$$

## Pre-Lab Activity

### Setting the stage for the activity

Review the properties of liquids and gases. Discuss how the heat gained by the system affects the motion of molecules, ultimately resulting in phase transition.

If possible, perform the following demonstration. Place an empty soda can on a hotplate and pour about 15 mL of water into it. Bring the water to boil and then quickly, with a pair of tongs, place the can into a cold water bath upside down. The can should be crushed.

The water vapor displaces air. When cooled, the water vapor molecules are removed from the gas phase instantaneously, resulting in a significantly lowered pressure in the can. The excess external pressure crushes the can.

Discuss with your teacher what happens in the can while the water is boiling and after it is suddenly cooled. The lab activity is an analogous experiment. You will replace the air in an

### Lab 3: Determine the Molar Mass of a Volatile Liquid

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Erlenmeyer flask with the vapor of a volatile liquid. When cooled, the vapors will condense in the Erlenmeyer flask, and air will rush into the Erlenmeyer flask to compensate for the pressure drop. Unlike the can made of thin aluminum, the strong glass of the Erlenmeyer flask will not crush.

#### Example calculation to try

The mass of an empty Erlenmeyer flask is measured and found to be 25.334 g. The flask is covered with a piece of aluminum foil which has several pin holes and placed in a water bath and kept at 80.0 °C. An unknown liquid is added to the flask and boiled. Care is taken to assure that there is always liquid in the flask. Finally, after about 5 minutes of boiling, allow all the liquid to evaporate at which stage there are only molecules of the unknown substance in the gas phase and there is no liquid left. Then the flask is placed immediately into a cold water bath, wiped dry, and the mass is measured very quickly.

The Erlenmeyer flask with the condensed drops of the liquid is found to be 25.437 g. The liquid in the flask is 0.103 g (25.437 g – 25.334 g).

The flask is filled to the rim with water and the volume of the water measured. The volume of the water is found to be 65.0 mL. The pressure sensor shows 101 kPa of atmospheric pressure.

Necessary unit conversions for doing the calculations:

$$101 \text{ kPa} = 1.01 \times 10^5 \text{ Pa} = 1.01 \times 10^5 \frac{\text{N}}{\text{m}^2}$$

$$65.0 \text{ mL} = 6.50 \times 10^{-5} \text{ m}^3$$

$$80.0 \text{ }^\circ\text{C} = 353 \text{ K}$$

Applying the Ideal Gas Law:

$$pV = nRT$$

$$pV = \frac{m}{FW} RT$$

Solving that for FW:

$$FW = \frac{mRT}{pV}$$

Applying the relevant data to the equation:

$$FW = \frac{(0.103 \text{ g}) \left( 8.314 \frac{\text{Nm}}{\text{mol K}} \right) (353 \text{ K})}{\left( 1.01 \times 10^5 \frac{\text{N}}{\text{m}^2} \right) (6.50 \times 10^{-5} \text{ m}^3)}$$

$$FW = 46.0 \frac{\text{g}}{\text{mol}}$$

#### 1. What happens to the molecules of a liquid if the liquid is heated?

The average velocity of the molecules increases. The average kinetic energy of the molecules also increases.

**2. What happens if a molecule of liquid possesses enough kinetic energy to break the bonds to other molecules?**

It leaves the liquid phase and enters the gas phase.

**3. Under what circumstances (pressure and temperature) is the Ideal Gas Law valid?**

When there is no substantial interaction between molecules, which is generally true under low pressure and high temperature conditions.

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## Lab Preparation

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These are the materials and equipment to set up prior to the lab:

1. Use acetone as the "unknown liquid." Its formula weight is 58 g/mol. Its boiling point is 57 °C (330 K). For safety reasons, if you decide to use other liquids, they should not have a boiling point lower than 40 °C.
2. Inspect the Erlenmeyer flasks for cracks or other defects.

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## Safety

---

Add these important safety precautions to your normal laboratory procedures:

- ◆ If you feel dizzy or light-headed during the lab, notify your instructor immediately.
- ◆ Under no circumstances is an open flame (such as a Bunsen burner) allowed in the lab during the experiment. Most volatile liquids are extremely flammable.
- ◆ If the skin is exposed to a volatile liquid, rinse the exposed surface thoroughly with running water. Some volatile liquids irritate the skin.
- ◆ Dispose of liquids properly. Unused liquids must not be poured down the drain.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

1	3	5	4	2
Measure the empty Erlenmeyer flask to the nearest milligram.	Continue adding liquid, as it boils and evaporates, for about 5 minutes.	Measure the Erlenmeyer flask with the condensed liquid. Calculate the formula weight.	Cool the Erlenmeyer flask in a beaker of cold water. Wipe the Erlenmeyer flask dry.	Mount the Erlenmeyer flask in a water bath. Heat the water bath and put 2 mL of the unknown liquid into the flask.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "♦" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

- Start a new experiment on the data collection system. ♦<sup>(1,2)</sup>
- Connect the stainless steel temperature sensor and the pressure sensor to the data collection system. ♦<sup>(2,2)</sup>
- Display temperature and pressure in a digits display. ♦<sup>(7.3.1)(7.3.2)</sup>
- Prepare a 400-mL beaker by filling it with about 300 mL of water.

5.  Place the 400-mL beaker of water on the hotplate with the magnetic stirrer, and begin heating the water to 85 °C.

6.  Clamp the stainless steel temperature sensor to the ring stand, and immerse the tip in the water.

**Note:** Do not allow the temperature sensor to touch the sides or bottom of the flask.

7.  Place the stirring bar into the water and turn on the magnetic stirrer.

8.  Place a small piece of aluminum foil over the opening of the 125-mL Erlenmeyer flask.

9.  Poke a small hole in the aluminum foil.

10.  Measure the mass of the empty Erlenmeyer flask and foil, and record the mass to the nearest milligram in Table 1.

11.  Mount the Erlenmeyer flask on the ring stand with a clamp so that it is immersed in the water bath as far as possible without allowing water to get into the flask.

12.  Why is it important to immerse the flask as far as possible into the water bath?

Because the surface of the flask exposed to air will have a lower temperature, the vapors will have a lower temperature than the known temperature of the water bath. This introduces error when using the Ideal Gas Law.

13.  Fill a second 400-mL beaker with about 300 mL of cold water.

### Collect Data

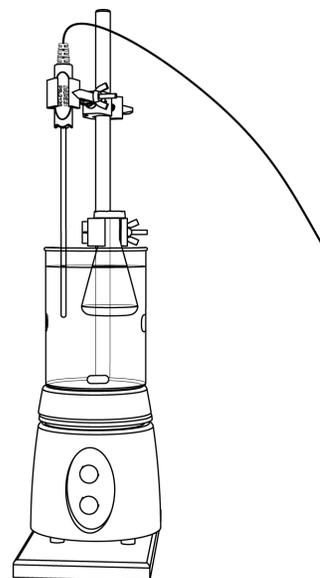
14.  With the dropper, add 1 to 2 mL of the unknown liquid into the Erlenmeyer flask through the hole in the aluminum foil.

15.  Monitor live data without recording.  $\diamond^{(6.1)}$

**Note:** Maintain the temperature of the bath close to 85 °C.

**Note:** Make sure that there is always liquid in the Erlenmeyer flask.

16.  When the sample is almost boiled away, use the dropper to add 1 to 2 mL more of the unknown liquid through the hole in the aluminum foil.



### Lab 3: Determine the Molar Mass of a Volatile Liquid

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17.  What was in the Erlenmeyer flask before the experiment and what do you think is in the Erlenmeyer flask at this point?

Before the experiment it was filled with air. At this point the air is flushed out and it is filled with the vapor of the unknown liquid.

18.  After adding and evaporating 2 mL of the unknown liquid two more times, with the water bath temperature at least 85 °C, record the temperature of the bath in Table 1.

19.  Change the units of the pressure measurement to Pascal. ♦<sup>(5.3)</sup>

20.  Measure the atmospheric pressure in the laboratory in Pascals and record the value in Table 1.

**Important:** Make sure that all the liquid is evaporated inside of the Erlenmeyer flask before removing it from the hot water bath. If there is liquid left, it will add to the mass of the liquid formed from the condensed vapor, introducing error.

21.  With the aluminum foil still in place, remove the Erlenmeyer flask and place it in the beaker with cold water for 15 to 20 seconds.

22.  Wipe the outside of the Erlenmeyer flask completely dry with paper towels.

**Important:** The flask must be completely dry. Any amount of water on the flask introduces significant error.

23.  Quickly measure the mass of the flask with the condensed liquid to the nearest milligram, and record it in Table 1.

24.  After drying, obtain the mass of the Erlenmeyer flask and aluminum foil, immerse the Erlenmeyer flask in the hot water bath, and repeat these steps and those in the Collect Data section two more times. You should have three sets of measurements.

25.  Stop monitoring live data without recording. ♦<sup>(6.1)</sup>

26.  Fill the Erlenmeyer flask to the top with water.

27.  Measure the volume of water in the Erlenmeyer flask with a graduated cylinder and record it in Table 1.

28.  Save your experiment ♦<sup>(11.1)</sup> and clean up according to your teacher's instructions.

## Data Analysis

Table 1: Measurements and calculated results

Parameters	Trial 1	Trial 2	Trial 3
Mass of dry Erlenmeyer flask and foil (g)	32.303	32.192	32.273
Mass of Erlenmeyer flask, foil and condensed vapor (g)	32.427	32.329	32.397
Mass of condensed vapor (g)	0.124	0.137	0.124
Temperature of water bath (°C)	94.3	94.0	94.0
Temperature of water bath (K)	367.5	367.2	367.2
Volume of Erlenmeyer flask (mL)	67.0	67.0	67.0
Volume of Erlenmeyer flask (m <sup>3</sup> )	$6.70 \times 10^{-5}$	$6.70 \times 10^{-5}$	$6.70 \times 10^{-5}$
Atmospheric pressure in lab (Pa)	101176	101168	101188
Molar mass of unknown liquid (g/mol)	55.9	61.7	55.8
Average molar mass of unknown liquid (g/mol)	57.8		

- Calculate the mass of the condensed vapor of the unknown volatile liquid and record the value in Table 1 for each trial.

Calculations for Trial 1:

$$32.427 \text{ g} - 32.303 \text{ g} = 0.124 \text{ g of condensed vapor}$$

- Convert the temperature of the water bath to Kelvin and convert the flask volume from milliliters to cubic meters. Record the values in Table 1 for each trial.

Calculations for Trial 1:

$$\text{To convert from degrees Celsius to Kelvin, add 273: } 94.3 \text{ }^\circ\text{C} + 273 = 367 \text{ K}$$

To convert from milliliters to cubic meters, multiply by  $1 \times 10^{-6} \text{ m}^3/\text{mL}$ :

$$67.0 \text{ mL} \times (1 \times 10^{-6} \text{ m}^3/\text{mL}) = 67.0 \times 10^{-6} \text{ m}^3 = 6.70 \times 10^{-5} \text{ m}^3$$

### Lab 3: Determine the Molar Mass of a Volatile Liquid

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3.  Calculate the molar mass of the unknown liquid for each trial and record the values in Table 1. Calculate the average molar mass for the three trials.

Calculations for Trial 1:

$$pV = \frac{mRT}{FW}$$

$$FW = \frac{mRT}{pV}$$

$$FW = \frac{(0.124 \text{ g}) \left( 8.314 \frac{\text{Nm}}{\text{mol K}} \right) (367 \text{ K})}{\left( 1.01 \times 10^5 \frac{\text{N}}{\text{m}^2} \right) (6.70 \times 10^{-5} \text{ m}^3)}$$

$$FW = 55.9 \frac{\text{g}}{\text{mol}}$$

To calculate the average:  $(55.9 \text{ g/mol} + 61.7 \text{ g/mol} + 55.8 \text{ g/mol}) / 3 = 57.8 \text{ g/mol}$

### Analysis Questions

---

1. What happens to the molecules of the unknown substance that are in the gas phase when the Erlenmeyer flask is cooling? What is going to be in the gas phase after it has cooled?

The molecules leave the gas phase and enter the liquid phase. Molecules from the air from outside the Erlenmeyer flask rush into the Erlenmeyer flask to make up for the absence of gas molecules.

2. Why is it necessary to cool the vapor?

The mass of gas molecules does not register on the balance as the atmospheric pressure is the baseline, and these molecules are at atmospheric pressure. Objects with higher density than air, such as the liquid drops of the unknown substance, will be exposed to higher gravitational pull from Earth. Therefore they will register on the balance.

3. What is the purpose of the aluminum foil?

The foil helps to keep air from getting in and molecules of the unknown substance from getting out.

4. What are some of the sources of experimental error?

Error could arise if not all of the air is flushed out of the Erlenmeyer flask, if some of the liquid evaporates after the flask is cooled but before the mass of the flask is measured, or if water is left on the outside of the Erlenmeyer flask when its mass is measured.

### Synthesis Questions

---

Use available resources to help you answer the following questions.

1. Why would the method in this lab activity not work for an unknown liquid with a boiling point of 186 °C?

The liquid would not evaporate sufficiently to displace air when its temperature is raised close to the boiling point of water (100 °C).

**2. How would you modify the setup of this activity to measure the formula weight of an unknown liquid with a boiling point of 186 °C?**

A liquid with a boiling point above 186 °C would be needed instead of a hot water bath.

**3. Assume you have to determine the formula weight of a liquid with a boiling point slightly above room temperature. At which step in the experiment would a significant error be introduced? (Hint: Liquids with such low boiling point evaporate very quickly.)**

Between cooling the Erlenmeyer flask with the condensed vapor and measuring the mass, a significant amount of liquid could evaporate at room temperature. This means a loss in the mass of the liquid, which introduces an error when measuring its mass.

**4. How would you minimize the error presented in the previous question?**

Cooling the Erlenmeyer flask in an ice bath lowers the temperature of the flask. Assuring a cool temperature reduces the evaporation during the time it takes to measure the mass.

## Multiple Choice Questions

Select the best answer or completion to each of the questions or incomplete statements below.

**1. In an experiment, we obtained a *greater* formula weight than expected. Which of the following reasons can account for the error?**

- A. Not all of the air was flushed out and replaced with molecules of the unknown substance.
- B. The Erlenmeyer flask was not completely dry.
- C. Not all of the liquid evaporated before measuring the mass.
- D. Only B and C are true.
- E. Only A and B are true.

**2. A substance has a boiling point of 28 °C. Determination of its formula weight resulted in a significant error. What could be the most obvious reason for this error?**

- A. Contamination of the liquid.
- B. Inaccuracy of the pressure and temperature sensors.
- C. High humidity content of the air.
- D. Too high a boiling point, which results in significant evaporation between cooling the Erlenmeyer flask and measuring its mass on the balance.
- E. Too low a boiling point, which results in significant evaporation between cooling the Erlenmeyer flask and measuring its mass on the balance.

**3. Why is the method in this activity unable to determine the formula weight of substances with a much higher boiling point than water?**

- A. Because they evaporate too quickly, introducing error.

### Lab 3: Determine the Molar Mass of a Volatile Liquid

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- B.** Because they do not evaporate enough to flush the air out and fill the Erlenmeyer flask.
- C.** Because those liquids usually are contaminated.
- D.** The question is misleading. The formula weight of such substances can be determined very accurately with this method.
- E.** Because of much greater viscosity of such substances, they do not evaporate enough to flush the air out and fill the Erlenmeyer flask.
- 4.** Does it matter how much of the liquid evaporates during the experiment and why?
- A.** No, what matters is that at the end, the Erlenmeyer flask is filled with vapor which will condense when cooled.
- B.** Yes, the mass of the liquid that was evaporated is in the Ideal Gas Law equation.
- C.** It depends on the liquid. Liquids with a high boiling point essentially do not evaporate at all so the loss would not matter.
- D.** The Ideal Gas Law does depend on the mass of the substance, which makes the amount of liquid placed into the flask important.
- E.** All of the above.

### Extended Inquiry Suggestions

---

Have students determine the density of the vapor of the unknown substance given the following information:

$$pV = \frac{m}{FW}RT$$

$$\frac{pFW}{RT} = \frac{m}{V} = d$$

$$d = \frac{pFW}{RT}$$

Density of the unknown volatile liquid vapor obtained using students' experimental results

Parameters	Trial 1	Trial 2	Trial 3
Volume of Erlenmeyer flask (m <sup>3</sup> )	6.70 × 10 <sup>-5</sup>	6.70 × 10 <sup>-5</sup>	6.70 × 10 <sup>-5</sup>
Mass of condensed vapor (g)	0.124	0.137	0.124
Density (kg/m <sup>3</sup> )	1.85	2.04	1.85
Average density (kg/m <sup>3</sup> )	1.92		

## Lab 4: Molecular Weight by Freezing Point Depression

### Objectives

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Students determine the molecular weight of a compound by measuring the freezing point depression of a solution.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Measuring the freezing point of a solvent
- ◆ Making a solution with a solvent that has a higher freezing point than room temperature
- ◆ Using phase diagrams and obtaining freezing point curves

### Time Requirement

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◆ Preparation time	20 minutes
◆ Pre-lab discussion and activity	30 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

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#### *For each student or group:*

- |                                      |   |
|--------------------------------------|---|
| ◆ Data collection system             | ◆ Hotplate  |
| ◆ Stainless steel temperature sensor | ◆ Stirring bar  |
| ◆ Erlenmeyer flask, 250-mL           | ◆ Clamp (2), utility  |
| ◆ Beaker, 400-mL                     | ◆ Lauric acid, $\text{CH}_3(\text{CH}_2)_{10}\text{COOH}$ , 8 g |
| ◆ Test tube, 20-mL                   | ◆ Unknown solute, 0.5 g <sup>2</sup>                            |
| ◆ Copper wire coil <sup>1</sup>      | ◆ Water, 300 mL   |
| ◆ Ring stand                         |   |

<sup>1</sup> To prepare the copper wire coil, refer to the Lab Preparation section.

<sup>2</sup> To prepare the unknown solute sample, which is benzoic acid,  $\text{C}_6\text{H}_5\text{COOH}$ , refer to the Lab Preparation section.

## **Concepts Students Should Already Know**

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Students should be familiar with the following concepts:

- ◆ Colligative properties
- ◆ Definition of electrolytes
- ◆ Molality concentration
- ◆ Phase diagrams, heat of vaporization, heat of fusion

## **Related Labs in This Guide**

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There are no labs conceptually related to this one.

## **Using Your Data Collection System**

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a sensor to the data collection system ◆<sup>(2.1)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>
- ◆ Adjusting the scale of the graph ◆<sup>(7.1.2)</sup>
- ◆ Display two data runs in a graph. ◆<sup>(7.1.3)</sup>
- ◆ Print the graph. ◆<sup>(11.2)</sup>
- ◆ Saving your experiment ◆<sup>(11.1)</sup>

## **Background**

---

Understanding the process by which solutions are made helps one understand colligative properties. The solution process involves breaking bonds and forming new bonds. The bonds between ions and molecules in solid phase have to be broken (ionic bonds in ionic lattices, or intermolecular forces between molecules in molecular compounds) and some bonds among the solvent molecules have to be broken to accommodate the dissolved particles. These steps require energy.

The ions formed or molecules freed form bonds with the free solvent molecules. This process releases energy. The result of these three energy changes is the “heat of solution,” which can be positive or negative. A positive energy change indicates an endothermic solution process; a negative energy change indicates an exothermic process.

Because of the bonds formed between the solute and solvent molecules, a solution has fewer “free” solvent molecules available to escape from the liquid phase into the gas phase. This important consequence means that the vapor of the solvent builds a lower vapor pressure over the solution. Use the phase diagram to understand the effect of the lower vapor pressure.

The top diagram applies to the states of matter of a substance in general. The bottom diagram compares the freezing and boiling points of a solution and the pure solvent.

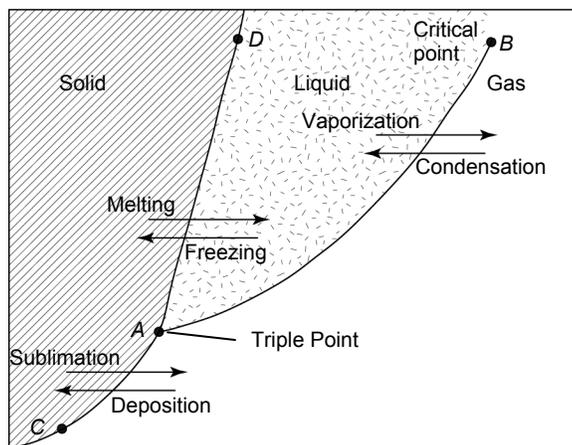


Figure 1: Phase diagram

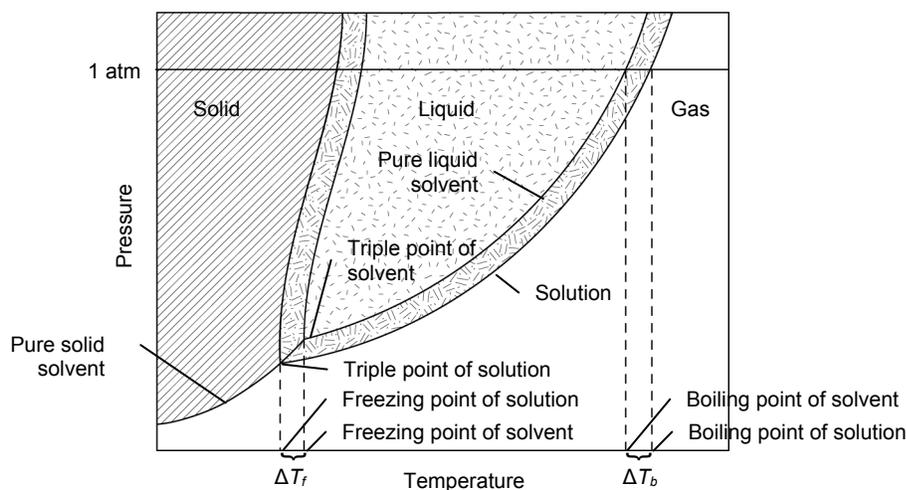


Figure 2: Phase diagram comparing a solution to the pure solvent

The phase diagram shows how the vapor pressure over a solution is lower at any given temperature compared with the solvent. Because of the lower vapor pressure over solutions, the vapor pressure reaches the atmospheric pressure at a higher temperature than the boiling point and at a lower temperature than the freezing point of the pure solvent. Compared with pure solvent, the freezing point of solutions is lower and the boiling point of solutions is higher.

Note that the standard freezing point phase transition occurs at atmospheric pressure and the standard boiling point phase transition occurs at atmospheric pressure.

## Lab 4: Molecular Weight by Freezing Point Depression

The respective temperature changes are referred to as the freezing point depression ( $\Delta T_f$ ) and boiling point elevation ( $\Delta T_b$ ).

Another concept to understand is the effects of heat on substances. This can be observed on the heating curve below.

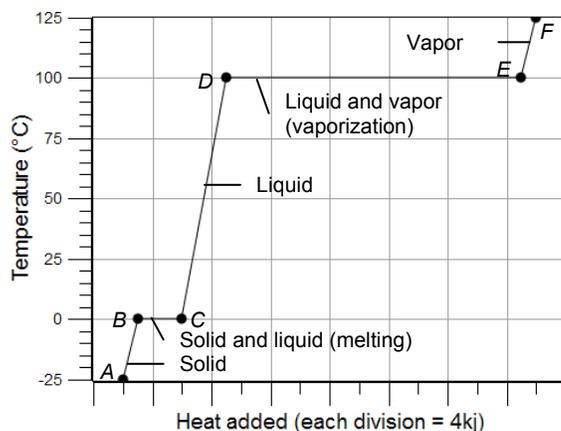


Figure 3: Heating curve

The temperature of solid (AB), liquid (CD), and gas (EF) phases increases as heat is absorbed. However, at the freezing point and boiling point the temperature remains constant while the heat of fusion (BC) and heat of vaporization (DE) are absorbed.

In this activity students will perform the process in the reverse direction: instead of heating, they will cool a liquid solvent and solution until it freezes.

## Pre-Lab Activity

### Setting the stage for the activity

As a solute is added to a solvent, the freezing point of the resulting solution is lowered. The freezing point depression of the solution is dependent on two things: the solvent and the amount of solute added. However, it does not depend on the nature of the solute. The following equation describes this relationship:

$$\Delta T_f = K_f m$$

where

$\Delta T_f$  = the freezing point depression (K)

$K_f$  = the freezing point depression constant for a particular solvent ( $K \text{ kg mol}^{-1}$ )

$m$  = the molality of the solution (mol/kg)

Stated another way:

$$\Delta T_f = K_f \frac{n_s}{m_{\text{solvent}}}$$

where

$n_s$  = number of moles of solute (mol)

$m_{\text{solvent}}$  = mass of solvent (kg)

Moles of solute can be determined from the following equation:

$$n_s = \frac{m_s}{FW_s}$$

where

$m_s$  = mass of solute (g)

$FW_s$  = formula weight of solute (g/mol)

Replacing  $n_s$  with this expression yields

$$\Delta T_f = K_f \frac{\left( \frac{m_s}{FW_s} \right)}{m_{\text{solvent}}} = K_f \frac{m_s}{m_{\text{solvent}} FW_s}$$

Rearranging, the molecular weight of the solute is

$$FW_s = K_f \frac{m_s}{\Delta T_f m_{\text{solvent}}}$$

In this experiment, you will measure the freezing point of a pure solvent, lauric acid (dodecanoic acid). You will measure the freezing point of a mixture of lauric acid and an unknown solute. From the freezing point change, the formula weight of the solute can be calculated.

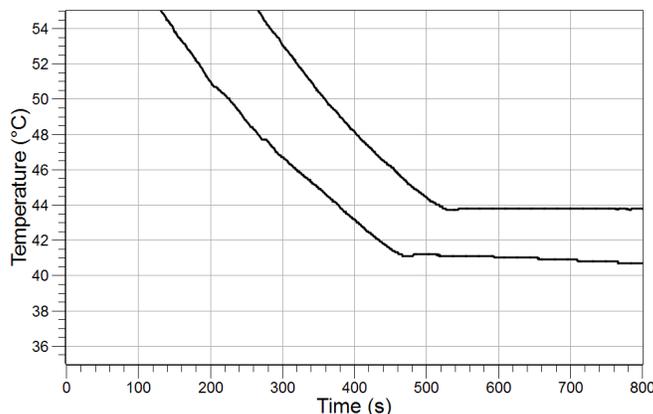
To obtain the cooling curve, the melted solvent or the melted solution must be cooled very slowly. During this time, the temperature of the system is monitored and the contents of the test tube are continuously mixed.

### **Example calculation to try**

In an experiment, the formula weight of an unknown organic substance is determined. First, the freezing point is determined for 8.50 g (0.00850 kg) of solvent placed in a test tube. Then the contents of the test tube are melted and 0.9407 g of the unknown substance is dissolved in the liquid. As the graph shows, the freezing point of the solution is also measured.

## Lab 4: Molecular Weight by Freezing Point Depression

$K_f$  for the solvent is  $3.90 \text{ K kg mol}^{-1}$ .



The freezing point of the pure solvent is  $43.8 \text{ }^\circ\text{C}$ . The freezing point of the solution is  $41.2 \text{ }^\circ\text{C}$ . Therefore the freezing point depression is  $2.60 \text{ }^\circ\text{C}$ , or  $2.60 \text{ K}$ .

Substituting these values into the relevant equation to find the formula weight:

$$FW_s = \left( 3.90 \frac{\text{K kg}}{\text{mol}} \right) \frac{(0.9407 \text{ g})}{(2.60 \text{ K})(0.00850 \text{ kg})} = 166 \frac{\text{g}}{\text{mol}}$$

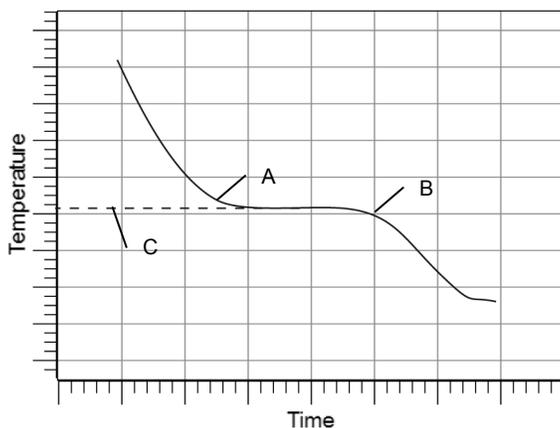
### 1. Why is the vapor pressure lower over solutions than over the pure solvent?

The solute species bind solvent molecules. Therefore there are fewer "free" solvent molecules available to escape into the gas phase.

### 2. How does a lower vapor pressure result in standard boiling point elevation?

Standard boiling point is defined as the temperature where the vapor pressure reaches the atmospheric pressure (1 atm). Since the vapor pressure over a solution at any temperature is less than the vapor pressure over a pure solvent, solutions must be heated to a higher temperature to reach that atmospheric pressure. So the boiling point of solutions is higher than the boiling point of solvents.

### 3. Knowing how the heating curve looks, sketch a graph of the cooling curve starting from the liquid phase and ending with the solid phase. Label the axes, the points where freezing starts, freezing ends, and label the freezing point.



A: freezing starts  
B: freezing ends  
C: freezing point

**4. Why does the temperature remain constant at the freezing point?**

The temperature remains constant because the heat is used for the heat of fusion rather than for heating the system.

**5. Using the fact that the temperature remains constant at the freezing point for a an interval of time, how would you measure the freezing point change of a solution relative to the freezing point of a pure solvent?**

Because the temperature remains constant for a while at the freezing point, that temperature can be measured very accurately. We can measure the freezing point of the solvent and then the freezing point of the solution made from a known amount of solvent and solute. The difference in the freezing points can be calculated easily.

---

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

1. For stirring purposes, loosely wrap copper wire around the stainless steel sensor so it can easily slide up and down on the sensor. Make one coil for each group.
2. Place an appropriate amount of the benzoic acid ( $C_6H_5COOH$ ) in a container labeled “Unknown Solute” for students to use.
3. For cleaning, use an organic solvent, such as toluene or xylene. The coil is disposable.

---

## Safety

Add these important safety precautions to your normal laboratory procedures:

- ◆ Handle the hot water bath carefully while mounting and removing the test tube.
- ◆ Upon completion, wash all equipment properly with the assigned solvent for cleaning.

### Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

4	3	1	5
Melt the contents of the test tube and dissolve the solution with the unknown solute.	Measure about 0.50 g of the unknown sample to the nearest milligram and add it to the solvent in the test tube.	Measure about 8.0 g of solvent and place it into a test tube. Melt the contents of the test tube.	Record the cooling curve of the solution.
	2	6	
	Start data collection and record the cooling curve while stirring the solvent in the test tube.	From the freezing point depression, calculate the molecular weight.	

### Procedure with Inquiry

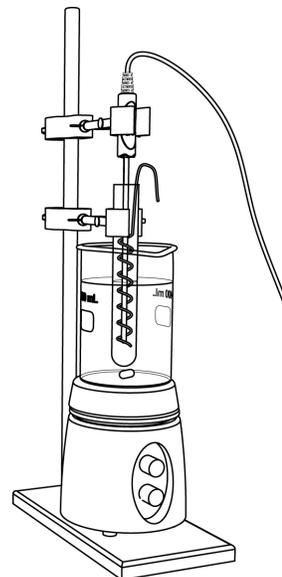
After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "♦" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

#### Set Up

- Start a new experiment on the data collection system ♦<sup>(1.2)</sup>
- Connect the stainless steel temperature sensor to the data collection system. ♦<sup>(2.1)</sup>
- Display data on the graph to show Temperature versus Time. ♦<sup>(7.1.1)</sup>
- Measure about 8 g of lauric acid and record the mass of this solvent to the nearest 0.01 g in Table 1.

5.  Place a 400-mL beaker 3/4 full of water on a hotplate.
6.  Place a stirring bar into the beaker.
7.  Use a clamp to connect the test tube to the ring stand so it is immersed in the water as far as possible. If necessary, add more water to the beaker.
8.  Wrap the copper wire coil around the stainless steel sensor. Clamp it to the ring stand and insert it into the test tube as shown in the illustration.



### Collect Data

9.  Carefully transfer the solvent into the test tube.

**Important:** Make sure there is no loss of the sample when doing a transfer

10.  Turn on the hotplate and the stirring bar.
11.  When the crystals melt, place the stainless steel sensor with the copper coil wrapped around it into the melted crystals.

**Note:** Observe the white deposit on the surface of the sensor.

12.  What is the white deposit and why does it appear on the sensor?

Because the sensor is cold, the lauric acid freezes temporarily on the surface of the sensor. It melts when the sensor warms up to the temperature of the liquid.

13.  Once the test tube is clear (the crystals are melted), remove the test tube from the water bath, with the sensor and coil still in it, and clamp it in the empty Erlenmeyer flask.

**Important:** Do not stop stirring while collecting data. Even a few seconds without stirring can affect your graph.

14.  Start data recording  $\diamond^{(6.2)}$  and start stirring the contents of the test tube by moving the copper coil up and down.
15.  Adjust the scale of the graph.  $\diamond^{(7.1.2)}$
16.  Once the contents of the test tube are completely solidified, stop data recording.  $\diamond^{(6.2)}$

## Lab 4: Molecular Weight by Freezing Point Depression

17.  Mount the test tube into the water bath again and allow the contents of the test tube to melt.
18.  Measure about 0.5 g of the unknown sample to the nearest 0.01 g and record the mass in Table 1.
19.  Carefully transfer the solute into the test tube.

**Important:** Make sure there is no loss of the sample when making the transfer.

20.  What happens if you lose some of the sample during the transfer?

The measured freezing point depression is going to be less, but it will be attributed to the mass that was measured, resulting in a higher formula weight than should be obtained.

21.  Once the test tube is clear (the crystals are melted), remove the test tube from the water bath, with the sensor and coil still in it, and clamp it in the empty Erlenmeyer flask.

**Important:** Do not stop stirring while collecting data. Even a few seconds without stirring can affect your graph.

22.  Start the data recording  $\diamond^{(6.2)}$  and start stirring the contents of the test tube by moving the copper coil up and down.

23.  Once the contents of the test tube are completely solidified, stop data recording.  $\diamond^{(6.2)}$

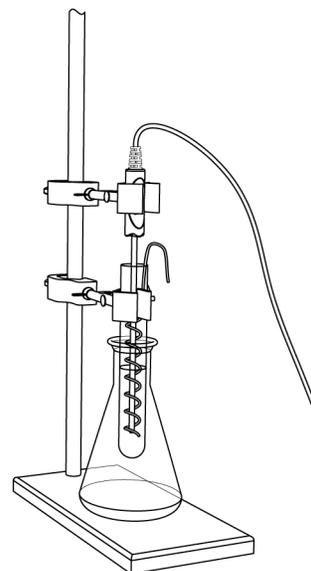
24.  Display both data runs in the graph.  $\diamond^{(7.1.3)}$

25.  From the curves, locate the freezing points of the pure solvent and the solution. Record these in Table 1.

26.  Print the graph.  $\diamond^{(11.2)}$

27.  Melt the contents of the test tube again and dispose of the chemicals into the designated waste container.

28.  Save your experiment  $\diamond^{(11.1)}$  and clean up according to your teacher's instructions.



## Data Analysis

Table 1: Freezing point depression measurements and calculations

Parameter	Value
Mass of the lauric acid (g)	7.96
Mass of the unknown sample (g)	0.500
Freezing point of lauric acid (°C)	43.5
Freezing point of lauric acid mixed with the unknown solute (°C)	41.3
Change in freezing point (°C)	2.20
Molar mass (experimental) of unknown solute (g/mol)	111.35
Molality of the solution (mol/kg)	0.566
Moles of the unknown sample (mol)	$4.49 \times 10^{-3}$
Possible solute	Benzoic acid
Molar mass (from Table 2) of possible solute (g/mol)	122.4
Percentage of error (%)	9.31

1.  Calculate the formula weight of the solute.

**Note:**  $K_f$  for lauric acid is  $3.90 \text{ K kg mol}^{-1}$ .

$$FW_s = K_f \frac{m_s}{\Delta T_f m_{\text{solvent}}}$$

$$FW_s = \left( 3.90 \frac{\text{K kg}}{\text{mol}} \right) \frac{(0.500 \text{ g})}{(2.20 \text{ K})(0.00796 \text{ kg})} = 1.11 \times 10^2 \frac{\text{g}}{\text{mol}}$$

2.  Calculate the number of moles of the solute used, based on the calculated formula weight and determine the molality of the solution.

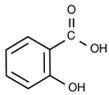
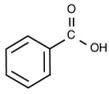
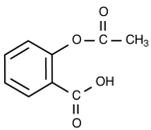
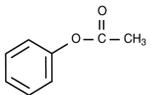
$$\frac{0.50 \text{ g solute}}{1.11 \times 10^2 \text{ g/mol solute}} = 0.00450 \text{ mol solute}$$

$$\frac{0.00450 \text{ moles solute}}{0.00796 \text{ kg lauric acid}} = 0.566 \text{ mol/kg}$$

### Lab 4: Molecular Weight by Freezing Point Depression

3.  Based on the formula weights of the compounds in Table 2, what solute have you been using? Record it and its formula weight in Table 1.

Table 2: Formula weights of different compounds

Name	Formula	Formula weight (g/mol)
Salicylic acid		138.1
Benzoic acid		122.4
Acetylsalicylic acid (aspirin)		180.2
Acetyl phenol		136.1

4.  Determine the percent error between the experimental molar mass and the molar mass from Table 2.

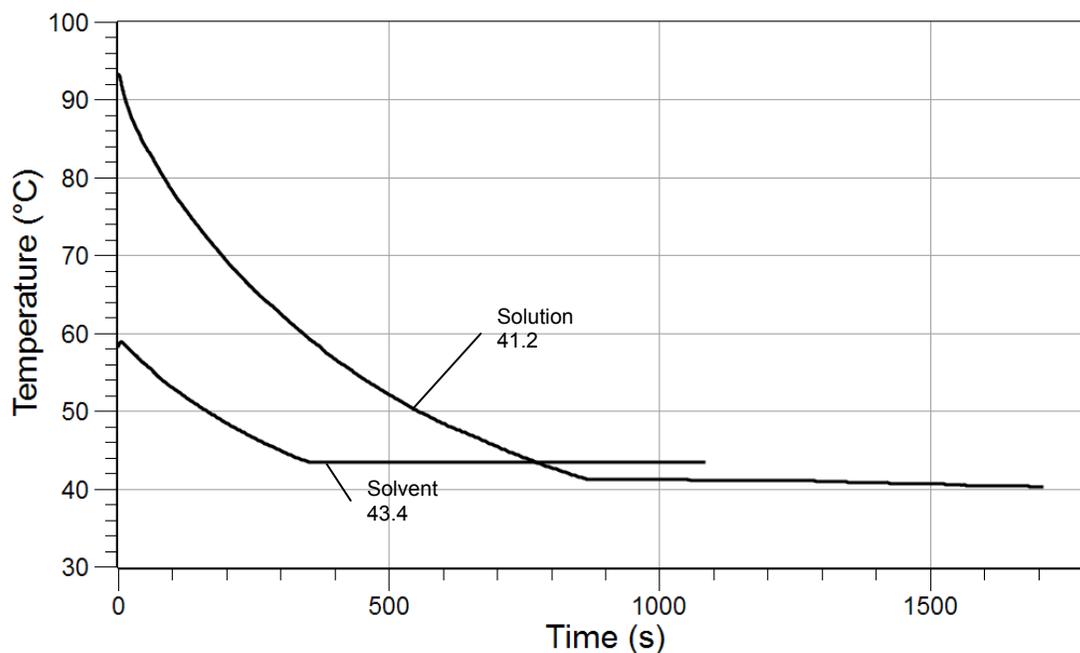
$$\text{Percent Error} = \left| \frac{\text{Actual Value} - \text{Experimental Value}}{\text{Actual Value}} \right| \times 100$$

$$\text{Percent Error} = \frac{122.4 - 1.11 \times 10^2}{122.4} \times 100 = 9.31\%$$

5.  What are the sources of error?

The loss of material during the mixing is one source of error. Also, reading the freezing point from the graph might be difficult because sometimes the portion of the cooling curve that is supposed to be horizontal is not, making it difficult to determine the freezing point exactly.

6.  Sketch or paste the graph with the cooling curves below. Label the curve corresponding to the solvent and the curve corresponding to the solution.



### Analysis Questions

1. Why is lauric acid the solvent in this experiment, even though lauric acid is solid at room temperature and solvents are usually liquids at room temperature?

Lauric acid was present in greater quantity than the unknown sample. It is considered by definition as the solvent.

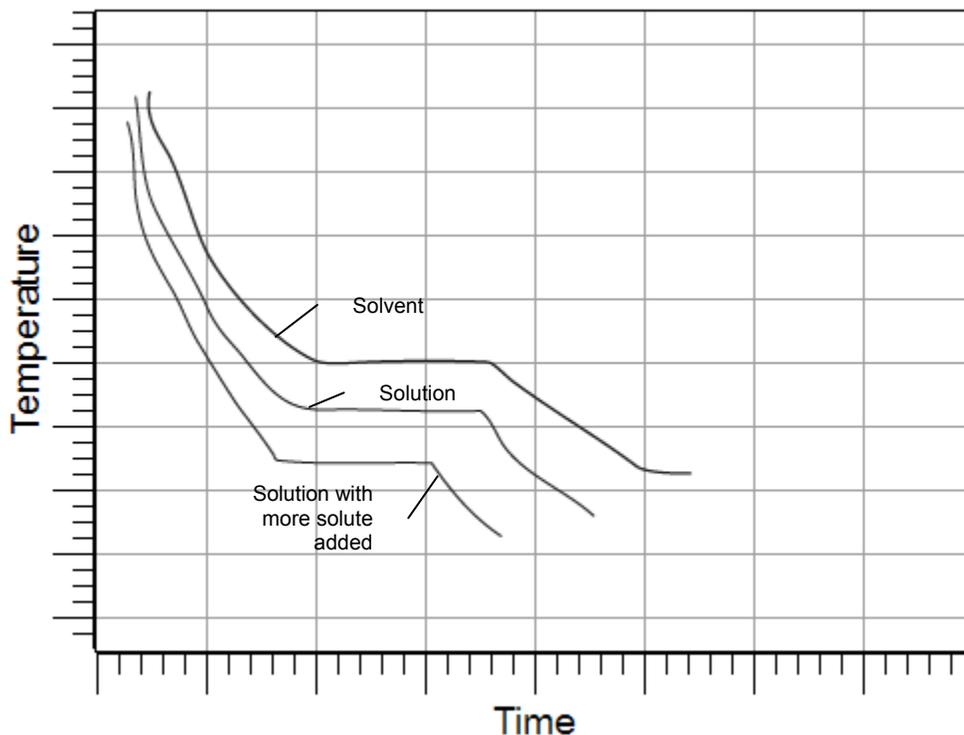
2. How would the error show itself if the test tube was dirty?

The contamination would lower the freezing point because of the increased number of non-solvent particles. This would result in a lower calculated formula weight.

## Synthesis Questions

Use available resources to help you answer the following questions.

1. Suppose you collected data for an additional cooling curve, adding additional unknown sample to the solution; sketch a graph with the three cooling curves and label each curve.



2. Why would you collect data for an additional cooling curve with additional unknown sample?

It would give an additional measured value for the formula weight. Then you could average the values to reduce experimental error.

## Multiple Choice Questions

Select the best answer or completion to each of the questions or incomplete statements below.

1. Which of the following statements is *not* correct regarding colligative properties?
  - A. Vapor pressure over solutions is lower than the vapor pressure of pure solvent.
  - B. The freezing point of solutions is lower than that of a pure solvent.
  - C. The boiling point is elevated by solutes.
  - D. All the statements are correct.

**2. Consider two unknowns, A and B. We measure the same mass for each to determine the formula weight for each. We measure the same mass of the solvent to mix with the two unknowns. The freezing point depression for unknown A is significantly greater than for unknown B. What does this say about the formula weights of unknowns A and B?**

- A. Assuming the same mass of unknown A and B ( $m_s$ ) and the same mass of solvent ( $m_{\text{solvent}}$ ) are used, a greater freezing point depression with A would mean that A has a smaller formula weight than B.**
- B. Assuming the same mass of unknown A and B ( $m_s$ ) and the same mass of solvent ( $m_{\text{solvent}}$ ) are used, a greater freezing point depression with A would mean that A has a greater formula weight than B.**
- C. Assuming the same mass of unknown A and B ( $m_s$ ) and the same mass of solvent ( $m_{\text{solvent}}$ ) are used, a greater freezing point depression with A would mean that B has a smaller formula weight than A.**
- D. Not enough information**

### Extended Inquiry Suggestions

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The freezing points of electrolytes are larger than expected because they dissociate, producing more species in solution.

Use this information to discuss colligative properties with students, that they depend on the number of species only and not on the nature of the solute, more species yields larger effect. Quantitatively this effect is taken into account with the coefficient  $i$ , the so called van't Hoff coefficient:

$$\Delta T_f = iK_f m$$

For substances that do not dissociate,  $i = 1$ , resulting in the original equation for the freezing point depression. For electrolytes, the limiting value of  $i$  is the number of species resulting from the dissociation. For example,  $\text{MgCl}_2$  yields three ions. Therefore, the limiting value of  $i$  is 3.

This "limiting" nature comes from the fact that at higher concentration there is no complete dissociation. Therefore the ion concentration is somewhat less than the predicted value.

Discuss, as well, how electrolytes have a much larger effect than non-electrolytes.

In a separate discussion, ask why the Department of Public Works uses  $\text{CaCl}_2$  and  $\text{MgCl}_2$  to de-ice roads instead of  $\text{NaCl}$ .



## Lab 5: Molar Volume of a Gas

### Objectives

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Students determine the molar volume of a gas by relating pressure, volume, and temperature.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Conducting an experiment in a closed container that generates hydrogen gas while maintaining constant temperature and monitoring pressure
- ◆ Calculating the molar volume of the generated gas and converting that to standard temperature and pressure conditions

### Time Requirement

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◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	45 minutes

### Materials and Equipment

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#### *For each student or group:*

- ◆ Data collection system
- ◆ Absolute pressure sensor with quick-release connectors and plastic tubing
- ◆ Stainless steel temperature sensor
- ◆ Sensor extension cable
- ◆ Beaker, 600-mL
- ◆ Erlenmeyer flask, 250-mL
- ◆ Graduated cylinder, 10-mL or 25-mL
- ◆ Graduated cylinder, 100-mL
- ◆ Balance (1 per class)
- ◆ Rubber stopper with one hole
- ◆ 3 M Hydrochloric acid (HCl), 20 mL<sup>1</sup>
- ◆ Magnesium ribbon, about 0.20 g
- ◆ Water, 300 mL
- ◆ Electrical tape, roll (optional)

<sup>1</sup> To prepare 3 M HCl using concentrated HCl, refer to the Lab Preparation section.

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Definition of an ideal gas
- ◆ Ideal Gas Law
- ◆ Relationship between temperature and pressure (Charles' Law), temperature and volume (Gay-Lussac's Law), and pressure and volume (Boyle's Law)
- ◆ Standard temperature and pressure (STP) conditions
- ◆ Avogadro's Law
- ◆ Dalton's Law
- ◆ Chemical formulas
- ◆ Balancing chemical equations
- ◆ Stoichiometric calculations

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 3: Determine the Molar Mass of a Volatile Liquid
- ◆ Lab 27: Identifying an Unknown Metal
- ◆ Lab 29: Exploring Gas Laws

## Using Your Data Collection System

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

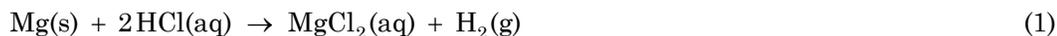
- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting sensors to the data collection system ◆<sup>(2.2)</sup>
- ◆ Changing the units of a measurement ◆<sup>(5.3)</sup>
- ◆ Monitoring live data without recording ◆<sup>(6.1)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>

- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>
- ◆ Displaying multiple graphs simultaneously ◆<sup>(7.1.11)</sup>
- ◆ Selecting data points in a graph ◆<sup>(7.2.1)</sup>

## Background

The volume occupied by one mole of substance at standard temperature and pressure (STP) is known as the “standard molar volume.” According to Avogadro's hypothesis, equal volumes of gases under equal conditions of temperature and pressure contain equal numbers of molecules. It follows from this hypothesis that all gas samples containing one mole of molecules will occupy the same volume at STP.

The basis of this experiment is the following reaction, in which a known mass of magnesium (Mg) is reacted with an excess of hydrochloric acid (HCl) to yield the products shown:



Inspection of this equation reveals that one mole of magnesium (24.3 g) yields one mole of hydrogen gas (2.02 g). Hydrogen gas is the product of interest in this experiment. By determining the number of moles of magnesium that react, you will indirectly determine the number of moles of hydrogen gas produced. The Ideal Gas Law is used to find the volume that the gas would occupy at STP. The number of moles and volume at STP is used to calculate the molar volume of hydrogen gas.

## Pre-Lab Activity

### Setting the stage for the activity

In order to determine the number of moles of hydrogen gas produced by the reaction of Equation 1, first calculate the number of moles of magnesium used:

$$n_{\text{Mg}} = \frac{m_{\text{Mg}}}{AW_{\text{Mg}}} \quad (2)$$

where

$n_{\text{Mg}}$  = number of moles of magnesium used in the experiment (mol)

$m_{\text{Mg}}$  = mass of magnesium measured (g)

$AW_{\text{Mg}}$  = atomic weight of magnesium (g/mol).

Avogadro's Law states that the number of moles of an ideal gas is proportional to its volume. Therefore the volume of hydrogen gas that can be made from 1 mol of magnesium can be calculated:

$$\frac{n_{\text{H}_2}}{n_0} = \frac{V_{\text{E}}}{V_1}$$

## Lab 5: Molar Volume of a Gas

---

where

$n_{\text{H}_2}$  = number of moles of  $\text{H}_2$  produced in the reaction (mol)

$n_0 = 1$  mol

$V_E$  = the volume of the Erlenmeyer flask ( $\text{m}^3$ )

$V_1$  = the volume of  $\text{H}_2$  gas produced from the reaction with 1 mol of magnesium ( $\text{m}^3$ )

Considering that 1 mol of Mg produces 1 mol of  $\text{H}_2$

$$n_{\text{Mg}} = n_{\text{H}_2}$$

$$\frac{n_{\text{Mg}}}{n_0} = \frac{V_E}{V_1}$$

where

$n_{\text{Mg}}$  = the number of moles of Mg that was used in the reaction (mol)

Solving for  $V_1$ :

$$V_1 = \frac{n_0}{n_{\text{Mg}}} V_E \quad (3)$$

The volume can be readily converted to STP:

$$\frac{p_1 V_1}{T_1} = \frac{p_0 V_0}{T_0}$$

Solving for  $V_0$ :

$$V_0 = \frac{p_1 T_0 V_1}{p_0 T_1} \quad (4)$$

where

the zero indices refer to the STP conditions ( $T_0 = 273$  K,  $p_0 = 1.013 \times 10^5$  Pa)

$p_1$  = the pressure inside the Erlenmeyer flask due to the gas produced (Pa )

$V_1$  = the volume of  $\text{H}_2$  gas produced from the reaction with 1 mol of magnesium ( $\text{m}^3$ )

$T_1$  = final temperature of the water bath (K)

Magnesium will instantaneously react with hydrochloric acid to form  $\text{H}_2$  gas. The gas is captured in a closed Erlenmeyer flask. The volume of the flask will be measured at the end of the experiment. The final pressure and final temperature will be measured by the data collection system.

**Example calculation to try**

In an experiment, 20 mL of 3 M HCl were placed into a 125-mL Erlenmeyer flask. The atmospheric pressure was recorded as 101.5 kPa. Then 0.180 g of magnesium was measured and dropped into the hydrochloric acid. The flask was immediately sealed with a one-hole rubber stopper attached to the pressure sensor via a piece of Tygon® tubing.

The flask was placed into a water bath. The temperature of the bath and the pressure inside the flask were monitored. When the reaction was completed, the pressure was 253.0 kPa and the temperature of the bath was 25 °C. The volume of the Erlenmeyer flask was measured by filling it with water, inserting the stopper, and measuring the volume of the water, which was 141.00 mL.

The volume of the flask that the gas could occupy was 121 mL (141 mL minus the 20 mL occupied by the hydrochloric acid). The pressure of the hydrogen produced was 151.5 kPa (253.0 kPa – 101.5 kPa).

Converting the volume to cubic meters and the pressure to Pascals results in the following:

$$V_E = 121 \text{ mL} \left( \frac{1 \text{ m}^3}{10^6 \text{ mL}} \right) = 1.21 \times 10^{-4} \text{ m}^3$$

$$p_1 = 151.5 \text{ kPa} \left( \frac{10^3 \text{ Pa}}{1 \text{ kPa}} \right) = 1.515 \times 10^5 \text{ Pa}$$

Using Equation 2, the number of moles of magnesium used in the experiment are

$$n_{\text{Mg}} = \frac{0.180 \text{ g}}{24.3 \frac{\text{g}}{\text{mol}}} = 7.41 \times 10^{-3} \text{ mol}$$

From Equation 3, the volume of hydrogen formed from 1 mol of magnesium is

$$V_1 = \frac{(1 \text{ mol})}{(7.41 \times 10^{-3} \text{ mol})} (1.21 \times 10^{-4} \text{ m}^3)$$

$$V_1 = 1.63 \times 10^{-2} \text{ m}^3$$

Using Equation 4 to convert the molar volume to STP conditions yields

$$V_0 = \frac{(1.515 \times 10^5 \text{ Pa})(293 \text{ K})(1.63 \times 10^{-2} \text{ m}^3)}{(1.013 \times 10^5 \text{ Pa})(298 \text{ K})}$$

$$V_0 = 2.40 \times 10^{-2} \text{ m}^3$$

The molar volume was found to be  $2.40 \times 10^{-2} \text{ m}^3$ .

**1. Why do you subtract the atmospheric pressure from the final pressure to obtain the pressure of the hydrogen gas generated?**

It's necessary to subtract atmospheric pressure from final pressure because the final pressure includes the atmospheric pressure and only the partial pressure of hydrogen is applicable for the calculations.

## Lab 5: Molar Volume of a Gas

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### 2. Would it matter if the volume of the Erlenmeyer flask used in the experiment was 250 mL?

The flask size would not matter because the number of moles of hydrogen depends only on the number of moles of magnesium.

## Lab Preparation

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These are the materials and equipment to set up prior to the lab:

1. **3M HCl:** Prepare 20 mL of 3 M hydrochloric acid solution for each student group by diluting a concentrated hydrochloric acid solution 1:4 with distilled water. The exact concentration is not critical in this experiment.

**CAUTION:** Remember to add the concentrated acid to the water to prevent spattering.

## Safety

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Add these important safety precautions to your normal laboratory procedures:

- ◆ If you get hydrochloric acid on your skin, wash it off with plenty of water.
- ◆ Handle the Erlenmeyer flask very carefully when it is pressurized. Knocking on the glass can cause the glass to crack and because of the pressure, a slight explosion can occur.

## Sequencing Challenge

---

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

2	3	5	4	1
Measure about 0.20 g of magnesium to the nearest mg. Transfer the magnesium into the Erlenmeyer flask that has the HCl solution in it.	Immediately plug the flask with a stopper having a connector and tubing to the pressure sensor. Keep it in a water bath to stabilize the temperature.	Obtain three sets of data. Calculate the molar volume of hydrogen.	Collect the hydrogen in the flask and measure the final pressure and temperature. Measure the volume of the flask.	Set up the data collection system with a temperature and pressure sensor. Then put 20.00 mL of an HCl solution into a flask.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box (☐) next to that step.

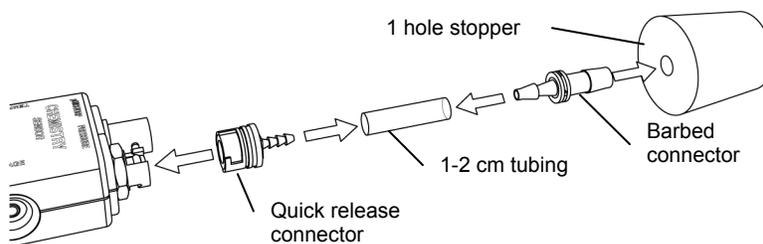
**Note:** When students see the symbol "◆" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

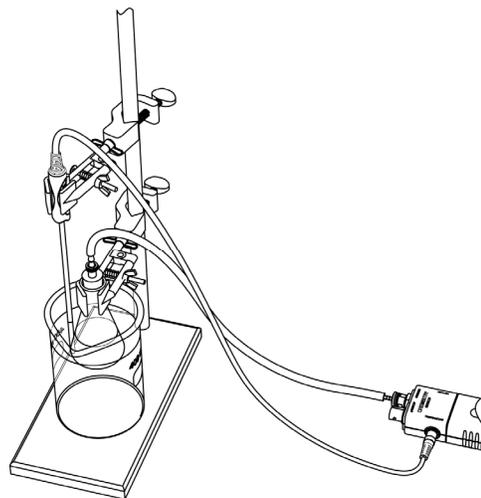
- ☐ Start a new experiment on the data collection system. ◆<sup>(1.2)</sup>
- ☐ Connect the absolute pressure sensor, using the sensor extension cable, and the temperature sensor to the data collection system. ◆<sup>(2.2)</sup>
- ☐ Set up one graph to monitor the pressure and another graph to monitor the temperature as a function of time. ◆<sup>(7.1.11)</sup>

**Note:** Change the units of the temperature measurement to Kelvin. ◆<sup>(5.3)</sup>

- ☐ Place the barbed connector of the pressure sensor tightly into the rubber stopper and connect it to the pressure port of the sensor with a piece of tubing.



- ☐ If electrical tape is available, wrap the Erlenmeyer flask with 10 to 15 rounds of electric tape. This is a preventive measure in case the flask cracks. The tape keeps the glass pieces together.
- ☐ Fill the 600-mL beaker 3/4 full with tap water.
- ☐ Mount the 250-mL Erlenmeyer flask in the water bath—place it inside the 600-mL beaker with water. The water should cover as much of the flask as possible. Add more water if needed.



8.  Place the temperature sensor in the water bath.
9.  Why do you need to immerse the flask as much as possible into the water bath?

The flask is immersed as much as possible so students know the temperature of the gas in the flask. If the flask is not completely immersed, the temperature of the gas will not be the same as the temperature of the water bath. Then students will not know what the temperature of the gas is.

### Collect Data

10.  Magnesium and hydrochloric acid reaction
- Measure a small piece of magnesium ribbon (between 0.150 g and 0.180 g) to the nearest milligram and record the mass in Table 1.
  - Measure 20 mL of 3 M HCl solution with a graduated cylinder and transfer it into the Erlenmeyer flask.
  - Start data recording.  $\diamond^{(6.2)}$
  - What does the pressure reading on the sensor represent at this point?

The pressure sensor shows the atmospheric pressure of air.

- Drop the pre-measured piece of magnesium into the flask and immediately insert the stopper airtight.

**Important:** Make sure that the stopper is sitting firmly in the flask because pressure is building in the flask and a loose stopper might pop out. If that happens, you will need to repeat the experiment.

- Continue to monitor the pressure.
- When the hissing in the flask ends and the pressure stabilizes, stop the data collection.  $\diamond^{(6.2)}$
- Record the initial and final pressure during the reaction and record those values in Table 1.  $\diamond^{(7.1.4)}$
- Record the initial and final temperature of the water bath and record those values in Table 1.  $\diamond^{(7.1.4)}$
- Stop data recording.  $\diamond^{(6.2)}$

**Note:** Aside from recording this data in the table, you do not need to save your data.

- Remove the stopper carefully and dispose of the spent acid solution properly.

11.  Repeat the magnesium and hydrochloric acid reaction two times.
12.  What does the final pressure reading represent? (Hint: What components contribute to the final pressure?)

Both the air (initial pressure) and the hydrogen gas generated by the reaction are responsible for the pressure. In fact, the pressure increase is due to the hydrogen gas produced.

- 13.**  Fill the Erlenmeyer flask to the top with water, insert the stopper, and then use the 100-mL graduated cylinder to measure the volume of the water, which is the volume of any gases in the flask. Record this value in Table 1.
- 14.**  Clean up according to your teacher's instructions.

## Data Analysis

- 1.**  Calculate the number of moles of magnesium used for each trial and record the values in Table 1.

For Trial 1:

$$n_{\text{Mg}} = \frac{0.106 \text{ g}}{24.3 \frac{\text{g}}{\text{mol}}} = 4.36 \times 10^{-3} \text{ mol}$$

- 2.**  Convert the pressure for each trial from kPa to Pa for the initial and final pressure values. Calculate the pressure due to the hydrogen gas generated and record the values in Table 1.

For Trial 1:

$$\text{Initial Pressure: } 102 \text{ kPa} \left( \frac{10^3 \text{ Pa}}{1 \text{ kPa}} \right) = 1.02 \times 10^5 \text{ Pa}$$

$$\text{Final Pressure: } 146 \text{ kPa} \left( \frac{10^3 \text{ Pa}}{1 \text{ kPa}} \right) = 1.46 \times 10^5 \text{ Pa}$$

Pressure change due to the hydrogen gas produced:  $(1.46 \times 10^5 \text{ Pa}) - (1.02 \times 10^5 \text{ Pa}) = 4.40 \times 10^4 \text{ Pa}$

- 3.**  Determine the volume the hydrogen gas can occupy in the flask and convert it from milliliters to cubic meters. Record the values in Table 1.

For Trial 1:

250 mL available volume – 20 mL HCl solution = 230 mL available for H<sub>2</sub>

$$250 \text{ mL} \left( \frac{1 \text{ m}^3}{10^6 \text{ mL}} \right) = 2.5 \times 10^{-4} \text{ m}^3$$

$$230 \text{ mL} \left( \frac{1 \text{ m}^3}{10^6 \text{ mL}} \right) = 2.3 \times 10^{-4} \text{ m}^3$$

- 4.**  Determine the volume of hydrogen that would be generated by 1 mol of magnesium. Record the value in Table 1.

For Trial 1:

$$V_1 = \frac{n_0}{n_{\text{Mg}}} V_E$$

$$V_1 = \left( \frac{1 \text{ mol}}{4.36 \times 10^{-3} \text{ mol Mg}} \right) (2.30 \times 10^{-4} \text{ m}^3) = 5.27 \times 10^{-2} \text{ m}^3$$

## Lab 5: Molar Volume of a Gas

5.  At standard temperature and pressure, what is the volume of 1 mol of hydrogen gas? Calculate the average volume of 1 mol of hydrogen gas at STP for all three trials. Record the values in Table 1.

For Trial 1:

$$V_0 = \frac{p_1 T_0 V_1}{p_0 T_1}$$

$$V_0 = \frac{(4.40 \times 10^4 \text{ Pa})(293 \text{ K})(5.27 \times 10^{-2} \text{ m}^3)}{(1.013 \times 10^5 \text{ Pa})(302 \text{ K})} = 2.27 \times 10^{-2} \text{ m}^3$$

The average volume is  $[(2.27 \times 10^{-2}) + (2.43 \times 10^{-2}) + (2.27 \times 10^{-2})]/3 = 2.33 \times 10^{-2} \text{ m}^3$

Table 1: Molar volume of H<sub>2</sub> calculated from the measured values

Parameters	Trial 1	Trial 2	Trial 3
Mass of magnesium ribbon (g)	0.106	0.104	0.106
Moles of magnesium (mol)	$4.36 \times 10^{-3}$	$4.28 \times 10^{-3}$	$4.36 \times 10^{-3}$
Initial pressure (kPa)	102	102	102
Initial pressure (Pa)	$1.02 \times 10^5$	$1.02 \times 10^5$	$1.02 \times 10^5$
Final pressure (kPa)	146	147	149
Final pressure (Pa)	$1.46 \times 10^5$	$1.47 \times 10^5$	$1.49 \times 10^5$
Pressure due to hydrogen gas (Pa)	$4.40 \times 10^4$	$4.50 \times 10^4$	$4.70 \times 10^4$
Initial temperature of water bath (K)	298.0	298.0	298.0
Final temperature of water bath (K)	302.0	302.0	302.9
Volume of flask (mL)	250		
Volume H <sub>2</sub> can occupy (mL)	230		
Volume of flask (m <sup>3</sup> )	$2.50 \times 10^{-4}$		
Volume H <sub>2</sub> can occupy (m <sup>3</sup> )	$2.30 \times 10^{-4}$		
Volume of H <sub>2</sub> generated by 1 mol of Mg (m <sup>3</sup> )	$5.27 \times 10^{-2}$	$5.37 \times 10^{-2}$	$5.27 \times 10^{-2}$
Volume of H <sub>2</sub> generated by 1 mol of Mg (m <sup>3</sup> ) under STP	$2.27 \times 10^{-2}$	$2.43 \times 10^{-2}$	$2.27 \times 10^{-2}$
Average molar volume at STP (m <sup>3</sup> ):	$2.33 \times 10^{-2}$		

## Analysis Questions

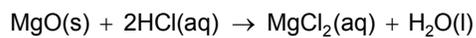
- 1.** The accepted value for the molar volume of an ideal gas at STP is 22.4 L/mol. Calculate the percent error of your average results.

$$\left| \frac{2.33 \times 10^{-2} \text{ m}^3 - 2.24 \times 10^{-2} \text{ m}^3}{2.24 \times 10^{-2} \text{ m}^3} \right| \times 100 = 4.02\%$$

- 2.** List possible sources of error in this experiment.

Delay of putting in the stopper or a leak in the system might allow some of the hydrogen to escape, resulting in too low a final pressure. If the flask isn't completely immersed in the water bath, the recorded temperature will not accurately reflect the temperature in the flask.

- 3.** Assume that the magnesium ribbon was covered with a thin layer of MgO which naturally forms on the surface of magnesium. Provide an equation between MgO and HCl.



- 4.** Does MgO generate hydrogen then (when combined with hydrochloric acid)?

No, it does not.

- 5.** Does the presence of MgO introduce an error then?

Yes, since it contributes to the mass of the sample but does not react to form hydrogen.

- 6.** How does the temperature change during the course of the reaction? Provide a possible explanation for the change of temperature

The temperature goes up usually a few degrees due the fact that the reaction is highly exothermic.

## Synthesis Questions

Use the available resources to help you answer the following questions.

- 1.** Assume we have only zinc to perform this experiment. Zinc reacts with hydrochloric acid according to the same stoichiometry as magnesium. How many grams of zinc would you need to form the same amount of hydrogen as 0.185 g magnesium?

$$0.185 \text{ g Mg} \left( \frac{65.54 \text{ g/mol Zn}}{24.3 \text{ g/mol Mg}} \right) = 0.499 \text{ g Zn}$$

## Lab 5: Molar Volume of a Gas

---

**2.** Consider the following reaction, which occurs when  $\text{CaCO}_3$  reacts with  $\text{HCl}$ . Describe how you would utilize this process to measure the molar volume of carbon dioxide.



A known amount of  $\text{CaCO}_3$  can be reacted with  $\text{HCl}$  solution much like in this experiment. The pressure increase is due to the  $\text{CO}_2$  generated. This calculation is identical to the calculation in the experiment that students just performed.

### Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

- What error would a layer of  $\text{MgO}$  on the surface of the magnesium ribbon cause?
  - The sample would have less magnesium that will react, which in turn would form fewer moles and a smaller volume of hydrogen.
  - The sample would have more magnesium that will react, which in turn would form a greater number of moles and a greater volume of hydrogen.
  - The sample would have less magnesium that will react, which in turn would form greater number of moles and a greater volume of hydrogen.
  - The presence of  $\text{MgO}$  would not cause any error because it forms no hydrogen gas.
- Would a water bath with higher temperature than room temperature cause an error?
  - Yes. A higher temperature is not STP.
  - Yes. A higher temperature would cause a higher pressure reading.
  - No. A higher temperature would proportionally increase pressure according to the gas laws, yielding the same volume.
- Does it matter how much hydrochloric acid was added to the flask?
  - Yes. It matters because the reactants have to be present in stoichiometric quantities.
  - No. It does not matter because we are interested in the amount of hydrogen made from the known amount of magnesium.
  - No. If there is enough to react with the magnesium, an excess amount of hydrochloric acid makes no difference.

### Extended Inquiry Suggestions

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Discuss with your students how the partial pressure of water (evaporating from the hydrochloric acid) can affect the results. Students can do research to see how significant the error from water vapor is at the temperature of the experiment. Have them redo the calculations with the water vapor pressure taken into account.

Challenge students to prove that the molar volume of a gas under standard pressure and temperature conditions is the same for all ideal gases.

## Lab 6: Standardizing a Solution of Sodium Hydroxide

### Objectives

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Students determine the concentration of a sodium hydroxide solution by titrating it with a standard solution of known concentration.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Calibrating a pH sensor and a drop counter
- ◆ Preparing a titrant solution
- ◆ Preparing a standard solution
- ◆ Performing a titration and using the results to calculate the concentration of the titrant

### Time Requirement

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- |                                   |            |
|-----------------------------------|------------|
| ◆ Preparation time                | 15 minutes |
| ◆ Pre-lab discussion and activity | 15 minutes |
| ◆ Lab activity                    | 60 minutes |

### Materials and Equipment

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#### *For each student or group:*

- |                                    |   |
|------------------------------------|---|
| ◆ Data collection system           | ◆ Clamp, right-angle                        |
| ◆ pH sensor                        | ◆ Funnel                                    |
| ◆ Drop counter with micro stir bar | ◆ Potassium hydrogen phthalate (KHP), 0.6 g |
| ◆ Magnetic stirrer                 | ◆ Sodium hydroxide (NaOH), 0.40 g           |
| ◆ Ring stand                       | ◆ Buffers, pH 4 and pH 10, 10 mL            |
| ◆ Beaker (2), 100-mL               | ◆ Water, deionized, 250 mL                  |
| ◆ Beaker (2), 10-mL                | ◆ Wash bottle with deionized water          |
| ◆ Volumetric flask, 100-mL         | ◆ Parafilm <sup>®</sup> or aluminum foil    |
| ◆ Buret, 50-mL                     | ◆ Cotton swab or tissue                     |
| ◆ Buret clamp                      |   |

## **Concepts Students Should Already Know**

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Students should be familiar with the following concepts:

- ◆ Acid-base reactions
- ◆ Balancing chemical equations
- ◆ Basic stoichiometric calculations
- ◆ Concentration calculations
- ◆ Formula weight

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 7: Acid–Base Titration
- ◆ Lab 8: Oxidation–Reduction Titration
- ◆ Lab 11: Using Different Indicators for pH Determination
- ◆ Lab 19: Properties of Buffer Solutions
- ◆ Lab 23: Determination of a Solubility Product
- ◆ Lab 24: Determining  $K_a$  by Half-Titration of a Weak Acid

## **Using Your Data Collection System**

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a sensor to the data collection system. ◆<sup>(2.1)</sup>
- ◆ Connecting multiple sensors to the data collection system ◆<sup>(2.2)</sup>
- ◆ Calibrating a drop counter ◆<sup>(3.4)</sup>
- ◆ Calibrating a pH sensor ◆<sup>(3.6)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>

- ◆ Adjusting the scale of the graph ◆<sup>(7.1.2)</sup>
- ◆ Displaying multiple runs in a graph ◆<sup>(7.1.3)</sup>
- ◆ Changing the variable on the x-axis and y-axis of a graph ◆<sup>(7.1.9)</sup>
- ◆ Finding the slope at a point on the data plot ◆<sup>(9.3)</sup>
- ◆ Saving your experiment ◆<sup>(11.1)</sup>
- ◆ Printing the graph. ◆<sup>(11.2)</sup>

## Background

Chemicals react in strict stoichiometric ratios based on the coefficients of the balanced chemical reaction. This fact allows one to predict the amount of one reactant or product when another is known. When reactions occur in solution, the amount of one reactant is determined by the relationship between concentration, volume, and amount of the substance:

$$c = \frac{n}{V}$$

where

$c$  = concentration of the substance (mol/L)

$V$  = volume of the solution (L)

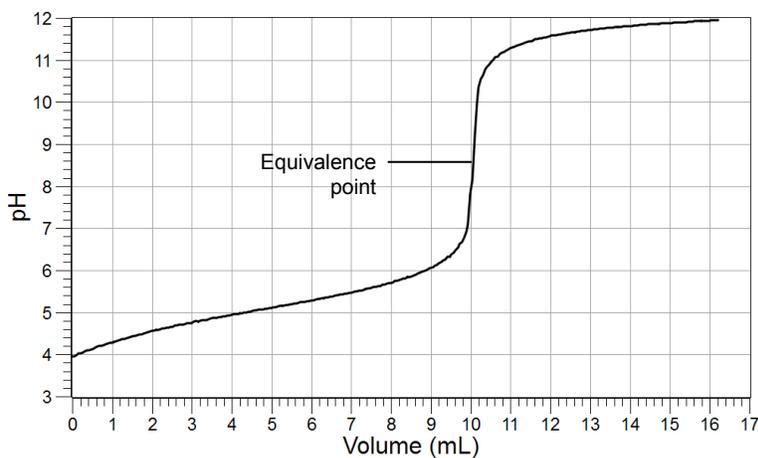
$n$  = amount of substance (mol)

Titration is a common technique used to determine the quantity of one reactant when another is known. In a titration, an indicator gives a visual signal to the experimenter when one reactant has been completely used up. At this point, called the equivalence point, the experimenter will know the volume and molarity, and, therefore, the number of moles of one reactant. By using the stoichiometric ratios, the experimenter can determine the number of moles of the other reactant.

In this lab, a titration is used to standardize a solution of sodium hydroxide. In other words, this experiment uses the laboratory procedure known as “titration” to accurately determine the molarity of a sodium hydroxide solution.

## Lab 6: Standardizing a Solution of Sodium Hydroxide

Determination of the equivalence point can be done using indicators, but there is always a level of subjectivity when looking for specific colors. This can lead to large uncertainties in the equivalence point. To avoid subjectivity, a pH sensor is used to detect the equivalence point. A pH sensor monitors the pH as the titrant solution is added. The steepest point of the titration curve represents the equivalence point.



### Pre-Lab Activity

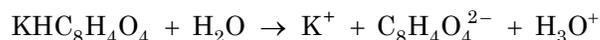
#### Setting the stage for the activity

Sodium hydroxide solution is often used as the base in acid-base titrations. Unfortunately, solid NaOH is hygroscopic, which means that it readily absorbs moisture from the air. Therefore, its mass does not provide precise information about the amount of NaOH present in a sample.

NaOH also absorbs  $\text{CO}_2$  from the air, forming sodium carbonate. This further reduces the amount of NaOH in the pellets. For these reasons, NaOH is not a suitable primary standard and a solution cannot be prepared with precisely known molarity using mass. Additionally, once prepared, a solution of NaOH will continue to react with  $\text{CO}_2$  from the air and can even react slowly with glass if stored in a glass container for too long.

To overcome these obstacles, we will standardize our NaOH solution using an acid as the primary standard. Potassium hydrogen phthalate ( $\text{KHC}_8\text{H}_4\text{O}_4$ ) is often referred to as KHP. KHP is available as a pure, stable, crystalline solid with an accurately measurable mass.

The dissociation process of  $\text{KHC}_8\text{H}_4\text{O}_4$  is:

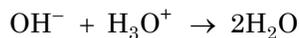


The  $\text{H}_3\text{O}^+$  represents the acid in the solution. The amount of that acid can be measured using NaOH.

NaOH also dissociates in aqueous solutions:



This means that 1 mol of NaOH will yield 1 mol of OH<sup>-</sup> ions. In an acid-base reaction the OH<sup>-</sup> ions react with the H<sub>3</sub>O<sup>+</sup> ions from the KHP solution:



The stoichiometric ratio between the OH<sup>-</sup> and H<sub>3</sub>O<sup>+</sup> ions, and therefore between NaOH and KHP, is 1:1:

$$n_1 = n_2$$

where

$n_x$  = number of moles and the subscript “1” represents NaOH and subscript “2” represents KHP.

Measuring the mass of KHP, we can easily calculate its number of moles:

$$n_2 = \frac{m_2}{FW_{\text{KHP}}} \quad (1)$$

where

$n_2$  = amount of KHP (mol)

$m_2$  = mass of KHP (g)

$FW_{\text{KHP}}$  = formula weight of KHP (g/mol)

The number of moles of KHP will react with the same number of moles of NaOH (using  $n_1 = c_1 V_1$ ) because there is a stoichiometric ratio of 1:1 between KHP and NaOH:

$$n_2 = c_1 V_1$$

$$c_1 = \frac{n_2}{V_1} = \frac{m_2}{FW_{\text{KHP}} V_1} \quad (2)$$

where

$n_2$  = amount of NaOH (mol)

$c_1$  = concentration of KHP (mol/L)

$V_1$  = volume of KHP (L)

In this way, the concentration of the NaOH solution can be calculated.

A question remains. How do we know what volumes of solutions will contain the stoichiometric ratio? The pH sensor answers this question. Immerse the tip of the pH sensor in one of the solutions and gradually add the other solution. When the amount of solution added is the stoichiometrically required amount of reactant, there will be a huge jump in the pH. This indicates that the reaction is over and that the equivalence point has been reached.

In the past, color change from using organic dyes indicated the endpoint of the reaction. However, the color change from an acidic to an alkaline medium was not distinct. It relied on the

## Lab 6: Standardizing a Solution of Sodium Hydroxide

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subjective impression of human eyes. The pH sensor more accurately and objectively detects the equivalence point.

The experiment starts by placing a KHP solution with a known amount of KHP on a stirring plate and adding, drop by drop, a NaOH solution with unknown concentration. While adding the NaOH, monitor the pH of the KHP solution with a pH sensor and record the pH readings as a function of the added amount of NaOH solution.

After each additional drop of NaOH solution is added to the KHP solution, the pH is recorded. The pH signals how much NaOH solution was necessary to react with KHP to reach the equivalence point.

### Example calculation to try

In this example, 0.41 g of solid NaOH was dissolved in a beaker with water. The solution was transferred into a 100-mL volumetric flask and filled to the 100 mL mark. 0.2400 g of KHP was dissolved in water in another beaker. By titrating the KHP solution while monitoring the pH and the volume of NaOH solution added, the equivalence point was reached after 10.50 mL of a NaOH solution was added to the KHP.

Necessary conversion:

$$V_1 = 10.50 \text{ mL} = 1.050 \times 10^{-2} \text{ L}$$

Using Equation 1, the number of moles of KHP is calculated:

$$n_2 = \frac{(0.2400 \text{ g})}{(204.22 \frac{\text{g}}{\text{mol}})}$$

$$n_2 = 1.175 \times 10^{-3} \text{ mol KHP}$$

Recall that the stoichiometry between KHP and NaOH suggests a 1:1 ratio. Using Equation 2, calculate the concentration of the NaOH solution as follows:

$$c_1 = \frac{(1.175 \times 10^{-3} \text{ mol NaOH})}{(1.050 \times 10^{-2} \text{ L})}$$

$$c_1 = 1.119 \times 10^{-1} \frac{\text{mol}}{\text{L}} = 1.119 \times 10^{-1} \text{ M}$$

The concentration of the NaOH solution was  $1.119 \times 10^{-1} \text{ M}$ .

### 1. Does it matter how much water you use to make the KHP solution?

No, it does not. The required volume of NaOH solution would depend only on the number of moles of KHP and that does not depend on how much water was used to make the solution.

### 2. If you accidentally overfill the volumetric flask with the NaOH solution above the mark would that introduce an error?

No, the titration determines the concentration of the solution, regardless of its preparation.

**3. If you accidentally spilled a few crystals of KHP when you make the KHP solution, would that introduce an error?**

Yes, the titration method relies on the fact that you know exactly how many moles of KHP are in the solution. Spilling some of it makes it impossible to know how much KHP was actually used to make the solution.

**Lab Preparation**

These are the materials and equipment to set up prior to the lab:

1. Before the activity, place the KHP in a desiccator, if available, to eliminate any moisture that the KHP might have absorbed.
2. An improperly closed jar can result in significant absorption of moisture and  $\text{CO}_2$  in the NaOH. Make sure that the surface of the NaOH pellets are not covered with moisture.

**Safety**

Add these important safety precautions to your normal laboratory procedures:

- ◆ NaOH is caustic. If NaOH gets on your skin, rinse the exposed surface thoroughly with running water.

**Sequencing Challenge**

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

2	1	3	5	4
Before preparing the titrant, make a solution with a precisely known quantity of KHP, measured to the nearest milligram.	Set up the titration apparatus with a pH sensor and drop counter. Calibrate the pH sensor.	Measure about 0.4 g NaOH and make 100 mL of solution in a volumetric flask. Fill the buret to the zero mark with this solution.	Calculate the concentration of the NaOH solution.	Perform the titration of the KHP solution and record the equivalence point volume.

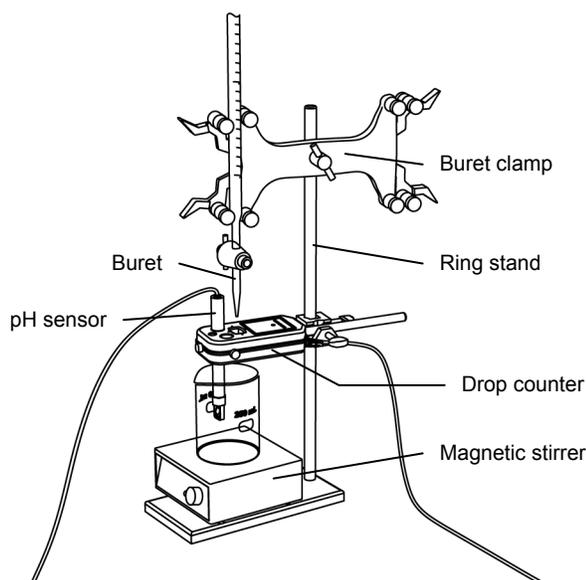
## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "♦" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

1.  Start a new experiment on the data collection system. ♦<sup>(1.2)</sup>
2.  Connect a pH sensor to the data collection system. ♦<sup>(2.1)</sup>
3.  Calibrate the pH sensor. ♦<sup>(3.6)</sup>
4.  Assemble the titration apparatus, using the steps below and the illustration as a guide.
  - a. Position the magnetic stirrer on the base of the ring stand.
  - b. Place a waste container (100-mL beaker) on the magnetic stirrer.
  - c. Use the buret clamp to attach the buret to the ring stand.
  - d. Position the drop counter over the waste container and attach it to the ring stand using the right-angle clamp.
  - e. Place the pH sensor through one of the slots in the drop counter.



**Note:** Do not connect the drop counter to the data collection system yet.

5.  Measure about 0.40 g of NaOH into a 100-mL beaker with 40 mL of deionized water to make a solution. Record the mass of the NaOH:  
  
Mass of NaOH (g):                      0.41
6.  Carefully transfer the solution into a 100-mL volumetric flask and fill the flask to the 100 mL mark with deionized water.

- 7.**  Seal the flask with its stopper, invert the flask, and shake it gently. Then vent the flask.
- 8.**  What happens if you do not mix the solution well?

The solution will not be homogeneous and every time you draw solution from the flask it will have a different concentration.

- 9.**  What is the expected concentration of the NaOH solution?

The expected concentration is 0.1 M, determined as follows:

$$c_1 = \frac{n_1}{V_1} = \frac{m_1}{FW_{\text{NaOH}} V_1}$$

$$c_1 = \frac{(0.40 \text{ g})}{(40 \frac{\text{g}}{\text{mol}})(0.1 \text{ L})} = 0.1 \text{ M}$$

- 10.**  Rinse the buret with several milliliters of the NaOH solution:
- Ensure that the stopcock is closed and rinse the inside of the buret with several milliliters of the NaOH solution.
  - Open the stopcock on the buret and drain the rinse NaOH into the waste container.
  - Repeat this process two more times.

- 11.**  Why is it necessary to rinse the buret with the NaOH solution?

If there is any residual water or contaminant in the buret, it will dilute the NaOH and change its concentration. Rinsing eliminates any such contamination.

- 12.**  Make sure the stopcock on the buret is in the “off” position and then use a funnel to fill the buret with about 50 mL of the NaOH solution (titrant).

- 13.**  Drain a small amount of the titrant through the drop counter into the waste beaker to remove any air in the tip of the buret.

- 14.**  Why is it important to remove air from the tip of the buret?

Any air trapped in the buret tip is counted as volume of NaOH. If this happens, the amount of titrant used will be inaccurate.

- 15.**  Cover the beaker containing the remaining titrant solution with a piece of Parafilm<sup>®</sup> or aluminum foil.

- 16.**  What happens if you do not cover the solution?

The solution will absorb CO<sub>2</sub> from the air and the NaOH concentration will change.

## Lab 6: Standardizing a Solution of Sodium Hydroxide

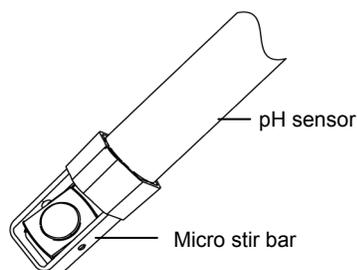
17.  Practice adjusting the stopcock on the buret so that the titrant goes through the drop counter in distinguishable drops that fall at about 1 to 2 drops per second.

**Note:** Good control of the stopcock is important. Each drop should result in a blink of the LED on the drop counter. If the LED is continuously lit, you have opened the stopcock too far and you will have to start over.

18.  Why will it be necessary to start your titration over again if you accidentally allow the titrant to stream out of the stopcock instead of emerging by drops?

The drop counter counts distinct drops. If the drops are not sufficiently distinct from one another, the drop counter will not function properly and the fluid volume will not be accurate.

19.  Add the micro stir bar to the end of the pH sensor.



20.  Why is it necessary to stir the solution during a titration?

Stirring thoroughly mixes the ions in the solution so that the recorded pH reflects the pH of the entire solution

21.  Add additional NaOH to the buret so the solution is above the zero mark. Allow some of the NaOH solution to drip into the waste container until the bottom of the meniscus is lined up with or just below the zero mark and record the initial reading in Table 1.

22.  Remove the waste container.

23.  Measure about 0.200 g of KHP to the nearest milligram and record this value in Table 2. Then place the KHP, with about 50 mL of water, into a 100-mL beaker.

24.  Place the beaker containing the KHP solution on the magnetic stirrer.

25.  Add enough deionized water to the solution so that the tip of the pH sensor is covered with solution.

26.  Turn on the magnetic stirrer at a slow and steady rate.

27.  What will happen if the solution is stirred too fast? What will happen if it is too slow?

If the solution is stirred too fast, the solution will spatter or spill, introducing error. If it is too slow, the titrated solution will not be homogeneous and the pH sensor will report a false reading.

28.  Connect the drop counter to the data collection system. ♦<sup>(2,2)</sup>

29.  Display the pH on the y-axis of a graph and Drop Count on the x-axis.  $\diamond^{(7.1.1)}$

### Collect Data

30.  Clean the lens of the drop counter inside the opening through which the drops are going with water and a cotton swab or tissue.
31.  Start recording data.  $\diamond^{(6.2)}$
32.  Turn the buret stopcock carefully, allowing the titrant to drip slowly (1 to 2 drops per second) into the solution.
33.  Continue the titration past the equivalence point until the pH curve flattens.
34.  Why is it important to go past the equivalence point?

It is necessary to go past the equivalence point in order to find the point where the slope is the steepest. The curve needs to continue past the steepest point to ensure you can tell when the slope begins to flatten.

35.  Stop recording data.  $\diamond^{(6.2)}$
36.  In Table 1, record the final drop count and the final volume of the titrant in the buret to a precision of 0.01 mL.
37.  Calculate the volume of titrant (final reading minus initial reading) and record this value in Table 1.

Table 1: Titration data

Titration Information	Trial 1	Trial 2	Trial 3
Initial reading of NaOH in the buret (to 0.01 mL)	0.00	0.00	0.00
Final reading of NaOH in the buret (to 0.01 mL)	17.20	16.90	16.20
Volume of titrant (to 0.01 mL)	17.20	16.90	16.20
Final drop count	192	201	202

38.  Calibrate the drop counter.  $\diamond^{(3.4)}$
39.  Set the horizontal axis to the calculated volume.  $\diamond^{(7.1.9)}$
40.  Print the graph.  $\diamond^{(11.2)}$

## Lab 6: Standardizing a Solution of Sodium Hydroxide

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- 41.**  In Table 2, record the volume of titrant used to reach the equivalence point. The equivalence point will be where the slope of the titration curve is the steepest. Find the steepest slope of the data plot to determine this point. ♦<sup>(9.3)</sup>
- 42.**  Refill the buret above the zero mark with the NaOH solutions.
- a.** Fill the buret above the zero mark and allow some of the NaOH solution to drip into a waste container until the bottom of the meniscus is lined up with the zero mark or just below.
- b.** Record the starting point in Table 1.
- 43.**  Clean the lens of the drop counter between runs with water and a cotton swab or tissue.
- 44.**  Repeat the titration procedure with two more KHP samples. Record the mass of the samples in Table 2.
- 45.**  Once you determine the concentration of the NaOH solution, label the solution and place it in a safe place for use in the “Acid–Base Titration” activity.
- 46.**  Save your experiment ♦<sup>(11.1)</sup> and clean up according to your teacher's instructions.

### Data Analysis

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- 1.**  What is the expected concentration of the NaOH titrant?

0.1 M

- 2.**  Determine the number of moles of KHP and NaOH used in the titration.

$$\text{For Trial 1: } n_2 = \frac{(0.2160 \text{ g})}{(204.22 \frac{\text{g}}{\text{mol}})} = 1.058 \times 10^{-3} \text{ mol KHP}$$

The number of moles of KHP reacts with the same number of moles of NaOH, so there is  $1.058 \times 10^{-3}$  mol of NaOH.

- 3.**  What is the concentration of the NaOH solution for each trial? What is the average concentration?

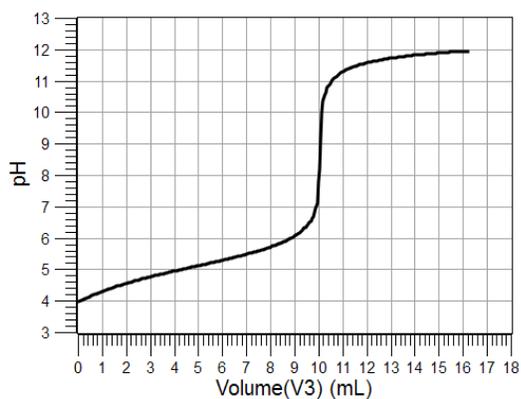
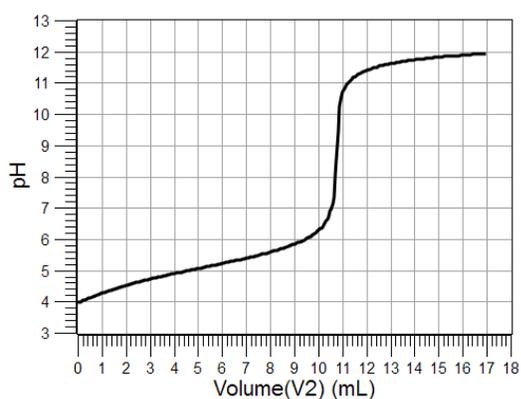
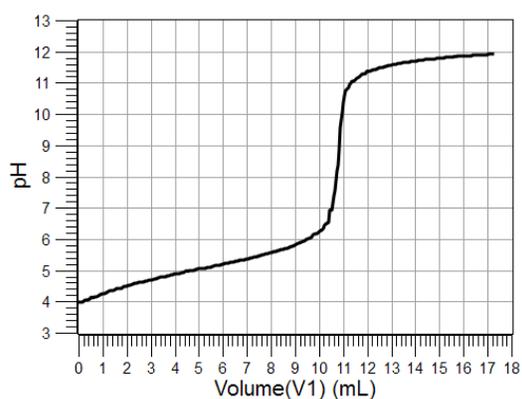
$$\text{For Trial 1: } c_1 = \frac{(1.058 \times 10^{-3} \text{ mol})}{(1.080 \times 10^{-2} \text{ L})} = 0.09790 \text{ mol/L}$$

The average concentration is  $(0.09790 \text{ mol/L} + 0.1000 \text{ mol/L} + 0.1000 \text{ mol/L})/3 = 0.09932 \text{ mol/L}$

Table 2: Determination of the NaOH solution concentration

Parameter	Trial 1	Trial 2	Trial 3
Mass of KHP (g)	0.2160	0.2210	0.2070
Number of moles of KHP (mol)	$1.058 \times 10^{-3}$	$1.082 \times 10^{-3}$	$1.014 \times 10^{-3}$
Number of moles of NaOH that can react the KHP (mol)	$1.058 \times 10^{-3}$	$1.082 \times 10^{-3}$	$1.014 \times 10^{-3}$
Volume of titrant to reach the equivalence point (mL)	10.80	10.80	10.10
Concentration of NaOH solution (mol/L)	0.09790	0.1000	0.1000
Average concentration of NaOH solution (mol/L)	0.09932		

4.  Paste or sketch the titration curves for each of the data runs.



## Analysis Questions

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**1.** If you measure 0.001 mol of KHP and you know the approximate concentration of NaOH is 0.1 M, what do you expect the volume of NaOH must be to reach the equivalence point?

$$V = \frac{n}{c}$$

$$V = \frac{(0.001 \text{ mol})}{0.1 \frac{\text{mol}}{\text{L}}} = 0.01 \text{ L} = 10 \text{ mL}$$

**2.** If you measure the NaOH and notice there is moisture on the pellets, how does the moisture affect the concentration of the NaOH solution?

It will lower the actual concentration since only part of the measured mass is NaOH.

**3.** Do you have to worry about experimental error if there is moisture on the NaOH pellets?

No, the solution will be standardized anyway.

## Synthesis Questions

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Use available resources to help you answer the following questions.

**1.** If no NaOH is available, would KOH work in place of NaOH?

Yes, it would. KOH, however, is more sensitive to both moisture and  $\text{CO}_2$ . You would need to be more careful.

**2.** Why did we not use diluted HCl solution to standardize the NaOH solution? (Hint: HCl solutions are dilutions of concentrated HCl, which is *about* a 12 M concentration).

The exact concentration of the concentrated HCl solution is not known. Therefore, we cannot measure an exact amount of it to standardize the NaOH solution.

**3.** What method will allow you to use diluted HCl solution as a standardizing solution?

If you standardized your NaOH solution, you can use that to standardize a diluted HCl solution that you prepared.

---

**Multiple Choice Questions**

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Select the best answer or completion to each of the questions or incomplete statements below.

**1. Which of the following statements is incorrect?**

- A. An error in measuring the mass of KHP will not cause experimental error.**
- B. An error in the final volume reading from the buret will result in experimental error.**
- C. KOH could be a viable option for replacing NaOH.**
- D. Overfilling the volumetric flask with the NaOH solution does not introduce experimental error.**

**2. If you had to add 65 mL more of water to make sure that the tip of the pH sensor is covered with KHP solution (more than the 50 mL of water you initially put in the beaker), do you have to consider this additional 65 mL in your calculations?**

- A. Yes. You have to recalculate the concentration of KHP accordingly.**
- B. You do if you measured the precise amount of water that you added.**
- C. No, regardless of how much water is in the beaker, the amount of KHP is the same. Therefore, it will use the same amount of NaOH solution.**
- D. Yes, regardless of how much KHP is in the beaker, the amount of water will determine how much NaOH solution is necessary.**

**3. Does moisture in the KHP introduce error?**

- A. No, only contaminations will. The solution of KHP is aqueous, in any case.**
- B. Yes, moisture may cause some crystals to stick to the tools you use to measure the KHP.**
- C. Yes, for KHP, the measurement of mass will include an unknown amount of water. You will not know how much of that mass is KHP.**
- D. It depends on how carefully you do the experiment.**

---

**Extended Inquiry Suggestions**

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Extend the standardization process to include diluted HCl solutions. To solve problems requiring an HCl solution with known concentration, an HCl solution must be standardized. For instance, the  $\text{CaCO}_3$  content of a stomach-acid neutralizing pill can be determined using an HCl solution with known concentration.

If  $\text{KHCO}_3$  is available, consider showing that there are other standardizing materials besides KHP.

If the class has a sufficiently solid math background, consider introducing the first derivative of the pH versus the volume curve to determine the equivalence point more accurately. As the equivalence point occurs where the slope of the pH versus the volume curve is the highest, the first derivative of that function will have a sharp maximum.



## Lab 7: Acid-Base Titration

### Objectives

Students determine the molar concentration of a strong acid solution by titrating measured volumes with a strong base of known concentration.

**Note:** Use the sodium hydroxide solution students determined the concentration of in the “Standardizing a Solution of Sodium Hydroxide” activity.

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Performing a titration and using the equivalence point volume of the titrant to calculate the concentration of the analyte
- ◆ Calibrating a pH sensor and drop counter

### Time Requirement

◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

*For each student or group:*

- |   |  |
|---|--|
| ◆ Data collection system                  | ◆ Ring stand   |
| ◆ pH sensor                               | ◆ Parafilm <sup>®</sup> or aluminum foil   |
| ◆ Drop counter and micro stir bar         | ◆ Funnel   |
| ◆ Magnetic stirrer                        | ◆ Hydrochloric acid, unknown concentration, 70 mL <sup>1</sup>                               |
| ◆ Buret, 50-mL                            | ◆ Sodium hydroxide (NaOH), standardized by student in previous activity, 100 mL <sup>2</sup> |
| ◆ Graduated pipet with rubber bulb, 25-mL | ◆ Buffers, pH 4 and pH 10, 10 mL   |
| ◆ Beaker, 100-mL (2)                      | ◆ Deionized water, 100 mL  |
| ◆ Beaker, 25-mL (2)                       | ◆ Wash bottle with deionized water   |
| ◆ Clamp, right-angle                      | ◆ Cotton swab or tissue  |
| ◆ Clamp, buret                            |  |

<sup>1</sup>To prepare the solution, refer to the Lab Preparation section.

<sup>2</sup>The NaOH solution should have been determined to at least three decimal places in the range of 0.1 M concentration.

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Acid-base reactions
- ◆ Balancing chemical equations
- ◆ Basic stoichiometric calculations
- ◆ Concentration calculations (mol/L)
- ◆ Formula weight
- ◆ Preparing standard solutions

## Related Labs in This Guide

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Pre-requisites:

- ◆ Lab 6: Standardizing a Solution of Sodium Hydroxide

Labs conceptually related to this one include:

- ◆ Lab 8: Oxidation–Reduction Titration
- ◆ Lab 11: Using Different Indicators for pH Determination
- ◆ Lab 19: Properties of Buffer Solutions
- ◆ Lab 24: Determining  $K_a$  by Half-Titration of a Weak Acid
- ◆ Lab 26: Conductometric Titration
- ◆ Lab 30: Determination of the  $K_a$  values of Two Isomer Multi-Protic Acids

## Using Your Data Collection System

---

Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a sensor to the data collection system. ◆<sup>(2.1)</sup>
- ◆ Connecting multiple sensors to the data collection system ◆<sup>(2.2)</sup>
- ◆ Calibrating a drop counter ◆<sup>(3.4)</sup>
- ◆ Calibrating a pH sensor ◆<sup>(3.6)</sup>

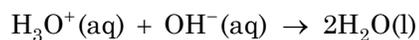
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>
- ◆ Adjusting the scale of the graph ◆<sup>(7.1.2)</sup>
- ◆ Displaying multiple data runs in a graph ◆<sup>(7.1.3)</sup>
- ◆ Changing the variable on the x-axis and y-axis of a graph ◆<sup>(7.1.9)</sup>
- ◆ Finding the slope at a point on the data plot ◆<sup>(9.3)</sup>
- ◆ Saving your experiment ◆<sup>(11.1)</sup>
- ◆ Printing the graph. ◆<sup>(11.2)</sup>

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## **Background**

Titration, a common quantitative laboratory method, determines the concentration of a reactant. A reagent of known concentration, called the titrant, is used to react with a measured volume of the reactant, called the analyte. Because volume measurements of the analyte and titrant are key factors in this type of analysis, titration is also known as a “volumetric analysis.”

In this activity the titration is between an acid and a base, using an acid-base reaction:

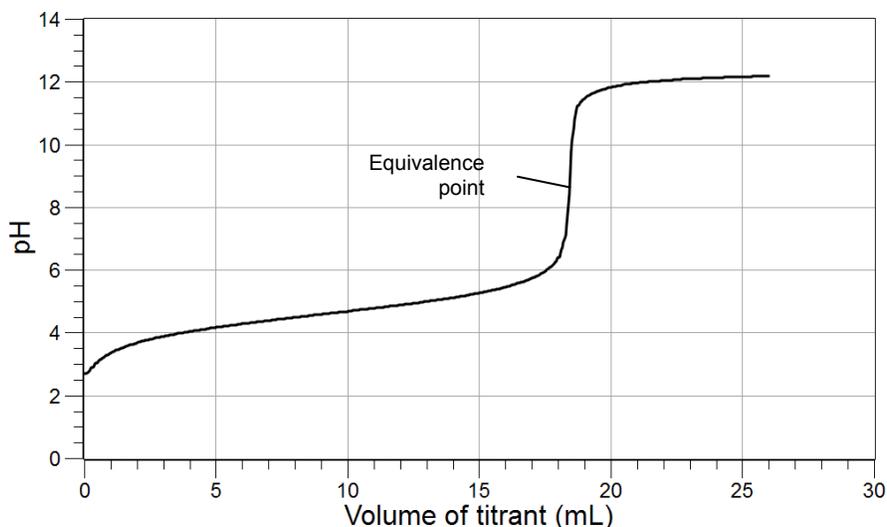


Acids contain hydronium ( $\text{H}_3\text{O}^+$ ) ions and bases contain hydroxide ( $\text{OH}^-$ ) ions. The product of an acid-base reaction is always water. This type of reaction is also known as a neutralization reaction; that is, this is a type of reaction where one reactant neutralizes the other.

## Pre-Lab Activity

### Setting the stage for the activity

When a basic solution is added to an acidic solution of unknown concentration, hydroxide ions from the basic solution react with hydronium ions from the acidic solution. This reaction forms neutral water. Since pH is a measure of the molarity of hydronium ions, the pH changes as hydronium ions react with the added hydroxide ions. The point at which the number of moles of hydroxide ions added is equal to the number of moles of hydronium ions is called the equivalence point. The detection of the equivalence point usually is easy since the pH tends to jump very sharply at that point, as shown in the graph:



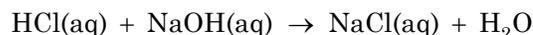
The net ionic equation shown below is an example of the reaction of a strong acid (HCl) with a strong base (NaOH).



The titration in this activity is performed using the same configuration as in the “Standardizing a Solution of Sodium Hydroxide” activity.

### Example calculation to try

In an experiment, 20.00 mL of an HCl solution with an unknown concentration was titrated with a 0.0980 M NaOH solution:



The equivalence point appeared when 22.45 mL of the NaOH solution was added to the HCl solution, which works out to the following number of moles of NaOH:

$$n_1 = c_1 V_1$$

$$n_1 = \left(0.0980 \frac{\text{mol}}{\text{L}}\right)(0.02245 \text{L})$$

$$n_1 = 2.20 \times 10^{-3} \text{ mol NaOH}$$

where

$n_1$  = number of moles of NaOH added to reach the equivalence point (mol)

$V_1$  = volume of NaOH added to reach the equivalence point (L)

$c_1$  = concentration of the NaOH standard (mol/L)

Since HCl and NaOH react in a 1:1 stoichiometric ratio, the number of moles of HCl in the sample with unknown concentration must be the same. Knowing the number of moles, the concentration of the hydrochloric acid solution can be calculated:

$$n_1 = n_2$$

$$n_2 = c_2 V_2$$

$$c_2 = \frac{n_2}{V_2}$$

$$c_2 = \frac{(2.20 \times 10^{-3} \text{ mol HCl})}{(0.02000 \text{ L})}$$

$$c_2 = 0.110 \text{ M}$$

where

$n_1$  = number of moles of NaOH added to reach the equivalence point (mol)

$n_2$  = number of moles of HCl in the sample solution (mol)

$V_2$  = volume of the HCl sample (L)

$c_2$  = concentration of the HCl solution (mol/L)

Therefore, the concentration of the unknown HCl solution is 0.1100 M.

**1. Does it matter what the volume is of the unknown solution you choose to titrate?**

No, if more solution was chosen, then the reaction would require proportionally more NaOH solution.

**2. Assuming an experiment is performed using a 50-mL buret filled to the zero mark with a standardized 0.10 M NaOH solution, and the approximate concentration of the HCl solution being titrated is 0.05 M, what is the logical amount of the unknown that you would titrate? Explain.**

With a 50-mL buret, using about 25 mL of the standard is common. Knowing the concentration of HCl to be about half the concentration of NaOH, twice the volume of the HCl solution, 50 mL, will have the same number of moles as 25 mL of NaOH solution.

**3. How would you measure precisely 20.00 mL of the solution?**

For the most precise measurement, use a 20-mL volumetric pipet. A 20-mL or 25-mL graduated pipet can provide sufficient accuracy as well.

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

1. **0.100 M HCl:** Carefully add 16.7 mL of 36% (concentrated) HCl to some distilled water in a 2-L volumetric flask. Fill the flask to the mark with distilled water. Label the solution “HCl—Unknown Concentration.”

## Safety

Add these important safety precautions to your normal laboratory procedures:

- ◆ If the NaOH or HCl comes in contact with skin or eyes, rinse the exposed surface thoroughly with running water.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

3	2	4	1	5
Cover the remaining NaOH solution. Pipet a precise amount of HCl solution of unknown concentration into a 100-mL beaker.	Fill the buret with the NaOH solution you standardized in a previous activity. Record the initial reading.	Start the data collection. Perform the titration of the HCl solution of unknown concentration.	Set up the titration apparatus with a pH sensor and drop counter. Calibrate the pH sensor.	Calculate the concentration of the hydrochloric acid solution.

## Procedure with Inquiry

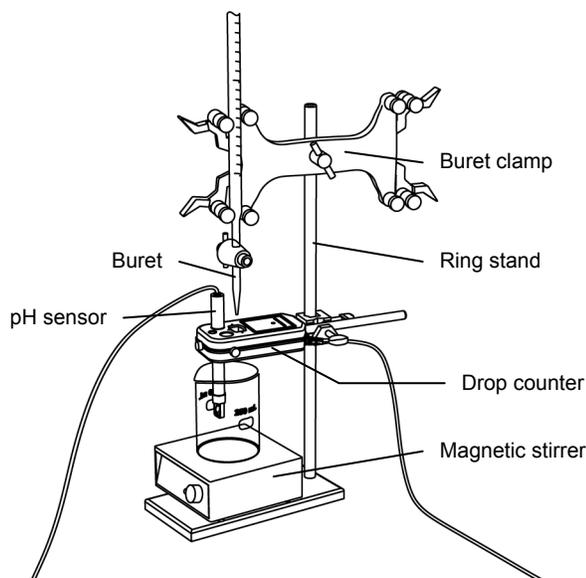
After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "◆" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

1.  Start a new experiment on the data collection system. ◆<sup>(1,2)</sup>
2.  Connect a pH sensor to the data collection system. ◆<sup>(2,1)</sup>

3.  Calibrate the pH sensor. ♦<sup>(3.6)</sup>
4.  Assemble the titration apparatus, using the steps below and the illustration as a guide.
- Position the magnetic stirrer on the base of the ring stand.
  - Place a waste container (100-mL beaker) on the magnetic stirrer.
  - Use the buret clamp to attach the buret to the ring stand.
  - Position the drop counter over the waste container and attach it to the ring stand using the right-angle clamp.
  - Place the pH sensor through one of the slots in the drop counter.



**Note:** Do not connect the drop counter to the data collection system yet.

5.  Rinse the buret with several milliliters of the standardized NaOH solution:
- Ensure that the stopcock is closed and rinse the inside of the buret with several milliliters of the NaOH solution.
  - Open the stopcock on the buret and drain the rinse NaOH into the waste container.
  - Repeat this process two more times.
6.  Why is it necessary to rinse the buret with the NaOH solution?

If there is any residual water or contaminant in the buret, it will dilute the NaOH and change its concentration. Rinsing eliminates any such contamination.

7.  Make sure the stopcock on the buret is in the “off” position and then use a funnel to fill the buret with about 50 mL of the NaOH solution (titrant).
8.  Drain a small amount of the titrant through the drop counter into the waste beaker to remove any air in the tip of the buret.
9.  Why is it important to remove air from the tip of the buret?

Any air trapped in the buret tip is counted as volume of NaOH. If this happens, the amount of titrant used will be inaccurate.

## Lab 7: Acid-Base Titration

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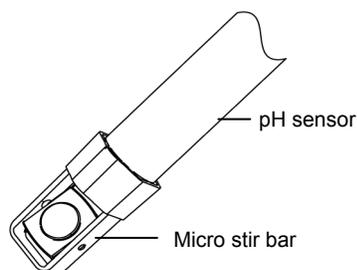
10.  Practice adjusting the stopcock on the buret so that the titrant goes through the drop counter in distinguishable drops that fall at about 1 to 2 drops per second.

**Note:** Good control of the stopcock is important. Each drop should result in a blink of the LED on the drop counter. If the LED is continuously lit, you have opened the stopcock too far and you will have to start over.

11.  Why will it be necessary to start your titration over again if you accidentally allow the titrant to stream out of the stopcock instead of emerging by drops?

The drop counter counts distinct drops. If the drops are not sufficiently distinct from one another, the drop counter will not function properly and the fluid volume will not be accurate.

12.  Add the micro stir bar to the end of the pH sensor.



13.  Why is it necessary to stir the solution during a titration?

Stirring thoroughly mixes the ions in the solution so that the recorded pH reflects the pH of the entire solution.

14.  Add additional standardized NaOH solution to the buret so the solution is above the zero mark. Allow some of the NaOH solution to drip into the waste container until the bottom of the meniscus is lined up with or just below the zero mark and record the initial reading in Table 1.

15.  Cover the beaker containing the remaining titrant solution with a piece of Parafilm<sup>®</sup> or aluminum foil.

16.  Why is it necessary to cover the beaker? (Hint: What is in the air that can potentially react with the solution?)

The CO<sub>2</sub> content of air can react with NaOH. This can change the concentration of the standardized solution, resulting in error.

17.  Remove the waste container.

### **Perform the titration of the HCl solution three times, following the steps below.**

18.  Use the graduated pipet to transfer 20.00 mL of the HCl solution into a 100-mL beaker and set the beaker on the magnetic stirrer.

19.  Add enough deionized water to the solution so the tip of the pH sensor is covered with solution.

- 20.**  Turn on the magnetic stirrer at a slow and steady rate.
- 21.**  Connect the drop counter to the data collection system.  $\diamond^{(2.2)}$
- 22.**  Display the pH on the y-axis of a graph and Drop Count on the x-axis.  $\diamond^{(7.1.1)}$

### Collect Data

- 23.**  Clean the lens of the drop counter inside the opening through which the drops are going with water and a cotton swab or tissue.
- 24.**  Start recording data.  $\diamond^{(6.2)}$
- 25.**  Turn the buret stopcock carefully, allowing the titrant to drip slowly (1 to 2 drops per second) into the solution.
- 26.**  Continue the titration past the equivalence point until the pH curve flattens.
- 27.**  Why is it important to go past the equivalence point?

It is necessary to go past the equivalence point in order to find the point where the slope is the steepest. The curve needs to continue past the steepest point to ensure you can tell when the slope begins to flatten.

- 28.**  Stop recording data.  $\diamond^{(6.2)}$
- 29.**  In Table 1, record the final drop count and the final reading of the titrant in the buret to a precision of 0.01 mL.
- 30.**  Which part of the meniscus do you read?
- Almost always, you read the bottom of the meniscus. If a solution is dark or cloudy, reading from the top of the meniscus may be necessary. Initial and final measurements always should be read from the same part of the meniscus.
- 31.**  Calculate the volume of titrant (final reading minus initial reading) and record this value in Table 1.

Table 1: Titration data

Titration Information	Trial 1	Trial 2	Trial 3
Initial reading of HCl on the buret (mL)	00.00	00.00	00.00
Final reading of HCl on the buret (mL)	28.00	26.00	34.50
Volume of titrant (mL)	28.00	26.00	34.50
Final drop count	515	486	655

## Lab 7: Acid-Base Titration

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32.  Calibrate the drop counter. ♦<sup>(3.4)</sup>
33.  Set the horizontal axis to the calculated volume. ♦<sup>(7.1.9)</sup>
34.  In Table 2, record the volume of titrant, to 2 decimal places, used to reach the equivalence point. The equivalence point will be where the slope of the titration curve is the steepest. Find the steepest slope of the data plot to determine this point. ♦<sup>(9.3)</sup>
35.  Print the graph. ♦<sup>(11.2)</sup>
36.  Clean the lens of the drop counter between runs with water and a cotton swab or tissue.
37.  Refill the buret above the zero mark with the NaOH solution:
- Fill the buret above the zero mark and allow some of the NaOH solution to drip into a waste container until the bottom of the meniscus is lined up with the zero mark or just below.
  - Record the initial reading in Table 1.
38.  Remove the beaker and dispose of its contents according to the teacher's instructions.
39.  Rinse the beaker with distilled water.
40.  Repeat the titration procedure with two more samples of the HCl solution.
41.  Save your experiment ♦<sup>(11.1)</sup> and clean up according to your teacher's instructions.

## Data Analysis

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1.  Calculate the number of moles of NaOH needed to reach the equivalence point for each trial. Record the values in Table 2.

For Trial 1:

$$n_1 = c_1 V_1$$

$$n_1 = \left(0.100 \frac{\text{mol}}{\text{L}}\right)(0.01865 \text{ L}) = 1.86 \times 10^{-3} \text{ mol}$$

2.  What is the number of moles of HCl that reacted to reach the equivalence point for each trial? Record the values in Table 2.

Since HCl and NaOH react in a 1:1 stoichiometric ratio, the number of moles of HCl in the unknown sample must be the same.

For Trial 1:  $1.86 \times 10^{-3}$  mol HCl

3.  Calculate the concentration of the HCl solution for each trial. Record the values in Table 2.

For Trial 1:

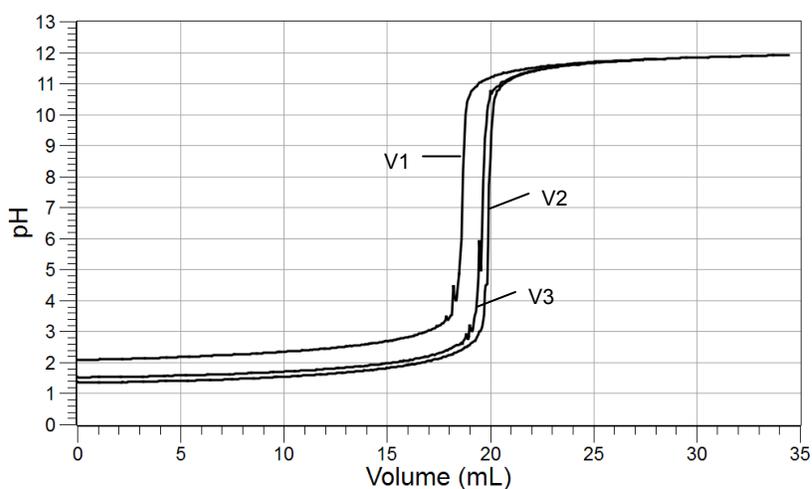
$$c_2 = \frac{n_2}{V_2}$$

$$c_2 = \frac{(1.86 \times 10^{-3} \text{ mol})}{(0.02000 \text{ L})} = 0.0930 \text{ M}$$

Table 2: Determine the concentration of the HCl solution

Parameter	Trial 1	Trial 2	Trial 3
Initial volume of acid (mL)	20.00	20.00	20.00
Concentration of NaOH solution (M)	0.100	0.100	0.100
Volume of NaOH solution to reach the equivalence point (mL)	18.65	19.90	19.59
Amount of NaOH to reach the equivalence point (mol)	$1.86 \times 10^{-3}$	$1.99 \times 10^{-3}$	$1.96 \times 10^{-3}$
Amount of HCl in the solution (mol)	$1.86 \times 10^{-3}$	$1.99 \times 10^{-3}$	$1.96 \times 10^{-3}$
Concentration of the HCl solution (M)	0.0930	0.0995	0.0980
Average concentration of the HCl solution (M)	0.0968		

4.  Sketch or paste the relevant graph below. Label the axes and each of the 3 curves.



## Analysis Questions

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**1. How would your results be different if you used a graduated cylinder to measure the unknown HCl solution instead of a pipet?**

Expect less precise results using a graduated cylinder. This means a less accurate determination of the concentration of the unknown and a greater standard deviation from the average.

**2. Why perform the experiment three times?**

Each step results in random error. In order to minimize the human error, the experiment has to be performed at least three times.

**3. What is the pH of the mixture of NaOH and HCl at the equivalence point? Explain.**

The pH is about 7. At this point the NaOH neutralized the HCl in the sample, and the pH of a neutral solution is 7.

## Synthesis Questions

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Use available resources to help you answer the following questions.

**1. If you analyze (using titration) a sulfuric acid ( $\text{H}_2\text{SO}_4$ ) solution with approximately the same concentration and volume as the HCl solution, how would the volume of NaOH consumed be different? (Hint:  $\text{H}_2\text{SO}_4$  yields two  $\text{H}^+$  ions when it dissociates.)**

About twice the volume of the NaOH solution is needed to reach the equivalence point.

**2. How would your results be different if you pipet some of the NaOH standard solution into a beaker and titrate with the unknown HCl solution?**

The results would be the same. It makes no difference which solution is the titrant and which the analyte.

## Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

**1. On which of the following does the accuracy of the measurement *not* depend?**

- A. The accuracy of the buret you are using.
- B. The accuracy of the pipet you are using.
- C. Concentration of the standard NaOH solution.
- D. How long the NaOH solution is left uncovered, exposed to air.

**2. If you substitute the same concentration of a  $\text{Ca}(\text{OH})_2$  solution for the NaOH solution, which of the following statements is correct? (Hint:  $\text{Ca}(\text{OH})_2$  yields two  $\text{OH}^-$  ions when it dissociates.) To reach the equivalence point,**

- A. It would require the same volume of  $\text{Ca}(\text{OH})_2$  as of the NaOH solution.
- B. It would require half the volume of  $\text{Ca}(\text{OH})_2$  as of the NaOH solution.
- C. It would require twice the volume of  $\text{Ca}(\text{OH})_2$  as of the NaOH solution.
- D.  $\text{Ca}(\text{OH})_2$  would not work for this experiment.

### **Extended Inquiry Suggestions**

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The acid content of waste water is a real environmental concern. If you can secure waste water samples from a chrome/nickel plating plant or a food processing plant, for example, students can analyze the samples.

Consider the additional challenge that no approximate acid concentration is available. Have students discuss how to address this problem.



## Lab 8: Oxidation–Reduction Titration

### Objectives

Students use the change in potential during an oxidation-reduction reaction to determine the concentration of a commercial, nominally 3% hydrogen peroxide solution.

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Applying titration skills to oxidation-reduction reactions; using an oxidation reduction potential probe
- ◆ Applying oxidation-reduction equations to obtain the stoichiometric ratio between the titrant and analyte
- ◆ Using the equivalence point volume of the titrant to calculate the concentration of the hydrogen peroxide solution

### Time Requirement

◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

#### *For each student or group:*

- |  |  |
|--|--|
| ◆ Data collection system                                     | ◆ Clamp, buret   |
| ◆ Oxidation reduction potential (ORP) electrode <sup>1</sup> | ◆ Ring stand   |
| ◆ Drop counter   | ◆ Hydrogen peroxide, approximately 3%,<br>1:20 dilution, 40 mL <sup>2</sup>                      |
| ◆ Magnetic stirrer and stir bar                              | ◆ 1.000 × 10 <sup>-2</sup> M Potassium permanganate (KMnO <sub>4</sub> ),<br>100 mL <sup>3</sup> |
| ◆ Buret, 50-mL   | ◆ 4 M Sulfuric acid (H <sub>2</sub> SO <sub>4</sub> ), 70 mL <sup>4</sup>                        |
| ◆ Beaker (2), 150-mL   | ◆ Water, deionized, 250 mL   |
| ◆ Volumetric pipet with rubber bulb, 10-mL                   | ◆ Wash bottle with deionized water   |
| ◆ Graduated cylinder, 50-mL                                  |  |
| ◆ Clamp, right-angle   |  |

<sup>1</sup> The ORP electrode needs an amplifier; use it with either the PASPORT Chemistry Sensor or PASPORT High Resolution pH/ORP/ISE Amplifier with Temperature Sensor.

<sup>2-4</sup> To prepare the solutions, refer to the Lab Preparation section.

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Conversion between molarity and percent by mass
- ◆ Redox reactions
- ◆ Titration

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 7: Acid–Base Titration
- ◆ Lab 12: Determination of the Rate of the Decomposition of Hydrogen Peroxide
- ◆ Lab 20: Determination of Electrochemical Series
- ◆ Lab 21: Electroplating

## Using Your Data Collection System

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a sensor to the data collection system. ◆<sup>(2.1)</sup>
- ◆ Connecting multiple sensors to your data collection system ◆<sup>(2.2)</sup>
- ◆ Calibrating the drop counter ◆<sup>(3.4)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>
- ◆ Adjusting the scale of a graph ◆<sup>(7.1.2)</sup>
- ◆ Changing the variable on the x-axis and y-axis of a graph ◆<sup>(7.1.9)</sup>

- ◆ Finding the slope at a point on the data plot ◆<sup>(9.3)</sup>
- ◆ Saving your experiment ◆<sup>(11.1)</sup>
- ◆ Printing the graph. ◆<sup>(11.2)</sup>

## Background

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Hydrogen peroxide is a commonly used oxidizing agent in a broad range of situations ranging from medical applications to propellant materials in rockets. A 3% solution of hydrogen peroxide is available in pharmacies. It is used as a common topical disinfectant solution.

Solutions with higher concentrations of hydrogen peroxide are used in industrial processes (60% to 70%) and in rocket propellants (90%). While hydrogen peroxide is a desired oxidizing agent in industrial processes because the product of the reactions is water, hydrogen peroxide solutions are highly unstable. Particularly at higher concentration, they can decompose explosively.

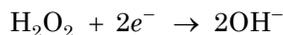
Even though the 3% hydrogen peroxide solution is stable, it decomposes slowly; the actual concentration may differ from the value printed on the label.

## Pre-Lab Activity

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### Setting the stage for the activity

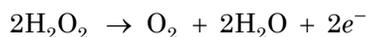
Hydrogen peroxide, in most reactions, acts as an oxidizer. It picks up electrons.



When the reaction is performed in an acidic medium, the product is water.



If, however, it is mixed with a stronger oxidizer, it can act as a reducing agent; that is, it loses electrons.



Potassium permanganate ( $\text{KMnO}_4$ ) is a stronger oxidizer than hydrogen peroxide. Therefore, it oxidizes the  $\text{H}_2\text{O}_2$ .



This reaction provides a means to determine the precise concentration of a 3%  $\text{H}_2\text{O}_2$  solution.

The titration of the  $\text{H}_2\text{O}_2$  solution of unknown concentration is performed with 0.01 M  $\text{KMnO}_4$  solution. The reaction can be followed using the ORP sensor (also referred to as an ORP electrode), which monitors the reduction-oxidation (redox) potential in the solution. (This is similar to a pH sensor monitoring the pH in a solution.)

## Lab 8: Oxidation-Reduction Titration

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The titration curve looks similar to one obtained with a pH sensor during an acid-base titration. The curve is the steepest at the equivalence point, when all the  $\text{H}_2\text{O}_2$  molecules have reacted. Also, the  $\text{KMnO}_4$  solution is purple and the products of the reaction are colorless. When there is no more  $\text{H}_2\text{O}_2$  for a reaction to continue, the first extra drop of  $\text{KMnO}_4$  colors the titrated solution.

### Example calculation to try

A 50.00 mL solution, nominally 3%  $\text{H}_2\text{O}_2$ , was titrated with a 1.000 M  $\text{KMnO}_4$  solution. The volume of  $\text{KMnO}_4$  needed to reach the equivalence point was 21.50 mL. The number of moles of  $\text{KMnO}_4$  necessary for the titration was

$$(21.50 \text{ mL KMnO}_4) \left( \frac{1.000 \text{ mol KMnO}_4}{1000 \text{ mL KMnO}_4} \right) = 0.02150 \text{ mol KMnO}_4$$

According to the stoichiometric equation (see Equation 1), 2 moles of  $\text{KMnO}_4$  reacts with 5 moles of  $\text{H}_2\text{O}_2$ . Applying this ratio to determine the number of moles of hydrogen peroxide that reacted with the permanganate gives

$$(0.02150 \text{ mol KMnO}_4) \left( \frac{5 \text{ mol H}_2\text{O}_2}{2 \text{ mol KMnO}_4} \right) = 0.05375 \text{ mol H}_2\text{O}_2$$

Therefore, the 50.00 mL sample contained  $5.375 \times 10^{-2}$  mol  $\text{H}_2\text{O}_2$ . The concentration of the sample was

$$(0.05375 \text{ mol}) \left( \frac{1000.00 \text{ mL}}{50.00 \text{ mL}} \right) = 1.075 \text{ M}$$

The number of grams of  $\text{H}_2\text{O}_2$  in 1000 mL of solution is:

$$(1.075 \text{ mol}) \left( 34.00 \frac{\text{g}}{\text{mol}} \right) = 36.55 \text{ g}$$

Therefore, the number of grams of  $\text{H}_2\text{O}_2$  in 100 mL of this solution is 3.655 g. Since the density of the solution is assumed to be 1.000 g/mL, the 100 mL solution has the mass of 100 g. With 3.655 g of  $\text{H}_2\text{O}_2$  in a 100 g solution, the result is a 3.655%  $\text{H}_2\text{O}_2$  solution.

### 1. Why is $\text{H}_2\text{O}_2$ a preferred oxidizing agent these days in industrial processes? (Hint: consider one of the most important concerns in today's society.)

$\text{H}_2\text{O}_2$  is preferred because the products are environmentally friendly.

### 2. Oxidizing agents gain electrons while reducing agents lose electrons in a reaction. What do you know about the number of transferred electrons in a reaction (that is, the number of lost electrons and the number of gained electrons)?

The number of lost electrons should be the same as the number of gained electrons; electrons are neither created nor destroyed in a redox reaction.

**3. How can  $\text{H}_2\text{O}_2$  be a reducing agent when it is known as one of the strongest oxidizing agents?**

Being either an oxidizing or reducing agent is not absolute. It always depends on the partner in the reaction. Since the  $\text{KMnO}_4$  is a stronger oxidizer, it forces the hydrogen peroxide to lose electrons, which are taken by the  $\text{KMnO}_4$ . The  $\text{KMnO}_4$  is reduced. In this case the hydrogen peroxide is a reducing agent.

**4. Which method is better to follow the titration: using an ORP electrode or using the  $\text{KMnO}_4$  as an indicator, since it is purple and the products of the titration are colorless? Explain.**

The ORP electrode provides an objective way of following the reaction. Determining the color is subjective. Therefore, using the probe is more accurate.

**5. In this reaction, how many electrons does the Mn gain in  $\text{MnO}_4^-$ ? How many electrons does an  $\text{H}_2\text{O}_2$  molecule lose?**

Manganese gains 5 electrons and each  $\text{H}_2\text{O}_2$  molecule loses 2 (each O loses one).

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**Lab Preparation**

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These are the materials and equipment to set up prior to the lab:

- 1. 0.15%  $\text{H}_2\text{O}_2$ :** A 3% hydrogen peroxide solution is readily available from any pharmacy. Measure 25 mL of a 3% hydrogen peroxide solution and pour it into a 0.5 L volumetric flask. Fill the flask to the mark with distilled water.
- 2.  $1.000 \times 10^{-2}$  M  $\text{KMnO}_4$ :** Measure 0.8 g of  $\text{KMnO}_4$  and transfer it into a 0.5 L volumetric flask, fill to the mark with distilled water and dissolve all the crystals.

**Note:** Because the solution is dark, make sure that all the crystals dissolve before filling the flask to the mark.

**Note:** This method prepares a solution with an approximate concentration. Standardization is necessary to determine the exact concentration of the solution using an appropriate primary standard.

- 3. 4 M  $\text{H}_2\text{SO}_4$ :** Measure 109 mL of 96 to 98% sulfuric acid solution. Add about 0.25 L of distilled water to an empty 0.5-L Erlenmeyer flask and *slowly* add the sulfuric acid solution to the water while constantly mixing.

**Important:** Do *not* perform a dilution of sulfuric acid in a volumetric flask. The solution may heat up excessively and crack it. Volumetric flasks are usually not heat resistant.

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**Safety**

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Add these important safety precautions to your normal laboratory procedures:

- ◆ Wash any  $\text{KMnO}_4$  solution that comes in contact with your skin with large amounts of water.  $\text{KMnO}_4$  can cause brown discoloration of the skin.
- ◆ Wash any  $\text{H}_2\text{O}_2$  solution or  $\text{H}_2\text{SO}_4$  solution that comes in contact with your skin with large amounts of water.  $\text{H}_2\text{O}_2$  can cause white discoloration of the skin.  $\text{H}_2\text{SO}_4$  can cause burns.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

1	3	5	4	2
Set up the titration apparatus with an oxidation reduction probe and drop counter. Fill the buret with $\text{KMnO}_4$ solution.	Start the data collection. Perform the titration.	Calculate the concentration of the $\text{H}_2\text{O}_2$ solution.	Stop adding the $\text{KMnO}_4$ solution after the curve begins to level off after the equivalence point.	Obtain a precise amount of the $\text{H}_2\text{O}_2$ solution. Add 4M $\text{H}_2\text{SO}_4$ solution to create an acidic environment.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

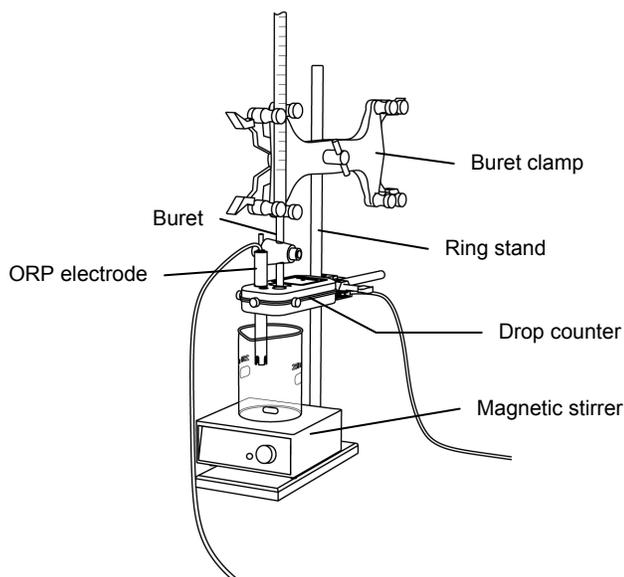
**Note:** When students see the symbol "♦" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

- Start a new experiment on the data collection system. ♦<sup>(1.2)</sup>
- Connect the oxidation reduction potential (ORP) electrode to the data collection system. ♦<sup>(2.1)</sup>

**Note:** No calibration procedure is needed for this experiment. You do not use the numerical redox value; you use the probe to determine the point at which there is a jump in the redox potential.

3.  Assemble the titration apparatus, using the steps below and the illustration as a guide.
- Position the magnetic stirrer on the base of the ring stand.
  - Place a waste container (150-mL beaker) on the magnetic stirrer.
  - Use the buret clamp to attach the buret to the ring stand.
  - Position the drop counter over the waste container and attach it to the ring stand using the right-angle clamp.
  - Place the ORP electrode through one of the slots in the drop counter.



**Note:** Do not connect the drop counter to the data collection system yet.

4.  Rinse the buret with several milliliters of the  $1.000 \times 10^{-2}$  M  $\text{KMnO}_4$  solution:
- Ensure that the stopcock is closed and rinse the inside of the buret with several milliliters of the standardized  $\text{KMnO}_4$  solution.
  - Open the stopcock on the buret and drain the rinse  $\text{KMnO}_4$  solution into the waste container.
  - Repeat this process two more times.

5.  Why is it necessary to rinse the buret with the  $\text{KMnO}_4$  solution?

If there is any residual water or contaminant in the buret, it will dilute the  $\text{KMnO}_4$  and change its concentration. Rinsing eliminates any such contamination.

6.  Make sure the stopcock on the buret is in the “off” position and then use a funnel to fill the buret with about 50 mL of the  $\text{KMnO}_4$  solution (titrant).
7.  Drain a small amount of the titrant through the drop counter into the waste beaker to remove any air in the tip of the buret.
8.  Why is it important to remove air from the tip of the buret?

Any air trapped in the buret tip is counted as volume of  $\text{KMnO}_4$ . If this happens, the amount of titrant used will be inaccurate.

## Lab 8: Oxidation-Reduction Titration

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9.  Practice adjusting the stopcock on the buret so that the titrant goes through the drop counter in distinguishable drops that fall at about 1 to 2 drops per second.

**Note:** Good control of the stopcock is important. Each drop should result in a blink of the LED on the drop counter. If the LED is continuously lit, you have opened the stopcock too far and you will have to start over.

10.  Why will it be necessary to start your titration over again if you accidentally allow the titrant to stream out of the stopcock instead of emerging by drops?

The drop counter counts distinct drops. If the drops are not sufficiently distinct from one another, the drop counter will not function properly and the fluid volume will not be accurate.

11.  Add additional  $1.000 \times 10^{-2}$  M  $\text{KMnO}_4$  to the buret so the solution is above the zero mark. Allow some of the  $\text{KMnO}_4$  solution to drip into the waste container until the top of the meniscus is lined up with or just below the zero mark and record the initial reading in Table 1.

12.  Why are you instructed to set the top of the meniscus to the zero mark instead of the bottom of the meniscus?

$\text{KMnO}_4$  solution has a deep purple color which makes it difficult to read the bottom of the meniscus.

13.  Remove the waste container.

**Perform the titration of a  $\text{H}_2\text{O}_2$  solution three times, following the steps below.**

14.  Use the volumetric pipet to transfer 10.00 mL of the  $\text{H}_2\text{O}_2$  solution into a 150-mL beaker.
15.  Using the graduated cylinder, transfer 20.00 mL of the 4 M  $\text{H}_2\text{SO}_4$  into the same 150-mL beaker.

**Note:** Handle sulfuric acid carefully.

16.  Add enough deionized water to the beaker so the tip of the ORP electrode is covered with solution.
17.  Add the magnetic stir bar to the beaker.
18.  Turn on the magnetic stirrer at a slow and steady rate.
19.  Connect the drop counter to the data collection system.  $\diamond^{(2,2)}$
20.  Display the Potential on the y-axis of a graph and Drop Count on the x-axis.  $\diamond^{(7.1.1)}$

**Collect Data**

**21.**  Clean the lens of the drop counter inside the opening through which the drops are going with water and a cotton swab or tissue.

**22.**  Start recording data.  $\diamond^{(6.2)}$

**23.**  Turn the buret stopcock carefully, allowing the titrant to drip slowly (1 to 2 drops per second) into the solution.

**24.**  Continue the titration past the equivalence point until the pH curve flattens.

**25.**  Why is it important to go past the equivalence point?

It is necessary to go past the equivalence point in order to find the point where the slope is the steepest. The curve needs to continue past the steepest point to ensure you can tell when the slope begins to flatten.

**26.**  Stop recording data.  $\diamond^{(6.2)}$

**27.**  In Table 1, record the final drop count and the final reading of the titrant in the buret to a precision of 0.01 mL.

**28.**  Calculate the volume of titrant (final reading minus initial reading) and record this value in Table 1.

Table 1: Titration data for drop counter calibration

Titration Information	Trial 1	Trial 2	Trial 3
Initial reading of $\text{KMnO}_4$ on the buret (mL)	0.00	0.00	0.00
Final reading of $\text{KMnO}_4$ on the buret (mL)	21.00	18.80	17.90
Volume of titrant (mL)	21.00	18.80	17.90
Final drop count	428	386	357

**29.**  Calibrate the drop counter.  $\diamond^{(3.4)}$

**30.**  Set the horizontal axis to the calculated volume.  $\diamond^{(7.1.9)}$

**31.**  In Table 2, record the volume of  $\text{KMnO}_4$  (to a precision of 0.01 mL) used to reach the equivalence point. The equivalence point will be where the slope of the titration curve is the steepest. Find the steepest slope of the data plot to determine this point.  $\diamond^{(9.3)}$

**32.**  Print the graph.  $\diamond^{(11.2)}$

## Lab 8: Oxidation-Reduction Titration

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- 33.**  Refill the buret above the zero mark with the  $\text{KMnO}_4$  solution.
- Fill the buret above the zero mark and allow some of the  $\text{KMnO}_4$  solution to drip into a waste container until the top of the meniscus is lined up with the zero mark or just below.
  - Record the starting point in Table 1.
- 34.**  Rinse the ORP electrode tip with deionized water.
- 35.**  Remove the beaker and dispose of its contents according to the teacher's instructions.
- 36.**  Rinse the beaker with distilled water.
- 37.**  Repeat the titration procedure with two more  $\text{H}_2\text{O}_2$  samples.
- 38.**  Save your experiment  $\diamond^{(11.1)}$  and clean up according to your teacher's instructions.

### Data Analysis

---

- 1.**  Calculate the number of moles of  $\text{KMnO}_4$  added to reach the equivalence point. Record this value in Table 2.

For Trial 1:

$$(17.32 \text{ mL KMnO}_4) \frac{(1.000 \times 10^{-2} \text{ mol KMnO}_4)}{(1000 \text{ mL KMnO}_4)} = 1.732 \times 10^{-4} \text{ mol KMnO}_4$$

- 2.**  Calculate the number of moles of  $\text{H}_2\text{O}_2$  in the 10.00 mL sample. Record this value in Table 2.

According to the stoichiometric equation (Equation 1), 2 mol of  $\text{KMnO}_4$  reacts with 5 mol of  $\text{H}_2\text{O}_2$ :

For Trial 1:

$$(1.732 \times 10^{-4} \text{ mol KMnO}_4) \frac{(5 \text{ mol H}_2\text{O}_2)}{(2 \text{ mol KMnO}_4)} = 4.330 \times 10^{-4} \text{ mol H}_2\text{O}_2$$

- 3.**  Determine the concentration of the  $\text{H}_2\text{O}_2$  solution. Record these values in Table 2.

For Trial 1: The 10.00 mL sample contained  $4.330 \times 10^{-4}$  mol  $\text{H}_2\text{O}_2$ . The concentration of the sample is

$$(4.330 \times 10^{-4} \text{ mol}) \frac{(1000.00 \text{ mL})}{(10.00 \text{ mL})} = 4.330 \times 10^{-2} \text{ M}$$

- 4.**  How many grams of  $\text{H}_2\text{O}_2$  are in 1000 mL of this solution?

For Trial 1:

$$(4.330 \times 10^{-2} \text{ M}) \times (34.00 \text{ g/mol}) = 1.472 \text{ g H}_2\text{O}_2/\text{L}$$

5.  Determine the percent concentration of the  $\text{H}_2\text{O}_2$  solution. What is the average concentration, based on the three trials. (The density of the solution is assumed to be 1.000 g/mL, so the mass of 1000 mL is 1000 g.)

For Trial 1:

$$\frac{1.472 \text{ g H}_2\text{O}_2}{1000 \text{ g solution}} \times 100 = 0.1472\%$$

6.  Since the hydrogen peroxide solution was a 1:20 dilution of the 3% hydrogen, calculate the percent concentration of the original solution. Record this value in Table 2.

Using the average:

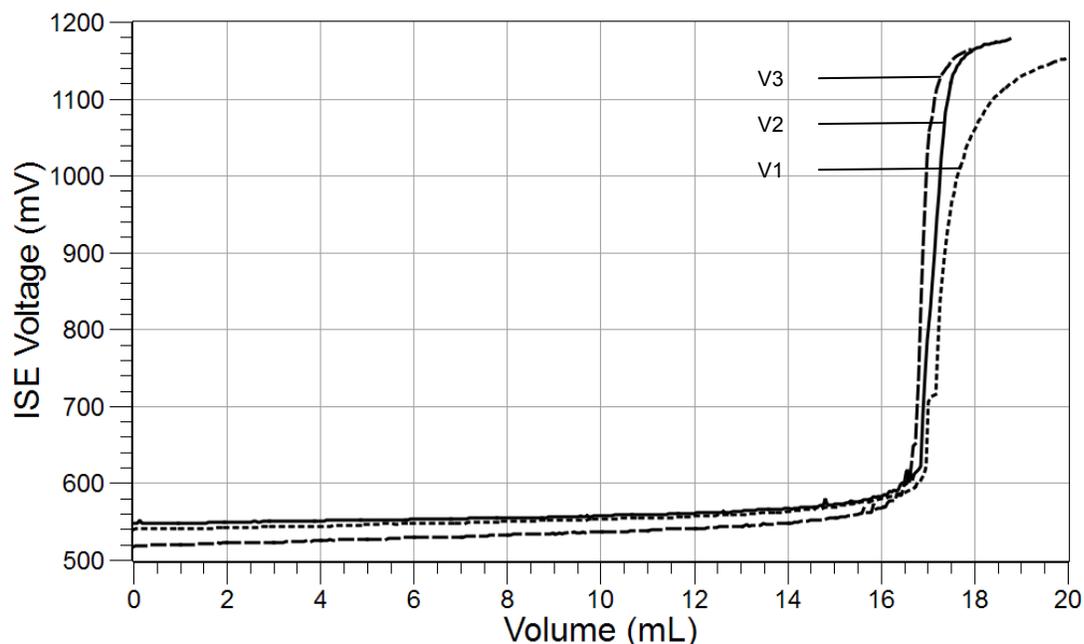
$$0.1458\% \times 20 = 2.916\%$$

Table 2: Determine the concentration of  $\text{H}_2\text{O}_2$

Parameter	Trial 1	Trial 2	Trial 3
Expected concentration of $\text{H}_2\text{O}_2$ (%)	3%		
Volume of $\text{KMnO}_4$ added to reach the equivalence point (mL)	17.32	17.10	17.05
Amount of $\text{KMnO}_4$ added to reach the equivalence point (mol)	$1.732 \times 10^{-4}$	$1.710 \times 10^{-4}$	$1.705 \times 10^{-4}$
Amount of $\text{H}_2\text{O}_2$ in 10.00 mL sample (mol)	$4.330 \times 10^{-4}$	$4.275 \times 10^{-4}$	$4.263 \times 10^{-4}$
Concentration of $\text{H}_2\text{O}_2$ solution (M)	$4.330 \times 10^{-2}$	$4.275 \times 10^{-2}$	$4.263 \times 10^{-2}$
Concentration of $\text{H}_2\text{O}_2$ solution (%)	0.1472	0.1453	0.1449
Average concentration of $\text{H}_2\text{O}_2$ solution (%)	0.1458		
Concentration of original $\text{H}_2\text{O}_2$ solution	2.916		

## Lab 8: Oxidation-Reduction Titration

7.  Sketch or attach the graph of your three data runs of oxidation reduction potential, measured as ISE Voltage versus Volume.



### Analysis Questions

1. If you were given a sample of  $\text{H}_2\text{O}_2$  from an old bottle of hydrogen peroxide to determine its concentration by titration, do you expect a greater or lesser volume of  $\text{KMnO}_4$  needed to reach the equivalence point than if it were from a new bottle? Explain.

A lesser volume of  $\text{KMnO}_4$  is expected because  $\text{H}_2\text{O}_2$  slowly decomposes, resulting in less  $\text{H}_2\text{O}_2$  content.

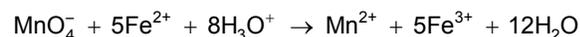
2. Does the fact that you set the top of the meniscus to the zero mark and take your final reading at the top of the meniscus introduce an error? Explain.

No, because it helps assure accuracy when finding the difference between the two readings. The volume of the solution between the tops of the two menisci and the bottoms of the two menisci are the same.

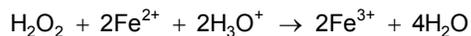
### Synthesis Questions

Use available resources to help you answer the following questions.

1. If you were to adapt this titration method to determine the amount of iron(II) ions in an unknown solution, what would be the balanced chemical equation between  $\text{MnO}_4^-$  and  $\text{Fe}^{2+}$  in the presence of  $\text{H}_2\text{SO}_4$  ( $\text{H}_3\text{O}^+$ )? (Hint: the products of this reaction are  $\text{Mn}^{2+}$ ,  $\text{Fe}^{3+}$ , and  $\text{H}_2\text{O}$ .)



**2.**  $\text{H}_2\text{O}_2$  is a strong enough oxidizer to oxidize iron(II) as well. What would be the balanced chemical equation between  $\text{H}_2\text{O}_2$  and  $\text{Fe}^{2+}$  in the presence of  $\text{H}_2\text{SO}_4$  ( $\text{H}_3\text{O}^+$ )? (Hint: the products of this reaction are  $\text{Fe}^{3+}$  and  $\text{H}_2\text{O}$ .)



## Multiple Choice Questions

Select the best answer or completion to each of the questions or incomplete statements below.

**1.** Glass surfaces are known to help the spontaneous decomposition of  $\text{H}_2\text{O}_2$ . How does the use of a buret, a pipet, and a beaker influence the  $\text{KMnO}_4$  consumption?

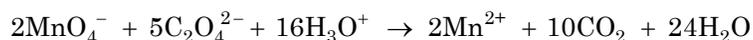
- A.** It would not affect the  $\text{KMnO}_4$  consumption since the glass has no effect on  $\text{KMnO}_4$ .
- B.** It would result in the consumption of more  $\text{KMnO}_4$  because the product of the decomposition of  $\text{H}_2\text{O}_2$ ,  $\text{O}_2$ , would also consume some  $\text{KMnO}_4$ .
- C.** Less  $\text{H}_2\text{O}_2$  in the solution in the glass beaker would result in less  $\text{KMnO}_4$  consumption.
- D.** It depends on the contamination of  $\text{H}_2\text{O}_2$ .

**2.** What is the electron change in  $\text{MnO}_4^-$  and  $\text{H}_2\text{O}_2$ ?

- A.**  $\text{MnO}_4^-$  gains 5 electrons and  $\text{H}_2\text{O}_2$  loses 2 electrons.
- B.**  $\text{MnO}_4^-$  gains 2 electrons and  $\text{H}_2\text{O}_2$  loses 5 electrons.
- C.**  $\text{MnO}_4^-$  loses 5 electrons and  $\text{H}_2\text{O}_2$  gains 2 electrons.
- D.**  $\text{MnO}_4^-$  loses 2 electrons and  $\text{H}_2\text{O}_2$  gains 5 electrons.

## Extended Inquiry Suggestions

$\text{KMnO}_4$  is not a stable chemical in solution. Because it readily oxidizes many materials, its precise concentration has to be determined with a primary standard before each use. Sodium oxalate ( $\text{Na}_2\text{C}_2\text{O}_4$ ) is such a standard.



If there is time available, it is worth performing the standardization of the  $\text{KMnO}_4$  titrant with  $\text{Na}_2\text{C}_2\text{O}_4$ :

Measure 0.8375 g of  $\text{Na}_2\text{C}_2\text{O}_4$  ( $FW = 134$  g/mol) and put it into a 100-mL beaker. Add 5 mL of 4 M  $\text{H}_2\text{SO}_4$ . Titrate the solution at 80 °C using the color of  $\text{KMnO}_4$  to determine the end of the reaction.

**Important:** Do not use the ORP electrode. The high temperature will ruin it. The high temperature is necessary to eliminate the produced  $\text{CO}_2$ .



## Lab 9: Mole Relationships in a Chemical Reaction

### Objectives

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Students determine the stoichiometric coefficients of the reactants of a chemical reaction using conductivity.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Using conductivity to monitor a reaction
- ◆ Determining the stoichiometric ratio of the reactants

### Time Requirement

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◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

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#### *For each student or group:*

- ◆ Data collection system
- ◆ Conductivity sensor
- ◆ Test tubes (9), 15-mL
- ◆ Beaker, 100-mL
- ◆ Graduated pipet (2), 10-mL
- ◆ Rubber bulb
- ◆ Test tube rack
- ◆ Unknown solution, 50 mL<sup>1</sup>
- ◆ 0.01 M Silver nitrate (AgNO<sub>3</sub>), 50 mL<sup>2</sup>
- ◆ Wash bottle with deionized water
- ◆ Parafilm<sup>®</sup>
- ◆ Marking pen

<sup>1-2</sup> To prepare the solutions, refer to the Lab Preparation section. The unknown solution is 0.01 M potassium chromate (K<sub>2</sub>CrO<sub>4</sub>).

## **Concepts Students Should Already Know**

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Students should be familiar with the following concepts:

- ◆ Balancing chemical equations
- ◆ Dissociation
- ◆ Molarity
- ◆ Precipitate-forming reactions
- ◆ Stoichiometric calculations

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 1: Determining the Empirical Formula of a Compound
- ◆ Lab 2: Determine the Percentage of Water in a Hydrate
- ◆ Lab 15b: Analysis of a Coordination Compound
- ◆ Lab 16: Gravimetric Determination of a Precipitate

## **Using Your Data Collection System**

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

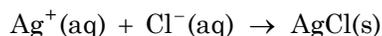
- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a sensor to your data collection system ◆<sup>(2.1)</sup>
- ◆ Monitoring live data without recording ◆<sup>(6.1)</sup>
- ◆ Displaying digits in a digits display ◆<sup>(7.3.1)</sup>

## **Background**

---

Ionic compounds dissociate in an aqueous medium to form ions. Ions are responsible for the ability of solutions to conduct electricity. The electrical conductivity of a solution increases with greater ion concentration or with solutions of ions that have a greater charge.

When a chemical reaction between two solutions removes the ions from the combined solution in the form of a gas or precipitate, the conductivity of the solution is reduced. For example, when an  $\text{AgNO}_3$  solution and a  $\text{NaCl}$  solution are combined, the  $\text{Ag}^+$  and  $\text{Cl}^-$  ions are removed in the form of  $\text{AgCl}$ , since  $\text{AgCl}$  is not soluble in water:



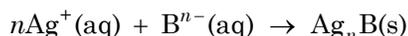
When the two solutions are combined in a ratio other than the stoichiometric ratio, the ions of one of the reactants will be in excess, resulting in greater conductivity. When the two solutions are combined in a ratio that has an equal amount of the reactants, the maximum number of ions is removed. In that case, the conductivity will be least.

## Pre-Lab Activity

---

### Setting the stage for the activity

You will study the reaction between  $\text{Ag}^+$  and an unknown ion,  $\text{B}^{n-}$ :



where:

$n$  = the number of silver ions in  $\text{Ag}_n\text{B}$ ; also, the number of negative charges on the unknown anion, B

B = the formula of the unknown anion

You will combine various volume combinations of the solution containing the unknown anion and the solution of  $\text{Ag}^+$  ions, until the conductivity is minimized. If the concentrations of the two solutions are the same, the ratio of the two solutions will allow you to determine the ratio between the stoichiometric coefficients of the two reactants.

The two solutions will be mixed in test tubes and the conductivity of the resulting solution will be measured with the conductivity sensor.

## Lab 9: Mole Relationships in a Chemical Reaction

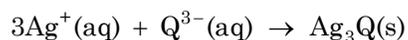
### Example calculation to try

In an experiment, various ratios of volumes of  $\text{Ag}^+$  solution and the solution of an unknown anion,  $\text{Q}^{n-}$ , with equal concentrations, were combined. Since the concentrations were the same, the ratio of the volumes combined is the same as the ratio of number of moles combined. The conductivity of the resulting solutions was measured, in microsiemens ( $\mu\text{S}$ ), and the following data was obtained:

Table 1: Determination of the stoichiometric ratio using conductivity

Volume of $\text{Ag}^+$ (mL)	Volume $\text{Q}^{n-}$ (mL)	Volume Ratio $\text{Ag}^+:\text{Q}^{n-}$	Likely Stoichiometric Ratio	Conductivity ( $\mu\text{S}$ )
2.00	8.00	1 : 4	1 : 4	2545
2.50	7.50	1 : 3	1 : 3	2134
3.00	7.00	1 : 2.33	1 : 2	1876
4.00	6.00	1 : 1.5	2 : 3	1544
5.00	5.00	1 : 1	1 : 1	1265
6.00	4.00	1.5 : 1	3 : 2	987
7.00	3.00	2.33 : 1	2 : 1	544
7.50	2.50	3 : 1	3 : 1	125
8.00	2.00	4 : 1	4 : 1	899

Since the minimum conductivity,  $125 \mu\text{S}$ , was achieved at the 3:1 ratio, it is most likely that the  $\text{Ag}^+$  ions react with the  $\text{Q}^{n-}$  ions in the 3:1 ratio:



If the minimum conductivity was achieved with a ratio of solutions other than a ratio between integers, the numbers would have to be multiplied with an integer to obtain a ratio between the lowest possible integers. For example, if the ratio was 1.5:1, after multiplying by 2 we will get 3:2, which is a plausible result.

Keep in mind that the whole-number ratio has to make sense for the particular chemical reaction. Since we know that silver always has the charge of +1, combinations like 2:1 or 3:1 are possible. However, combinations like 3:2 are not feasible, since that would assume that a +3 charge is compensated by two anions, which is impossible.

### 1. Why isn't the conductivity zero when the solutions are combined with the stoichiometric amounts?

The conductivity is not zero because not all of the ions are removed by the precipitation reaction. For example, the combination of  $\text{NaCl}$  and  $\text{AgNO}_3$  solution removes the  $\text{Ag}^+$  and  $\text{Cl}^-$  ions in the form of  $\text{AgCl}$ , but the  $\text{Na}^+$  and  $\text{NO}_3^-$  ions remain in solution.

**2. Why is it that we can use volumes of solutions with the same concentration instead of the number of moles to determine the stoichiometric ratio?**

The number of moles of a solute is proportional to the volume of the solution. Therefore, the ratio of the number of moles combined is the same as the ratio of the combined volumes of solutions having the same concentration.

**Lab Preparation**

These are the materials and equipment to set up prior to the lab:

- 1. 0.01 M  $K_2CrO_4$ :** Dissolve 3.884 g of  $K_2CrO_4$  in some water and dilute it to the mark in a 2-L volumetric flask.
- 2. 0.01 M  $AgNO_3$ :** Dissolve 3.398 g of  $AgNO_3$  in some water and dilute it to the mark in a 2-L volumetric flask.

**CAUTION:** Be aware of the potential toxicity from breathing or touching chemicals used in this lab. Prepare the room and oversee the activity accordingly.

**Safety**

Follow all standard laboratory procedures.

**Sequencing Challenge**

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

1	2	4	3	5
Label the test tubes. Accurately pipet the specified volumes of $AgNO_3$ solutions into each test tube.	Accurately pipet the specified volume of the unknown solutions into each test tube.	Measure the conductivity of each solution.	Shake the test tubes to mix the contents thoroughly.	Determine the stoichiometric ratio between the stoichiometric coefficients.

## Procedure with Inquiry

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After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "<sup>n</sup>" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

- Start a new experiment on the data collection system. <sup>◆(1.2)</sup>
- Connect the conductivity sensor to the data collection system. <sup>◆(2.1)</sup>

**Note:** The conductivity sensor does not need to be calibrated because the absolute value of the conductivity is not relevant. Only the relative conductivity values among the solution are important.

- Display Conductivity in a digit display. <sup>◆(7.3.1)</sup>
- Label the nine test tubes with numbers, from "1" to "9".

### Collect Data

- Pipet the amount of AgNO<sub>3</sub> solution prescribed in Table 2 into each test tube.
- Use the second pipet to pipet the amount of unknown solution prescribed in Table 2 into each test tube.
- All of the solutions have the same final volume. Why is it important to compare solutions with the same volume?

The same volume is used to avoid variability in concentration of ions arising from the dilution of the reactants. That way, the volume of each reactant is proportional to the established ion concentration in the final solution. Furthermore, considering that  $c = n/V$ , since the volume is the same, concentrations can be used in place of the number of moles in stoichiometric calculations.

- Place a small piece of Parafilm® on the top of the first test tube. Holding it firmly with your thumb, shake the test tube to mix the contents thoroughly.
- Shake the other test tubes to mix the contents thoroughly.
- Why must you mix the contents of the test tubes thoroughly?

The test tubes must be mixed thoroughly to assure that the solution will be homogeneous.

- Allow the mixtures to settle for a minute.

12.  Rinse the conductivity sensor with deionized water into a waste beaker (100-mL beaker).
13.  Set the conductivity sensor to monitor live data without recording. ♦<sup>(6.1)</sup>
14.  Place the conductivity sensor into the first test tube.
15.  Wait until the conductivity reading stabilizes (up to 30 seconds).
16.  Measure the conductivity of the first solution and record it in Table 2 in the Data Analysis section.
17.  Rinse the conductivity sensor with deionized water into a waste beaker.
18.  Measure the conductivity of the remaining solutions. Rinse the sensor thoroughly with deionized water between measurements. Record each conductivity reading in Table 2 in the Data Analysis section.
19.  Why is it important to rinse the sensor thoroughly between measurements?  
  
It is important to rinse the sensor in order to avoid contamination of the solutions.
20.  Clean up according to your teacher's instructions.

## Data Analysis

- Record the measurements from the procedure.

Table 2: Determination of the stoichiometric ratio using conductivity

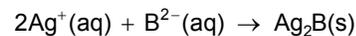
Test Tube	Volume of Ag <sup>+</sup> (mL)	Volume B <sup>n-</sup> (mL)	Volume Ratio Ag <sup>+</sup> :B <sup>n-</sup>	Likely Stoichiometric Ratio	Conductivity (μS)
1	2.00	8.00	1:4	1:4	2173
2	2.50	7.50	1:3	1:3	1943
3	3.00	7.00	1:2.3	1:2	1802
4	4.00	6.00	1:1.5	2:3	1548
5	5.00	5.00	1:1	1:1	1357
6	6.00	4.00	1.5:1	3:2	1074
7	7.00	3.00	2.33:1	2:1	972
8	7.50	2.50	3:1	3:1	981
9	8.00	2.00	4:1	4:1	987

## Analysis Questions

- 1.** Which ratio of solutions provided the lowest conductivity?

2:1

- 2.** What is the balanced equation between Ag<sup>+</sup> and B<sup>n-</sup>? What is the value of *n*? Explain.

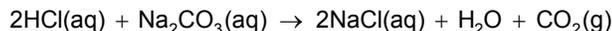


The value of *n* is 2. Since two Ag<sup>+</sup> ions react with one B<sup>n-</sup> ion to form a neutral precipitate, B has to have two negative charges.

## Synthesis Questions

Use available resources to help you answer the following questions.

**1. Can the stoichiometry of a gas-forming reaction be determined with the method applied in this experiment? Explain, considering the following example:**

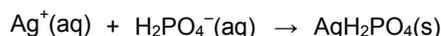
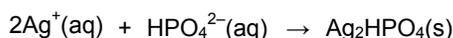
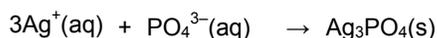


Yes, as long as the gas can be quantitatively removed from the solution. It is not relevant if the ions are removed as a precipitate or as a gas. The important thing is that ions are removed.

**2. Assume you are given three unlabeled solutions with the same concentration: 0.001 M. The solutions are  $\text{Na}_3\text{PO}_4$ ,  $\text{Na}_2\text{HPO}_4$ , and  $\text{NaH}_2\text{PO}_4$ . (In an aqueous medium only the sodium ions dissociate in these compounds, and they are replaced with  $\text{Ag}^+$  to form a precipitate.) Using a 0.001M  $\text{AgNO}_3$  solution, propose a strategy to identify the solutions. Provide the corresponding equations.**

Each of the compounds would bind as many  $\text{Ag}^+$  ions as it does  $\text{Na}^+$  ions. Therefore, each solution would have to be combined with 0.001 M  $\text{AgNO}_3$  in 3:1, 2:1, and 1:1 ratios. Among the solutions that were made with the 3:1 ratio, the one with the lowest conductivity is  $\text{Na}_3\text{PO}_4$ . Among the solutions that were made with the 2:1 ratio, the one with lowest conductivity is  $\text{Na}_2\text{HPO}_4$ . Among the solutions that were made with the 1:1 ratio, the one with lowest conductivity is  $\text{NaH}_2\text{PO}_4$ .

The corresponding ionic equations are



## Multiple Choice Questions

Select the best answer or completion to each of the questions or incomplete statements below.

**1. What would you conclude regarding the stoichiometric coefficients if the lowest conductivity obtained was with the 2.33:1 ratio?**

- A.** Since volumes are proportional to the number of moles, the  $\text{Ag}^+$  would react with the unknown anion in the 2.33:1 ratio.
- B.** Stoichiometric coefficients have to be integers. The closest ratio of integers is most likely the proper stoichiometric ratio, 2:1.
- C.** In order to obtain a ratio of integers, we would have to multiply the numbers by 3, and the proper ratio therefore is 7:3 between  $\text{Ag}^+$  and the unknown  $\text{B}^{n-}$  anion.
- D.** The experiment would have to be repeated.

- 2. Why is it necessary to use deionized water in this experiment?**
- A.** We always use deionized water in experiments just to be on the safe side.
  - B.** Tap water would have been equally acceptable as it dissolves the compounds that we worked with.
  - C. Tap water has ions at a high concentration and that would have falsified our data.**
  - D.** The solubility of  $\text{Ag}^+$  ions is better in deionized water.
- 3. Presume in the conductivity measurements that one data point is much higher than expected, and upon measuring that solution again, it is much lower than expected. What is the *most likely* reason for this discrepancy?**
- A. The solutions were not mixed sufficiently.**
  - B.** The sensor was not rinsed thoroughly and it was contaminated.
  - C.** The test tube was contaminated.
  - D.** The pipet is not accurate enough to measure the desired volume.

### **Extended Inquiry Suggestions**

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Conductivity is the lowest when the solutions of two compounds that form a precipitate are combined in a stoichiometric ratio. This fact can be utilized in quantitative analysis as well. A sample of one of the components with unknown concentration can be titrated with the solution of the other compound with known concentration. This method is called gravimetric titration. To challenge your students already familiar with titration, ask them how the method used in this activity can also be used to detect the end point of a titration.

## Lab 10: Determine the Equilibrium Constant for a Chemical Reaction

### Objectives

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Students determine the equilibrium constant for a chemical reaction using visible spectroscopy.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Preparing solutions
- ◆ Using visible spectroscopy to determine the concentration of an absorbing species
- ◆ Calculating the equilibrium using Beer's law, known initial concentrations, and the mathematical relationship between the concentrations of the reactants and products

### Time Requirement

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◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

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#### *For each student or group:*

- |                              |  |
|------------------------------|--|
| ◆ Data collection system     | ◆ Rubber pipet bulb  |
| ◆ Colorimeter and cuvette    | ◆ 0.01 M Iron ( $\text{Fe}^{3+}$ ), 20 mL <sup>1</sup>       |
| ◆ Extension cable            | ◆ 0.00300 M Potassium thiocyanate (KSCN), 20 mL <sup>2</sup> |
| ◆ Beaker (2), 50-mL          | ◆ Kimwipes®  |
| ◆ Test tube (5), 15-mL       | ◆ Deionized water, 40 mL                                     |
| ◆ Test tube rack             | ◆ Marker   |
| ◆ Graduated pipet (2), 10-mL |  |

<sup>1-2</sup>To prepare the solutions, refer to the Lab Preparation section.

## **Concepts Students Should Already Know**

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Students should be familiar with the following concepts:

- ◆ The concept of equilibrium
- ◆ Stoichiometry of a chemical reaction
- ◆ Reversible chemical reactions
- ◆ Le Chatelier's Principle
- ◆ Beer's Law

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 17a: Absorption Spectra
- ◆ Lab 17b: Colorimetric Analysis
- ◆ Lab 23: Determination of a Solubility Product

## **Using Your Data Collection System**

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

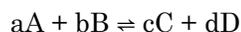
- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting sensors to the data collection system ◆<sup>(2.1)</sup>
- ◆ Calibrating the colorimeter ◆<sup>(3.2)</sup>
- ◆ Using the colorimeter to collect data with blue light ◆<sup>(4.1)</sup>
- ◆ Monitoring live data without recording ◆<sup>(6.1)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Saving your experiment ◆<sup>(11.2)</sup>

## Background

In many chemical reactions the products of the reaction can react to form the original reactants. The forward reaction occurs rapidly at first, but slows as the reactants are consumed. The reverse reaction occurs slowly at first and increases in rate as more products of the forward reaction become available. At some point the two reactions occur at the same rate, resulting in a constant amount of reactants and products. The state in which the concentrations of the reactants and products have no net change over time is known as chemical equilibrium.

The mathematical relationship between the concentrations of the reactants and products is given by the law of mass action, which states that the rate of a chemical reaction is proportional to the concentration of the reactants.

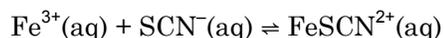
In general, for a reaction of the form,



the equilibrium constant  $K_{\text{eq}}$  is given by

$$K_{\text{eq}} = \frac{[C]^c [D]^d}{[A]^a [B]^b}$$

In this experiment, you use a colorimeter to help determine the equilibrium constant for the formation of  $\text{FeSCN}^{2+}$ . In dilute solutions, iron (III) nitrate,  $\text{Fe}(\text{NO}_3)_3(\text{aq})$ , and potassium thiocyanate,  $\text{KSCN}(\text{aq})$ , are completely dissociated. When these two solutions are mixed, the following equilibrium is established:



## Pre-Lab Activity

### Setting the stage for the activity

Of the five ions in solution,  $\text{K}^{+}(\text{aq})$ ,  $\text{NO}_3^{-}(\text{aq})$ , and  $\text{SCN}^{-}(\text{aq})$  are colorless,  $\text{Fe}^{3+}(\text{aq})$  is nearly colorless, and  $\text{FeSCN}^{2+}(\text{aq})$  is deep red. Changes in the concentration of  $\text{FeSCN}^{2+}(\text{aq})$  are indicated by changes in the intensity of the color of the solution.

The equilibrium expression for the reaction is

$$K_{\text{eq}} = \frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}][\text{SCN}^{-}]}$$

To calculate  $K_{\text{eq}}$  for this reaction, you need to determine the molar concentration of  $\text{Fe}^{3+}$ ,  $\text{SCN}^{-}$ , and  $\text{FeSCN}^{2+}$  at equilibrium.

The relationship between electromagnetic absorption and concentration of the absorbing species is given by Beer's law. Absorption of light is directly proportional to the distance that the light travels through an absorbing medium and the molar concentration of the absorbing species.

$$A = \epsilon \times l \times c \tag{1}$$

## Lab 10: Determine the Equilibrium Constant for a Chemical Reaction

---

where

$A$  = absorption

$\epsilon$  = absorptivity coefficient ( $\text{M}^{-1}\text{cm}^{-1}$ )

$l$  = path length that light travels through the solution (cm)

$c$  = molar concentration of the absorbing species (M).

**Note:** The AP exam version of Equation 1 is written as “ $A = a b c$ ” where  $a$  and  $b$  correspond to  $\epsilon$  and  $l$ , respectively.

The absorptivity coefficient  $\epsilon$  is a proportionality constant, and its value depends both on the nature of the absorbing species and on the wavelength of light chosen for the measurement. At a given wavelength and using a sample cell of constant path length, absorption is directly proportional to concentration. Thus, a measurement of  $A$  can be used to determine concentration. The red  $\text{FeSCN}^{2+}$  ion absorbs blue light and will be analyzed at 468 nm.

### Example calculation to try

In an experiment to determine the equilibrium constant for the reaction between  $\text{Fe}^{3+}$  and  $\text{SCN}^-$ , a student prepared a solution by mixing 1.00 mL of 0.0100 M  $\text{Fe}^{3+}$ , 1.00 mL of 0.00300 M  $\text{SCN}^-$  solution, and 8.00 mL of water. A portion of the solution was placed in a cell and the absorbance of the solution was measured. The cell path length  $l$  is 1.00 cm thick. The absorptivity coefficient  $\epsilon$  is  $5302 \text{ M}^{-1}\text{cm}^{-1}$  for  $\text{FeSCN}^{2+}$ .

The concentration of  $\text{Fe}^{3+}$  and  $\text{SCN}^-$  in the reaction solution before the reaction between  $\text{Fe}^{3+}$  and  $\text{SCN}^-$  occurs can be calculated by considering the dilution of the reactants.

$$c_i V_i = c_f V_f$$

where

$c_i$  = initial concentration (M)

$V_i$  = initial volume (mL)

$c_f$  = final concentration (M)

$V_f$  = final volume after the dilution (mL)

Using this equation, the concentration of  $\text{Fe}^{3+}$  in the reaction solution before the reaction occurs is  $[\text{Fe}^{3+}]_0(1.00\text{mL} + 1.00\text{mL} + 8.00\text{mL}) = (0.0100\text{M})(1.00\text{mL})$

The calculated  $[\text{Fe}^{3+}]_0$  is the concentration of iron(III) *after* the dilution (after mixing the reactants) but *before* the reaction starts.

Solving for  $[\text{Fe}^{3+}]_0$  and doing the same to calculate the initial concentration of  $\text{SCN}^-$  results in the following:

$$[\text{Fe}^{3+}]_0 = \frac{1.00 \text{ mL}}{1.00 \text{ mL} + 1.00 \text{ mL} + 8.00 \text{ mL}}(0.0100 \text{ M}) = 1.00 \times 10^{-3} \text{ M}$$

$$[\text{SCN}^-]_0(1.00 \text{ mL} + 1.00 \text{ mL} + 8.00 \text{ mL}) = (0.00300 \text{ M})(1.00 \text{ mL})$$

$$[\text{SCN}^-]_0 = \frac{1.00 \text{ mL}}{1.00 \text{ mL} + 1.00 \text{ mL} + 8.00 \text{ mL}}(0.00300 \text{ M}) = 3.00 \times 10^{-4} \text{ M}$$

To calculate the equilibrium constant, you need to calculate the equilibrium concentrations. The concentration of  $\text{FeSCN}^{2+}$  at equilibrium is determined using the absorbance of the solution:

$$[\text{FeSCN}^{2+}] = \frac{A}{\epsilon l} = \frac{0.293}{(5203 \text{ M}^{-1} \text{ cm}^{-1})(1.00 \text{ cm})} = 5.63 \times 10^{-5} \text{ M}$$

The equilibrium concentrations of  $\text{Fe}^{3+}$  and  $\text{SCN}^-$  are then the difference between their initial concentrations and the equilibrium concentration determined for  $\text{FeSCN}^{2+}$ .

$$[\text{Fe}^{3+}] = [\text{Fe}^{3+}]_0 - [\text{FeSCN}^{2+}] = (1.00 \times 10^{-3} \text{ M}) - (5.63 \times 10^{-5} \text{ M}) = 9.44 \times 10^{-4} \text{ M}$$

$$[\text{SCN}^-] = [\text{SCN}^-]_0 - [\text{FeSCN}^{2+}] = (3.00 \times 10^{-4} \text{ M}) - (5.63 \times 10^{-5} \text{ M}) = 2.44 \times 10^{-4} \text{ M}$$

Using the calculated concentrations, the equilibrium constant is:

$$K_{\text{eq}} = \frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}][\text{SCN}^-]} = \frac{(5.63 \times 10^{-5})}{(9.44 \times 10^{-4})(2.44 \times 10^{-4})} = 244$$

**1. What kind of mathematical relationship exists between the absorbance  $A$  and the concentration of the absorbing species  $c$ ?**

The relationship is linear. That is, the absorbance is directly proportional to the concentration.

**2. Looking at the equation (Equation 1) that describes the relationship between absorbance and the concentration, what would be the intercept on the plot of  $A$  versus  $c$ ? Explain!**

The intercept would be 0. This means that when the concentration is 0, so is the absorbance. This makes perfect sense: If there are no species absorbing light, the absorbance should be zero.

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

- 0.0100 M Iron solution:** In a 500-mL volumetric flask, dissolve 2.02 g of  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  in some distilled water, add 20 mL of concentrated  $\text{HNO}_3$  and fill the flask to the mark with distilled water.
- 0.00300 M KSCN:** In a 500-mL volumetric flask, dissolve 0.1383 g of KSCN in some distilled water and fill the flask to the mark with distilled water.

## Safety

---

Follow all standard laboratory procedures.

## Sequencing Challenge

---

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

4	2	1	3	5
Calculate the concentration of each species in each solution.	Calibrate the colorimeter with the prepared "blank" solution.	Set up the data collection system and colorimeter. Then, in 5 test tubes, combine the prescribed volumes of solutions.	Measure and record the absorbance of the 4 solutions.	Calculate the equilibrium constant for each solution and calculate the average value as well.

## Procedure with Inquiry

---

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "♦" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

- Start a new experiment on the data collection system. ♦<sup>(1,2)</sup>
- Connect the colorimeter to the data collection system using a sensor extension cable. ♦<sup>(2,1)</sup>
- Set the data collection system to monitor live data without recording. ♦<sup>(6,1)</sup>
- Label the first test tube "Blank" and the others from "1" to "4".
- Prepare the solutions in the labeled test tubes by combining the prescribed amounts of the reactants and water (see Table 1).

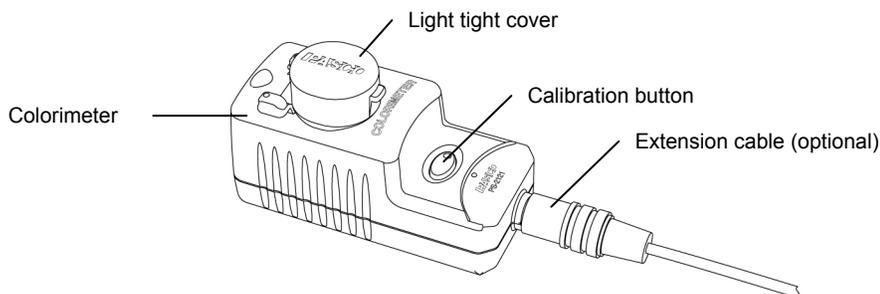
Table 1: Composition of solutions to study chemical equilibrium

Test Tube	$1.00 \times 10^{-2}$ M $\text{Fe}_2(\text{NO}_3)_3$ , acidic (mL)	$3.00 \times 10^{-3}$ M KSCN (mL)	Water (mL)	A
Blank	1.00	0.00	9.00	
1	1.00	1.00	8.00	0.360
2	1.00	2.00	7.00	0.764
3	1.00	3.00	6.00	1.144
4	1.00	4.00	5.00	1.475

6.  How do you think the intensity of the color changes among the solutions? Explain your prediction.

The intensity of the color increases with the concentration of the colorful species, KSCN. Therefore test tube #4 will have the most intense color.

7.  Calibrate the colorimeter with the blank solution.  $\diamond^{(3.2)}$



**Important:** Always make sure that the cell is clean and dry on the outside before placing it into the colorimeter.

8.  Throughout this activity, collect data using blue light (468 nm) in the colorimeter.  $\diamond^{(4.1)}$

### Collect Data

9.  Measure the absorbance of the four solutions following the steps below.
- Rinse the cell with a small portion of the first solution and fill the cuvette two-thirds full. Wipe the cuvette clean and dry and place it into the colorimeter.
  - Why do you have to rinse the cell with some of the solution?

If there is any residual water in the cuvette, it will dilute the concentration of the solution and falsify the data.

## Lab 10: Determine the Equilibrium Constant for a Chemical Reaction

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- c. After the reading stabilizes, record the absorbance in Table 1 and Table 2.
- d. Dispose of the solution and rinse the cell thoroughly with water.
- e. Why do you think it is important to rinse the cell thoroughly between measurements?

You need to rinse the cell to avoid contamination of the solutions.

10.  Clean up according to your teacher's instructions. ♦<sup>(11.1)</sup>

### Data Analysis

---

For your calculations, consider the product of absorptivity  $\epsilon$  and the cell thickness  $l$  to be

$$\epsilon \times l = 5900 \text{ M}^{-1}$$

1.  Calculate and record in Table 2 the initial concentrations of  $\text{Fe}^{3+}$  and  $\text{SCN}^-$  in each test tube.

For Test Tubes 1 to 4 for  $[\text{Fe}^{3+}]_0$  and for Test Tube 1 for  $[\text{SCN}^-]_0$ :

$$[\text{Fe}^{3+}]_0 (1.00 \text{ mL} + 1.00 \text{ mL} + 8.00 \text{ mL}) = (0.0100 \text{ M})(1.00 \text{ mL})$$

$$[\text{Fe}^{3+}]_0 = \frac{1.00 \text{ mL}}{1.00 \text{ mL} + 1.00 \text{ mL} + 8.00 \text{ mL}} (0.0100 \text{ M}) = 1.00 \times 10^{-3} \text{ M}$$

$$[\text{SCN}^-]_0 (1.00 \text{ mL} + 1.00 \text{ mL} + 8.00 \text{ mL}) = (0.00300 \text{ M})(1.00 \text{ mL})$$

$$[\text{SCN}^-]_0 = \frac{1.00 \text{ mL}}{1.00 \text{ mL} + 1.00 \text{ mL} + 8.00 \text{ mL}} (0.00300 \text{ M}) = 3.00 \times 10^{-4} \text{ M}$$

2.  Calculate the concentration of the  $\text{Fe}(\text{SCN})^{2+}$  ions from the absorbance measurements using Beer's Law and the above value of  $\epsilon \times l$ . Record the values in Table 2.

The concentration of  $\text{Fe}(\text{SCN})^{2+}$  in the Test Tube 1:

$$[\text{FeSCN}^{2+}] = \frac{A}{\epsilon l} = \frac{0.360}{5900 \text{ M}^{-1}} = 6.10 \times 10^{-5} \text{ M}$$

3.  Calculate the equilibrium concentration of the  $\text{Fe}^{3+}$  and  $\text{SCN}^-$  ions from the initial concentration of the ions and the amount of ions used to establish the equilibrium concentration of  $\text{FeSCN}^{2+}$ . Record the values in Table 2.

The concentration of  $\text{Fe}^{3+}$  and  $\text{SCN}^-$  ions in Test Tube 1:

$$[\text{Fe}^{3+}] = [\text{Fe}^{3+}]_0 - [\text{FeSCN}^{2+}] = (1.00 \times 10^{-3} \text{ M}) - (6.10 \times 10^{-5} \text{ M}) = 9.39 \times 10^{-4} \text{ M}$$

$$[\text{SCN}^-] = [\text{SCN}^-]_0 - [\text{FeSCN}^{2+}] = (3.00 \times 10^{-4} \text{ M}) - (6.10 \times 10^{-5} \text{ M}) = 2.39 \times 10^{-4} \text{ M}$$

4.  Calculate the equilibrium constant from the equilibrium concentration of the  $\text{Fe}(\text{SCN})^{2+}$ ,  $\text{Fe}^{3+}$ , and  $\text{SCN}^-$  ions. Record the values in Table 2.

Performing the calculation for the first solution:

$$K_{\text{eq}} = \frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}][\text{SCN}^-]} = \frac{(6.10 \times 10^{-5})}{(9.39 \times 10^{-4})(2.39 \times 10^{-4})} = 271$$

Table 2: Calculation of the equilibrium concentrations and the equilibrium constant

#	$[\text{Fe}^{3+}]_0$ (M)	$[\text{SCN}^-]_0$ (M)	A	$[\text{FeSCN}^{2+}]$ (M)	$[\text{Fe}^{3+}]$ (M)	$[\text{SCN}^-]$ (M)	$K_{\text{eq}}$
1	$1.00 \times 10^{-3}$	$3.00 \times 10^{-4}$	0.360	$6.10 \times 10^{-5}$	$9.39 \times 10^{-4}$	$2.39 \times 10^{-4}$	271
2	$1.00 \times 10^{-3}$	$6.00 \times 10^{-4}$	0.764	$1.29 \times 10^{-4}$	$8.71 \times 10^{-4}$	$4.71 \times 10^{-4}$	316
3	$1.00 \times 10^{-3}$	$9.00 \times 10^{-4}$	1.144	$1.93 \times 10^{-4}$	$8.06 \times 10^{-4}$	$7.06 \times 10^{-4}$	341
4	$1.00 \times 10^{-3}$	$1.20 \times 10^{-3}$	1.475	$2.500 \times 10^{-4}$	$7.50 \times 10^{-4}$	$9.50 \times 10^{-4}$	351

5.  Calculate the average equilibrium constant and record the value below.

Average value of  $K_{\text{eq}}$ : 320

## Analysis Questions

1. How did the absorbance change with increasing initial  $\text{SCN}^-$  concentration while the initial concentration of  $\text{Fe}^{3+}$  was kept constant? Why?

The absorbance increased because higher  $\text{SCN}^-$  concentration pushes the equilibrium of Equation 1 to the right, which results in more  $\text{FeSCN}^-$  ions.

2. Why do you think the equilibrium constant remained virtually constant, within experimental error, even though you were changing the concentrations?

The equilibrium constant does not depend on the concentrations. On the contrary, concentrations adjust to satisfy the equilibrium constant by shifting the concentration of reactants and products in the appropriate direction.

3. How do you think your results would have been different if you used a cell with twice the path length?

The absorbance values would have been doubled. However, we would have obtained the same concentrations.

4. Beer's Law (the linear relationship between concentration and absorbance) is accurate to about  $A = 1.5$ . How would you modify the experiment if the absorbance readings were higher than 1.5?

The solutions would have to be diluted until the absorbance was within the acceptable range.

## Synthesis Questions

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Use available resources to help you answer the following questions.

- 1. Does the absorbance you measured come only from the  $\text{FeSCN}^{2+}$  ion? Explain your answer.**

Only the  $\text{FeSCN}^{2+}$  ion absorbs appreciably at this wavelength. This is also proven by the fact that only the  $\text{FeSCN}^{2+}$  ion is colored.

- 2. The  $\text{Fe}^{3+}$  ion can react with three  $\text{SCN}^-$  ions according to the following equilibrium equations to form  $\text{Fe}(\text{SCN})_2^+$  and  $\text{Fe}(\text{SCN})_3$ . These products are also red.**



**In light of these reactions, propose an explanation as to why this experiment uses a large excess of  $\text{Fe}^{3+}$  ions.**

The experiment used a large excess of  $\text{Fe}^{3+}$  to suppress the products resulting from Equations SQ2 and SQ3, which require  $\text{SCN}^-$  ions. This ensures that the measured absorbance is completely attributed to  $\text{Fe}(\text{SCN})^{2+}$ : according to Le Chatelier's Principle, the first reaction is shifted to the right, removing most of the  $\text{SCN}^-$  ions; the second and third reactions are shifted to the left to increase the concentration of  $\text{SCN}^-$  ions.

## Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

- 1. The four measurements you made resulted in four substantially different equilibrium constants. Which statement could be correct?**
- A.** The equilibrium constant is determined by the concentrations. We used different concentrations, so the resulting equilibrium constants should be the same.
  - B.** The equilibrium of the second and third equations (SQ2 and SQ3) interfered with our measurements.
  - C.** The equilibrium constant depends on the temperature, so the fluctuation in room temperature might have interfered with our measurements.
  - D.** There was an error in mixing the solutions, or one (or both) of the stock solutions had the wrong concentration.

**2. How do you think doubling the initial iron concentration of  $\text{Fe}^{3+}$  ions would affect the obtained value for  $K_{\text{eq}}$ ?**

- A.** Doubling the initial concentration of  $\text{Fe}^{3+}$  ions will double  $K_{\text{eq}}$ , according to Le Chatelier's Principle.
- B.** Doubling the initial concentration of  $\text{Fe}^{3+}$  ions will not affect  $K_{\text{eq}}$ .
- C.** Doubling the initial concentration of  $\text{Fe}^{3+}$  ions will result in half of the value for  $K_{\text{eq}}$ .
- D.** The effect of doubling the  $\text{Fe}^{3+}$  initial concentration is not predictable; you would need to actually perform the experiments.

**3. The  $\text{FeSCN}^{2+}$  ion is red, the other species are practically colorless. Would it interfere with your measurements if another species had color?**

- A.** No, we measure only  $\text{FeSCN}^{2+}$ .
- B.** If the other colored species does not absorb at 468 nm where we perform the experiment, it would not interfere with our measurements.
- C.** Yes, but we would need to divide the measured absorbance by 2 to get the absorbance from  $\text{FeSCN}^{2+}$ .
- D.** The answer depends on which other species absorbs.

### Extended Inquiry Suggestions

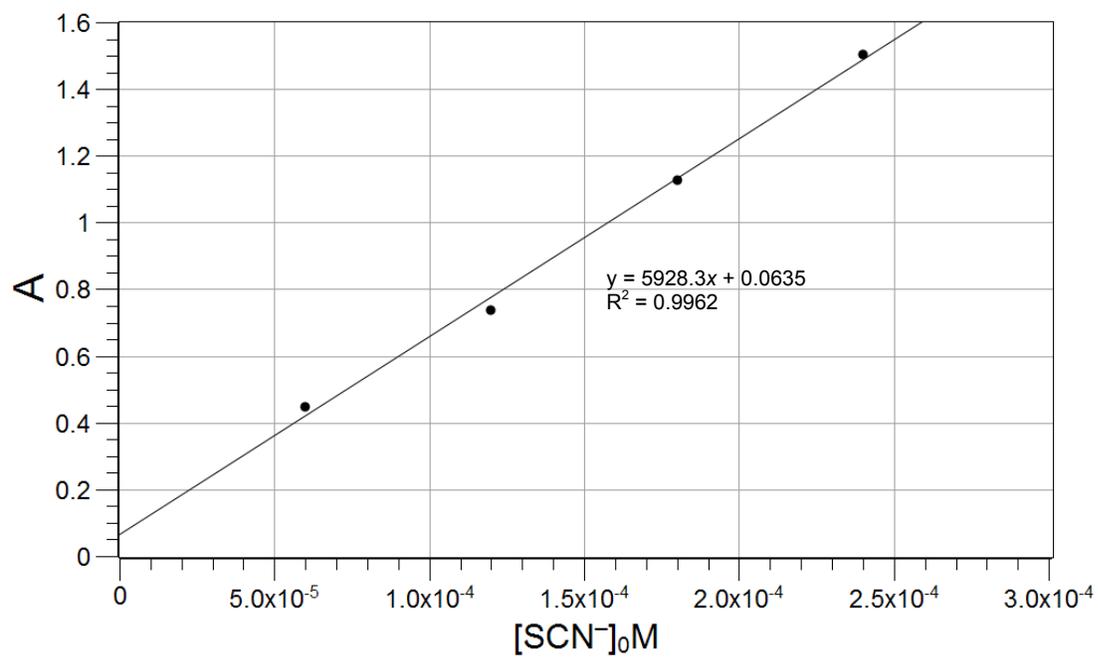
If time allows, ask your students to determine the product of absorptivity of the species and cell path length instead of giving them for the calculations. Please note that if the colorimeter is used, the path length is not well defined because the cell is round and not all of the photons pass through the same thickness of solution. Therefore, it is more appropriate to consider the product of absorptivity  $\epsilon$  and cell path length  $l$  as one constant that relates the absorbance to the concentration.

The product of  $\epsilon \times l$  can be determined by preparing 4 solutions with  $\text{Fe}^{3+}$  ions in overwhelming excess. In this case, it is safe to assume that all  $\text{Fe}^{3+}$  ions are quantitatively converted to  $\text{FeSCN}^{2+}$  (see the table below).

Absorptivity of the calibrating solutions

0.003 M KSCN (mL)	0.1 M $\text{Fe}^{3+}$ (mL)	Water (mL)	$[\text{FeSCN}^{2+}]$ (M)	$A$
0.50	20.00	4.50	$6.00 \times 10^{-5}$	0.446
1.00	20.00	4.00	$1.20 \times 10^{-4}$	0.737
1.50	20.00	3.50	$1.80 \times 10^{-4}$	1.126
2.00	20.00	3.00	$2.40 \times 10^{-4}$	1.502

Lab 10: Determine the Equilibrium Constant for a Chemical Reaction



The product of absorptivity  $\epsilon$  and path length  $l$  is  $5.93 \times 10^3 \text{ M}^{-1} \text{ cm}^{-1}$ .

## Lab 11: Using Different Indicators for pH Determination

### Objectives

Students determine the CO<sub>2</sub> content of a beverage.

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Performing titrations with multiple acid-base indicators.
- ◆ Comparing the use of pH indicators with electronic sensors for detecting the equivalence point of a titration

### Time Requirement

◆ Preparation time	30 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	90 minutes plus overnight sample preparation

### Materials and Equipment

#### *For each student or group:*

- |                                   |  |
|-----------------------------------|--|
| ◆ Data collection system          | ◆ Magnetic stirrer and stir bar                    |
| ◆ Drop counter and micro stir bar | ◆ Ring stand                                       |
| ◆ pH sensor                       | ◆ Commercial soda drink, 1 can                     |
| ◆ Clamp, right-angle              | ◆ Kimwipes <sup>®1</sup>                           |
| ◆ Clamp, buret                    | ◆ 4.00 M HCl solution, 100 mL <sup>1</sup>         |
| ◆ Buret, 50-mL                    | ◆ 1 M NaOH solution, 100 mL <sup>2</sup>           |
| ◆ Beaker (2), 25-mL               | ◆ Wash bottle with deionized water                 |
| ◆ Beaker (2), 250-mL              | ◆ Funnel   |
| ◆ Erlenmeyer flask, 250-mL        | ◆ Balloon (fits on Erlenmeyer flask; holds 100 mL) |
| ◆ Graduated cylinder, 100-mL      | ◆ Buffers, pH 4 and pH 10, 10 mL                   |
| ◆ Phenolphthalein, 5 drops        | ◆ Cotton swab or tissue                            |
| ◆ Methyl orange, 5 drops          |  |

<sup>1-2</sup>To prepare the solutions, refer to the Lab Preparation section.

## **Concepts Students Should Already Know**

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Students should be familiar with the following concepts:

- ◆ Titration
- ◆ Acid-base reactions
- ◆ Stoichiometry of chemical reactions
- ◆ Chemical equilibrium

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 6: Standardizing a Solution of Sodium Hydroxide
- ◆ Lab 7: Acid–Base Titration
- ◆ Lab 8: Oxidation–Reduction Titration
- ◆ Lab 19: Properties of Buffer Solutions
- ◆ Lab 23: Determination of a Solubility Product
- ◆ Lab 24: Determining  $K_a$  by Half-Titration of a Weak Acid

## **Using Your Data Collection System**

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a sensor to the data collection system. ◆<sup>(2.1)</sup>
- ◆ Connecting multiple sensors to the data collection system ◆<sup>(2.2)</sup>
- ◆ Calibrating a drop counter ◆<sup>(3.4)</sup>
- ◆ Calibrating a pH sensor ◆<sup>(3.6)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>
- ◆ Changing the variable on the x-axis and y-axis of a graph ◆<sup>(7.1.9)</sup>

- ◆ Identifying data points on a graph ◆<sup>(9.1)</sup>
- ◆ Finding the slope at a point on the data plot ◆<sup>(9.3)</sup>
- ◆ Saving your experiment ◆<sup>(11.1)</sup>
- ◆ Printing the graph. ◆<sup>(11.2)</sup>

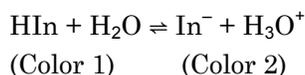
## Background

Certain organic substances have a property which allows them to change color in dilute solutions when the hydrogen ion concentration of the solution attains a specific value. Substances such as phenolphthalein, which is colorless in an acid solution but becomes pink or purple in a basic solution, are called acid-base indicators. (The pH of the color change depends on the dissociation constant of the indicator, which may not be 7.) They are often used for determining the pH of solutions or to determine the equivalence point of a titration.

The most common indicators, in addition to phenolphthalein, are litmus, methyl orange, and methyl red. Litmus is a naturally occurring organic dye which turns red in an acid and blue in a base. Methyl orange solution turns yellow in basic solutions and red in acidic solutions.

These indicators should be used in colorless solutions, otherwise the color of the solution may mask the color changes of the indicator.

The indicator (In) comes to equilibrium in aqueous solutions:



Therefore, indicators behave like weak acids with a specific  $\text{p}K_{\text{a}}$  value. The equilibrium is shifted as the pH of the solution changes. Knowing the pH at which an indicator will change color is useful for determining the equivalence point of an acid-base titration. However, the pH of the equivalence point depends on the  $\text{p}K_{\text{a}}$  value of the acid, which is different for each acid. Therefore, the indicator has to be chosen to match the  $\text{p}K_{\text{a}}$  value of the acid. The most common indicators are listed in Table 1 with the pH range in which they change color and the color change.

## Lab 11: Using Different Indicators for pH Determination

Table 1: pH indicators

Indicator	pH range	Colors
Crystal violet	0.0 – 1.6	yellow → blue
Thymol blue	1.2 – 2.8	red → yellow
Orange IV	1.4 – 2.8	red → yellow
Methyl orange	3.2 – 4.4	red → yellow
Bromocresol green	3.8 – 5.4	yellow → blue
Methyl red	4.8 – 6.2	red → yellow
Chlorophenol red	5.2 – 6.8	yellow → red
Bromothymol blue	6.0 – 7.6	yellow → blue
Phenol red	6.6 – 8.0	yellow → red
Neutral red	6.8 – 8.0	red → amber
Thymol blue	8.0 – 9.6	yellow → blue
Phenolphthalein	8.2 – 10.0	colorless → pink
Thymolphthalein	9.4 – 10.6	colorless → blue
Alizarin yellow	10.1 – 12.0	yellow → blue
Indigo carmine	11.4 – 13.0	blue → yellow

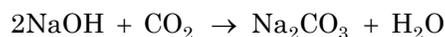
You will be using phenolphthalein and methyl orange indicators in this experiment to continuously monitor the pH of a titrated solution.

In this activity, indicators will be used to show the end point of titrations to determine the CO<sub>2</sub> content of a commercial beverage.

### Pre-Lab Activity

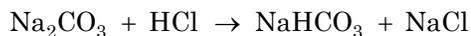
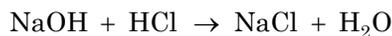
#### Setting the stage for the activity

Many commercial products contain dissolved carbon dioxide (CO<sub>2</sub>) or some type of carbonate. In this experimental procedure, CO<sub>2</sub> gas produced by a chemical reaction is transferred into a balloon containing excess sodium hydroxide (NaOH) solution. The CO<sub>2</sub> is absorbed and converted into an equivalent amount of sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>):

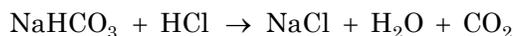


The resulting mixture, consisting of sodium carbonate and excess sodium hydroxide, will be titrated with a standardized hydrochloric acid solution using phenolphthalein and methyl orange

as indicators. Titration to the first end point, when phenolphthalein becomes colorless, neutralizes the excess sodium hydroxide and converts all the sodium carbonate to sodium bicarbonate ( $\text{NaHCO}_3$ ):



Continued titration to the methyl orange end point converts the sodium bicarbonate to sodium chloride ( $\text{NaCl}$ ),  $\text{CO}_2$  and water:



Therefore, the number of moles of the titrant necessary for the second step will be equivalent to the number of moles of  $\text{CO}_2$  absorbed.

### **Example calculation to try**

The content of a can of commercial soda was transferred into a 500-mL Erlenmeyer flask with a magnetic stirring bar already inside. A balloon containing 100 mL of 1.0 M NaOH solution was secured onto the neck of the flask. The flask was then placed on a magnetic stirrer and stirred overnight. Initially the balloon expanded but eventually collapsed, indicating that the  $\text{CO}_2$  had been removed from the beverage and absorbed by the NaOH solution.

The solution from the balloon was then transferred into a 250-mL beaker. A few drops of phenolphthalein indicator were added to the solution which was then titrated with a 4.00 M HCl solution. It took 16.5 mL of the 4.00 M solution to change the color of the indicator from purple to colorless. From there, a few drops of methyl orange indicator was added and titrated further with 4.00 M HCl. Once the solution changed from orange to red, indicating the equivalence point, it was found that the total HCl consumption was 25.00 mL.

The amount of HCl consumed in the second step was

$$(25.00 \text{ mL} - 16.48 \text{ mL}) \left( \frac{1 \text{ L}}{1000 \text{ mL}} \right) \left( \frac{4.00 \text{ mol}}{1 \text{ L}} \right) = 3.41 \times 10^{-2} \text{ mol HCl}$$

This is equivalent to the number of moles of  $\text{CO}_2$  in the sample, which were:

$$(3.41 \times 10^{-2} \text{ mol CO}_2) \left( \frac{44.01 \text{ g}}{1 \text{ mol}} \right) = 1.50 \text{ g CO}_2$$

The beverage sample had 1.50 g of  $\text{CO}_2$ .

### **1. Why is the number of moles of HCl consumed in the second step equivalent to the number of moles of $\text{CO}_2$ in the sample?**

The second step involves converting  $\text{NaHCO}_3$  to  $\text{NaCl}$  with HCl. In this reaction the HCl and  $\text{NaHCO}_3$  react in a 1:1 ratio. Also the number of moles of  $\text{NaHCO}_3$  is the same as the number of moles of  $\text{CO}_2$ , since each  $\text{CO}_2$  molecule is converted to one  $\text{NaHCO}_3$  molecule.

## Lab 11: Using Different Indicators for pH Determination

**2. How many moles of HCl were necessary to remove the excess amount of NaOH in the first step? (Hint: In the first step, the same number of moles of HCl was used to convert  $\text{Na}_2\text{CO}_3$  to  $\text{NaHCO}_3$  as in the second step to convert  $\text{NaHCO}_3$  to  $\text{CO}_2$  and water.)**

The amount of HCl necessary to convert  $\text{NaHCO}_3$  to  $\text{CO}_2$  is  $3.41 \times 10^{-2}$  mol. It took the same number of moles of HCl to convert  $\text{Na}_2\text{CO}_3$  to  $\text{NaHCO}_3$ . This latter number of moles has to be subtracted from the number of moles of HCl added up to the first equivalence point to get the number of moles of HCl that reacted with the excess amount of NaOH.

$$(16.48 \text{ mL HCl}) \left( \frac{4.00 \text{ mol HCl}}{1000 \text{ mL}} \right) - (3.41 \times 10^{-2} \text{ mol HCl}) = 3.18 \times 10^{-2} \text{ mol HCl}$$

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

- 1. 4.00 M HCl:** Under a hood, slowly add 650 mL of 36% HCl solution to about 1000 mL of water in a 2-L volumetric flask and fill it to the mark.
- 2. 1 M NaOH:** Dissolve 80 g of NaOH in approximately 500 mL of water in a 2-L volumetric flask. Cool the solution under running tap water and fill it to the mark.

## Safety

Add these important safety precautions to your normal laboratory procedures:

- ◆ If the NaOH or HCl solutions come in contact with your skin or eyes, rinse immediately with a large amount of running water.

## Sequencing Challenge

The steps below are parts of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

3	5	1	2	4
Add phenolphthalein to the NaOH solution.  Titrate the NaOH solution with a standardized HCl solution.	Knowing the equivalence points and corresponding titrant volumes, calculate the mass of $\text{CO}_2$ that was in the soda.	Transfer chilled soda into an Erlenmeyer flask. Connect a balloon containing 1 M NaOH to the flask.	Stir the soda overnight so the NaOH reacts with the $\text{CO}_2$ that is generated. Then transfer the solution to a 250 mL beaker.	After the color changes, add methyl orange and continue the titration. Note both points where the color changes.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "◆" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

#### Sample preparation

1.  Chill a can of soda.
2.  When the soda is cold, open the can and use the 100-mL graduated cylinder to slowly transfer 100 mL into a 250-mL Erlenmeyer flask.
3.  Place a stirring bar in the flask.
4.  Why do you think it is important to chill the soda before opening it? (Hint: how does temperature change the solubility of gases in liquids and how does that affect the loss of CO<sub>2</sub> when the can is opened.)

The solubility of CO<sub>2</sub> increases as the soda is cooled so the loss of CO<sub>2</sub> upon opening the can is reduced.

5.  Place 100.00 mL of 1 M NaOH in a balloon and secure the balloon on the mouth of the Erlenmeyer flask.
6.  Set the Erlenmeyer flask on the magnetic stirrer and leave it stirring overnight.

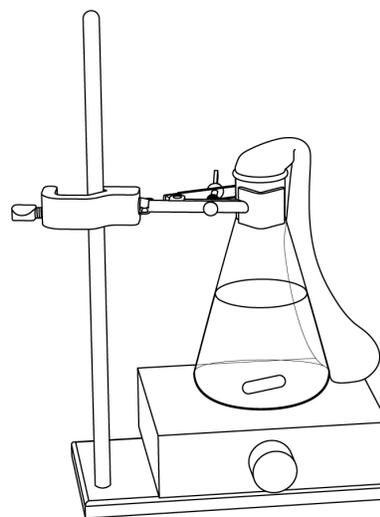
**Note:** Set the balloon next to the Erlenmeyer flask securely.

7.  What do you think will happen overnight?

The CO<sub>2</sub> escapes from the soda and dissolves in the NaOH solution.

#### Titration preparation

8.  Remove the Erlenmeyer flask and the attached balloon from the magnetic stirrer.
9.  Carefully transfer the NaOH solution from the balloon into a 250-mL beaker and add 3 to 5 drops of phenolphthalein indicator solution.
10.  Start a new experiment on the data collection system. ◆<sup>(1,2)</sup>



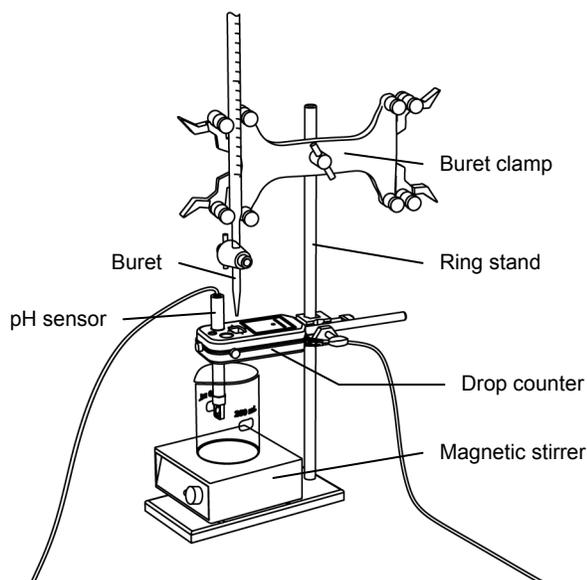
## Lab 11: Using Different Indicators for pH Determination

11. □ Connect a pH sensor to the data collection system. ♦(2.1)

12. □ Calibrate the pH sensor. ♦(3.6)

13. □ Assemble the titration apparatus, using the steps below and the illustration as a guide.

- Position the magnetic stirrer on the base of the ring stand.
- Place a waste container (250-mL beaker) on the magnetic stirrer.
- Use the buret clamp to attach the buret to the ring stand.
- Position the drop counter over the waste container and attach it to the ring stand using the right-angle clamp.
- Place the pH sensor through one of the slots in the drop counter.



**Note:** Do not connect the drop counter to the data collection system yet.

14. □ Rinse the buret with several milliliters of the 4.00 M HCl solution:

- Ensure that the stopcock is closed and rinse the inside of the buret with several milliliters of the standardized HCl solution.
- Open the stopcock on the buret and drain the rinse HCl into the waste container.
- Repeat this process two more times.

15. □ Why is it necessary to rinse the buret with the HCl solution?

If there is any residual water or contaminant in the buret, it will dilute the HCl and change its concentration. Rinsing eliminates any such contamination.

16. □ Make sure the stopcock on the buret is in the “off” position and then use a funnel to fill the buret with about 50 mL of the 4.00 M HCl solution (titrant).

17. □ Drain a small amount of the titrant through the drop counter into the waste beaker to remove any air in the tip of the buret.

18. □ Why is it important to remove air from the tip of the buret?

Any air trapped in the buret tip is counted as volume of HCl. If this happens, the amount of titrant used will be inaccurate.

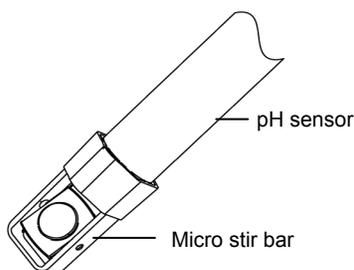
19. □ Practice adjusting the stopcock on the buret so that the titrant goes through the drop counter in distinguishable drops that fall at about 1 to 2 drops per second.

**Note:** Good control of the stopcock is important. Each drop should result in a blink of the LED on the drop counter. If the LED is continuously lit, you have opened the stopcock too far and you will have to start over.

20. □ Why will it be necessary to start your titration over again if you accidentally allow the titrant to stream out of the stopcock instead of emerging by drops?

The drop counter counts distinct drops. If the drops are not sufficiently distinct from one another, the drop counter will not function properly and the fluid volume will not be accurate.

21. □ Add the micro stir bar to the end of the pH sensor.



22. □ Why is it necessary to stir the solution during a titration?

Stirring thoroughly mixes the ions in the solution so that the recorded pH reflects the pH of the entire solution.

23. □ Add additional 4.00 M HCl to the buret so the solution is above the zero mark. Allow some of the HCl solution to drip into the waste container until the bottom of the meniscus is lined up with or just below the zero mark and record the starting volume in Table 2.

24. □ Remove the waste container.

25. □ Set the beaker with the NaOH solution on the magnetic stirrer.

26. □ Turn on the magnetic stirrer at a slow and steady rate.

27. □ Connect the drop counter to the data collection system. ♦<sup>(2.2)</sup>

28. □ Display the pH on the y-axis of a graph and Drop Count on the x-axis. ♦<sup>(7.1.1)</sup>

### Collect Data

29. □ Clean the lens of the drop counter inside the opening through which the drops are going with deionized water and a cotton swab or tissue.

## Lab 11: Using Different Indicators for pH Determination

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- 30.**  Start recording data. ♦<sup>(6.2)</sup>
- 31.**  Turn the buret stopcock carefully, allowing the titrant to drip slowly (1 to 2 drops per second) into the solution.
- 32.**  When the solution turns colorless, add 3 to 5 drops of methyl orange indicator solution and continue the titration
- 33.**  How did the pH change at the equivalence point?  
The pH suddenly dropped at the equivalence point.
- 34.**  What species are in the solution at this time?  
All  $\text{Na}_2\text{CO}_3$  has been converted to  $\text{NaHCO}_3$  and all excess  $\text{NaOH}$  has been converted to  $\text{NaCl}$ .
- 35.**  When the solution turns red, continue the titration past the equivalence point until the pH curve flattens.
- 36.**  How did the pH change at the equivalence point?  
The pH suddenly dropped at the second equivalence point as well.
- 37.**  Why is it important to go past the equivalence point?  
It is necessary to go past the equivalence point in order to find the point where the slope is the steepest. The curve needs to continue past the steepest point to ensure you can tell when the slope begins to flatten.
- 38.**  Stop recording data. ♦<sup>(6.2)</sup>
- 39.**  In Table 2, record the final drop count and the final reading of the titrant in the buret to a precision of 0.01 mL.
- 40.**  Calculate the volume of titrant (final reading minus initial reading) and record this value in Table 2.

Table 2: Titration data

Parameter	Measurement or Calculation
Starting volume of HCl in the buret (to 0.01 mL)	0.00
Final volume of HCl in the buret (to 0.01 mL)	29.00
Volume of titrant (to 0.01 mL)	29.00
Final drop count	562

- 41.**  Calibrate the drop counter. ♦<sup>(3.4)</sup>

- 42.**  Set the horizontal axis to the calculated volume. ♦<sup>(7.1.9)</sup>
- 43.**  From the graph, locate the phenolphthalein end point and record the volume in Table 3. ♦<sup>(9.1)</sup>
- Note:** The equivalence point will be where the slope of the titration curve is the steepest. Find the steepest slope of the data plot to determine this point. ♦<sup>(9.3)</sup>
- 44.**  From the graph, locate the methyl orange end point and record the volume in Table 3. ♦<sup>(9.1)</sup>
- 45.**  Print the graph. ♦<sup>(11.2)</sup>
- 46.**  Save your experiment. ♦<sup>(3.4)</sup> and clean up according to your teacher's directions.

## Data Analysis

- 1.**  Calculate the volume of HCl consumed to convert  $\text{NaHCO}_3$  to NaCl and  $\text{CO}_2$ .
- $$(24.56 \text{ mL}) - (22.32 \text{ mL}) = 2.24 \text{ mL HCl}$$
- 2.**  Calculate the number of moles of HCl consumed to convert  $\text{NaHCO}_3$  to NaCl and  $\text{CO}_2$ .
- $$(2.24 \text{ mL}) \left( \frac{4.00 \text{ mol}}{1000 \text{ mL}} \right) = 8.96 \times 10^{-3} \text{ mol HCl}$$
- 3.**  Calculate the number of moles of  $\text{NaHCO}_3$  converted to  $\text{CO}_2$  from the number of moles of HCl consumed.

The number of moles of  $\text{NaHCO}_3$  is the same as the number of moles of HCl consumed:  $8.96 \times 10^{-3}$  mol, because of the 1:1 stoichiometry.

- 4.**  Calculate the number of moles of  $\text{CO}_2$  that was needed to produce the number of moles of  $\text{NaHCO}_3$ .

It is the same as the number of moles of  $\text{NaHCO}_3$ , since 1 mol of  $\text{CO}_2$  yields 1 mol of  $\text{NaHCO}_3$ . Therefore the number of moles of  $\text{CO}_2$  is  $8.96 \times 10^{-3}$  mol.

- 5.**  Calculate the mass of  $\text{CO}_2$  in the sample.

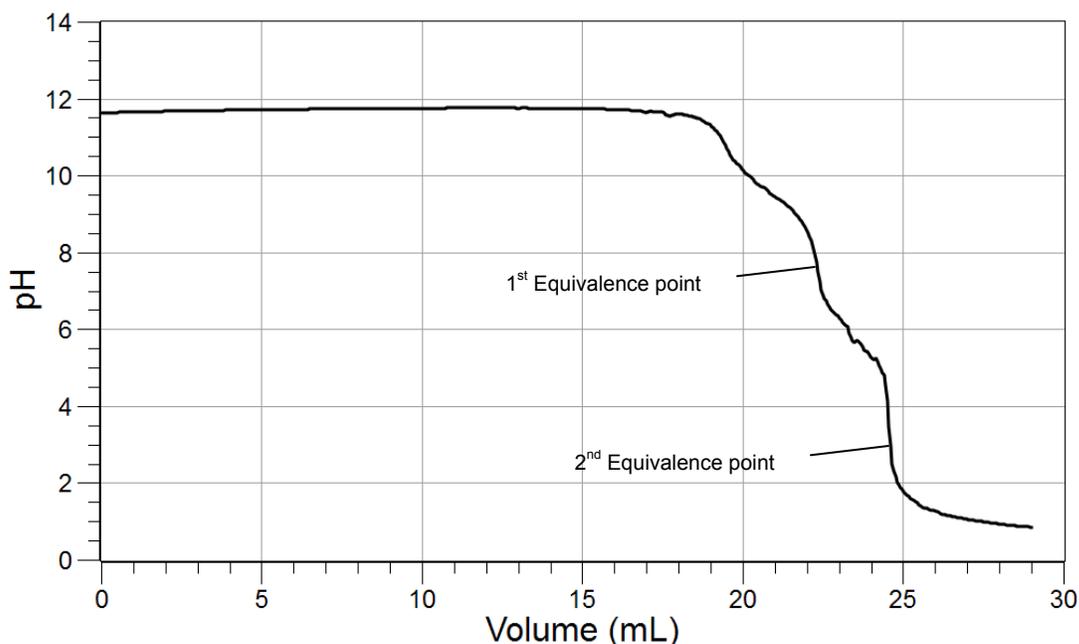
$$(8.96 \times 10^{-3} \text{ mol}) \left( \frac{44.0 \text{ g}}{1 \text{ mol}} \right) = 0.394 \text{ g CO}_2$$

## Lab 11: Using Different Indicators for pH Determination

Table 3: Determination of the amount of CO<sub>2</sub> in 100 mL of soda

Beverage	Diet Coke
Molarity of HCl of titrant (M)	4.0
Volume of titrant at phenolphthalein end point (mL)	22.32
Volume of titrant at methyl orange end point (mL)	24.56
Volume of titrant, 1st end point to 2nd end point	2.24
Amount of titrant, 1st end point to 2nd end point (mol)	$8.96 \times 10^{-3}$
Amount of CO <sub>2</sub> in the sample (mol)	$8.96 \times 10^{-3}$
Mass of CO <sub>2</sub> in sample (g)	0.394

6.  Sketch or attach the titration curve of pH versus Volume below.



### Analysis Questions

1. Usually the CO<sub>2</sub> content obtained this way is lower than the actual value listed on the container of the drink. List a few possible reasons.

Not all the CO<sub>2</sub> leaves the drink, some escapes when the can is opened, and CO<sub>2</sub> that didn't dissolve in the NaOH escapes when the balloon is removed from the Erlenmeyer flask.

2. How would you minimize the error introduced by losing CO<sub>2</sub> in the process?

Cooling down the drink as much as possible, minimizing the time it takes to transfer the drink into the Erlenmeyer flask.

**3. Why can't we simply add the NaOH solution to the soda and titrate the excess NaOH?**

We cannot just add the NaOH solution to the drink because most drinks have unknown amounts of various acids making it impossible to know how much NaOH actually reacts with the CO<sub>2</sub>.

**4. What aspect of this method is subjective and therefore prone to human error?**

Detection of color changes makes this method subjective.

**5. How would you minimize the error introduced by the detection of colors in the titration process?**

Using a pH electrode instead of the results obtained with the indicator solutions minimize the error.

**6. Many of the limitations in a titration using an indicator can be eliminated by using a drop counter and a pH sensor. What is a drop counter and why is it used in a titration? How does it overcome some of the limitations of indicator-based titrations?**

A drop counter is exactly what its name implies; it counts the number of drops that pass through it. The volume of each drop can be determined by dividing the volume of titrant used by the total number of drops used to dispense that volume. A calculation can then be used to convert the number of drops at the equivalence point of a titration curve to the volume of titrant used.

This procedure eliminates the need for stopping the titration exactly at the equivalence point, which can be tricky when using an indicator. The procedure can also be completed for titrations that cannot use indicators.

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**Synthesis Questions**

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Use available resources to help you answer the following questions.

**1. How would you adapt this method to determine the CaCO<sub>3</sub> content of a stomach acid pill (Hint: CaCO<sub>3</sub> reacts with HCl to form CO<sub>2</sub>) ?**

The acid could be reacted with excess amount of HCl to form CO<sub>2</sub>.



The rest of the method would be the same as in the original activity.

**2. Why do you think we don't usually use this method to determine the CaCO<sub>3</sub> content of a stomach acid pill but rather, react three pills with known amounts of HCl and then titrate the excess amount of NaOH? (Hint: what was the major source of error in the process?)**

The method applied in this activity is prone to significant error due to the potential loss of CO<sub>2</sub>. The back titration method is more accurate.

## Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

- 1. The number of moles of  $\text{CO}_2$  is equal to:**
  - A.** The number of moles of NaOH in the solution.
  - B.** The number of moles of HCl added up to the 1st equivalence point.
  - C.** The number of moles of HCl added up to the 2nd equivalence point.
  - D.** The number of moles of HCl added between the the 1st and 2nd equivalence points.
  
- 2. The number of moles of excess NaOH is:**
  - A.** The number of moles of NaOH in the solution.
  - B.** The number of moles of HCl added up to the 1st equivalence point.
  - C.** The number of moles of HCl added up to the 2nd equivalence point.
  - D.** The number of moles of HCl added up to the 1st equivalence point minus the number of moles of HCl added between the 1st and 2nd equivalence points.
  
- 3. Up to the first equivalence point:**
  - A.** All NaOH was converted to NaCl.
  - B.** All  $\text{CO}_2$  was converted to  $\text{NaHCO}_3$  and all excess NaOH was converted to NaCl.
  - C.** All  $\text{CO}_2$  was converted to NaCl.
  - D.** All NaOH was converted to  $\text{NaHCO}_3$ .
  
- 4. Between the 1st and 2nd equivalence points:**
  - A.** All NaOH was converted to NaCl.
  - B.** All  $\text{NaHCO}_3$  was converted to NaCl and  $\text{CO}_2$ .
  - C.** All  $\text{CO}_2$  was converted to NaCl.
  - D.** All NaOH was converted to  $\text{NaHCO}_3$ .

## Extended Inquiry Suggestions

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Have students use the back-titration method to determine the  $\text{CaCO}_3$  content of a stomach acid pill. Assuming the pills have 500 mg  $\text{CaCO}_3$ , two pills can be ground and reacted with 25.00 mL of 1.0 M HCl. The excess amount of HCl can be back-titrated with a 1.0 M NaOH solution. For this titration, methyl orange indicator can be used.

Then 25.00 mL of the HCl solution would be titrated with 1.0 M NaOH solution. The difference between the two titrations is due to the  $\text{CaCO}_3$  present in the pill. The number of moles of NaOH that is the difference between the two titrations is the same as the number of moles of HCl that reacted with the  $\text{CaCO}_3$  in the sample.

In a sample experiment, two pills were ground and transferred into a 100-mL beaker. The mass of the sample was 0.995 g. Then 25.00 mL of 1.00 M HCl was added and the solution was gently heated to eliminate the  $\text{CO}_2$  generated. The solution was titrated with 1.0 M NaOH in the

presence of methyl orange indicator and it took 16.50 mL titrant solution to reach the equivalence point. Then, 25.00 mL of 1.0 M HCl solution was titrated with 1.0 M NaOH in a separate experiment. It took 23.95 mL titrant solution to reach the equivalence point. The  $\text{CaCO}_3$  that was present in the sample used up:

$$(23.95 \text{ mL} - 16.50 \text{ mL}) \left( \frac{1.00 \text{ mol HCl}}{1000 \text{ mL}} \right) = 7.45 \times 10^{-3} \text{ mol HCl}$$

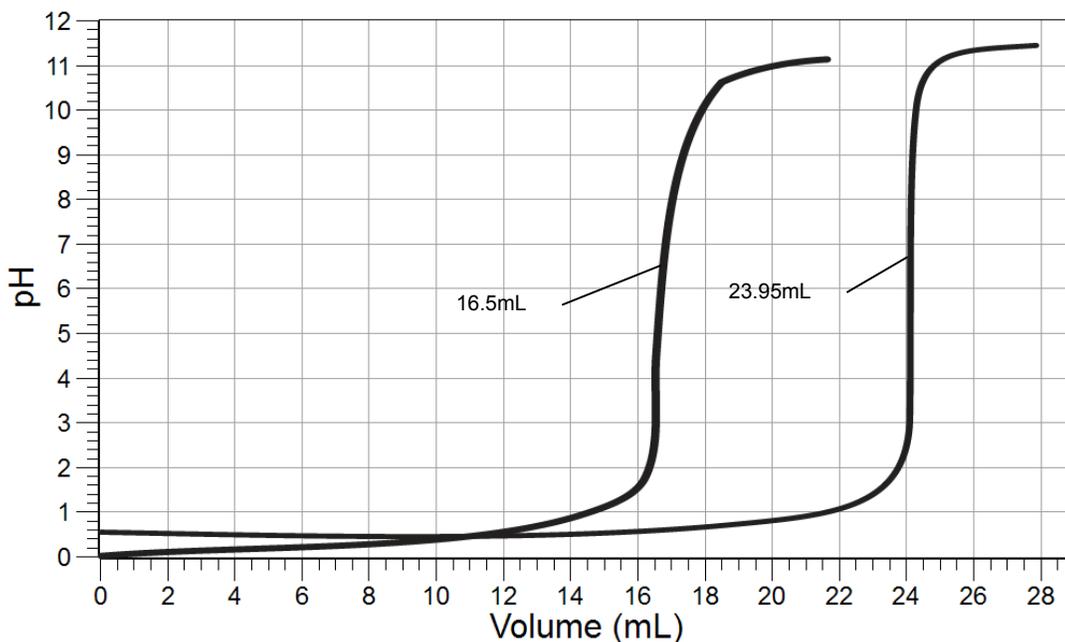
This much HCl is equivalent to

$$(7.45 \times 10^{-3} \text{ mol HCl}) \left( \frac{1 \text{ mol CaCO}_3}{2 \text{ mol HCl}} \right) \left( \frac{100.1 \text{ g CaCO}_3}{1 \text{ mol CaCO}_3} \right) = 3.72 \times 10^{-1} \text{ g CaCO}_3$$

The percentage of the stomach acid pills that is  $\text{CaCO}_3$  is

$$\text{Percent CaCO}_3 \text{ content} = \left( \frac{0.372 \text{ g}}{0.995 \text{ g}} \right) \times 100 = 37.2\% \text{ CaCO}_3$$

The titration curve obtained using a pH electrode appears as follows:





## Lab 12: Determination of the Rate of the Decomposition of Hydrogen Peroxide

### Objectives

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Students determine the rate constant of a chemical reaction.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Using a catalyst to increase the rate of a reaction
- ◆ Measuring the pressure generated by the reaction in order to determine the change in concentration of a reactant
- ◆ Calculating the order of the reactants as a means to calculating the rate constant of the limiting reaction

### Time Requirement

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- |                                   |            |
|-----------------------------------|------------|
| ◆ Preparation time                | 50 minutes |
| ◆ Pre-lab discussion and activity | 30 minutes |
| ◆ Lab activity                    | 90 minutes |

### Materials and Equipment

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#### *For each student or group:*

- |   |   |
|---|---|
| ◆ Data collection system  | ◆ Stopper with two holes for the Erlenmeyer flask                           |
| ◆ Absolute pressure sensor with quick-release connectors and plastic tubing | ◆ Beaker, 50-mL   |
| ◆ Stainless steel temperature sensor  | ◆ Glycerin, several drops   |
| ◆ Sensor extension cable  | ◆ 0.1000 M Potassium iodine (KI), 60 mL <sup>1</sup>                        |
| ◆ Beaker (3), 100-mL  | ◆ 3% Hydrogen peroxide (H <sub>2</sub> O <sub>2</sub> ), 40 mL <sup>2</sup> |
| ◆ Erlenmeyer flask, 250-mL  | ◆ Deionized water , 100 mL  |
| ◆ Graduated pipet (3), 25-mL with rubber bulb                               | ◆ Electrical tape, 60 in. (optional)  |

<sup>1</sup> To prepare the 0.1000 M KI solution, refer to the Lab Preparation section.

<sup>2</sup> 3% hydrogen peroxide solution is readily available in pharmacies.

## **Concepts Students Should Already Know**

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Students should be familiar with the following concepts:

- ◆ Titration
- ◆ Acid-base reactions
- ◆ Stoichiometry of chemical reactions
- ◆ Rate of chemical reaction
- ◆ Order of reactants and overall order of reactions

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 5: Molar Volume of a Gas
- ◆ Lab 25: Order of Reaction
- ◆ Lab 27: Identifying an Unknown Metal
- ◆ Lab 29: Exploring Gas Laws

## **Using Your Data Collection System**

---

Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a temperature sensor and a pressure sensor to your data collection system ◆<sup>(2.2)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Displaying data on a graph ◆<sup>(7.1.1)</sup>
- ◆ Displaying multiple data runs in a graph. ◆<sup>(7.1.3)</sup>
- ◆ Finding the slope and an intercept of a best-fit line ◆<sup>(9.6)</sup>
- ◆ Saving your experiment ◆<sup>(11.1)</sup>
- ◆ Printing ◆<sup>(11.2)</sup>

## Background

Hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) in aqueous solution decomposes very slowly under ordinary conditions. The equation for the decomposition is

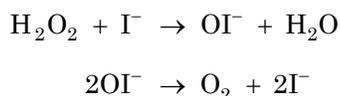


A catalyst such as potassium iodide, manganese dioxide, or catalase enzyme may be used to increase the rate of reaction. Conducting a catalyzed decomposition of  $\text{H}_2\text{O}_2$  in a closed vessel enables the determination of the reaction rate based on the pressure increase from the production of oxygen gas. Each  $\text{H}_2\text{O}_2$  molecule yields one  $\text{O}_2$  molecule; therefore, the rate at which  $\text{H}_2\text{O}_2$  disappears is the same rate at which  $\text{O}_2$  is formed:

$$-\frac{\Delta[\text{H}_2\text{O}_2]}{\Delta t} = \frac{\Delta[\text{O}_2]}{\Delta t}$$

Because the concentration of oxygen is proportional to its pressure, we can calculate the rate at which  $\text{H}_2\text{O}_2$  decomposes by monitoring the rate of increase of the pressure due to the formation of oxygen. By varying the initial molar concentration of  $\text{H}_2\text{O}_2$  solution, the rate law for the reaction can be determined.

There are two steps involved in the decomposition of hydrogen peroxide with potassium iodide as the catalyst:



The first reaction determines the rate, that is, it goes much slower than the second reaction. The rate of the rate-determining reaction is calculated as follows:

$$-\frac{\Delta[\text{H}_2\text{O}_2]}{\Delta t} = k_1[\text{H}_2\text{O}_2]^m[\text{I}^-]^n \quad (2)$$

where

$k_1$  = the rate constant of the first reaction

$m$  = the order of  $\text{I}^-$  in the first reaction

$n$  = the order of  $\text{H}_2\text{O}_2$  in the first reaction

In this experiment, we determine  $n$  and  $m$ , as well as  $k_1$ .

As  $\text{O}_2$  is a gas, it makes more sense to work with the number of moles than with concentration to obtain the concentration of  $\text{H}_2\text{O}_2$ . For Equation 1,

$$-\frac{1}{2} \frac{\Delta n_{\text{H}_2\text{O}_2}}{\Delta t} = \frac{\Delta n_{\text{O}_2}}{\Delta t}$$

## Lab 12: Determination of the Rate of the Decomposition of Hydrogen Peroxide

While we cannot measure the change of number of moles of  $O_2$ , using the ideal gas law we can calculate it from the change of pressure, which we can measure:

$$-\frac{1}{2} \frac{\Delta n_{H_2O_2}}{\Delta t} = \frac{V}{RT} \frac{\Delta p}{\Delta t}$$

where

$V$  = the volume that the  $O_2$  can occupy

$R$  = the gas constant

$T$  = the temperature inside the flask

$p$  = the partial pressure of the  $O_2$  generated by the reactions

Now we can return to calculating the change of concentration for  $H_2O_2$  as well:

$$V_s [H_2O_2] = n_{H_2O_2}$$

$$V_s (\Delta [H_2O_2]) = \Delta n_{H_2O_2}$$

$$-\frac{V_s}{2} \frac{\Delta [H_2O_2]}{\Delta t} = \frac{V}{RT} \frac{\Delta p}{\Delta t}$$

where

$V_s$  = volume of the solution

We can rearrange the formula to get the rate of the reaction:

$$\frac{\Delta [H_2O_2]}{\Delta t} = -\frac{2V}{V_s RT} \frac{\Delta p}{\Delta t}$$

Substituting this into Equation 2, we can calculate the rate constant:

$$\frac{2V}{V_s RT} \frac{\Delta p}{\Delta t} = k_1 [H_2O_2]^m [I^-]^n$$

$$k_1 = \frac{2V}{[H_2O_2]^m [I^-]^n V_s RT} \frac{\Delta p}{\Delta t} \quad (3)$$

To determine the order of the reactants,  $n$  and  $m$ , the reactions are performed according to the following table.

Table1: Concentration ratios between the two reactants

Reaction	Conc. of $\text{H}_2\text{O}_2$	Conc. of $\text{I}^-$
1	$[\text{H}_2\text{O}_2]$	$[\text{I}^-]$
2	$[\text{H}_2\text{O}_2]$	$2[\text{I}^-]$
3	$2[\text{H}_2\text{O}_2]$	$[\text{I}^-]$

Determining the rate for reactions 1 and 2:

$$\text{Rate}_1 = k_1[\text{H}_2\text{O}_2]^m[\text{I}^-]^n$$

$$\text{Rate}_2 = k_1[\text{H}_2\text{O}_2]^m(2[\text{I}^-])^n$$

The ratio of the two rates yields

$$\frac{\text{Rate}_2}{\text{Rate}_1} = \frac{k_1[\text{H}_2\text{O}_2]^m(2[\text{I}^-])^n}{k_1[\text{H}_2\text{O}_2]^m[\text{I}^-]^n}$$

$$\frac{\text{Rate}_2}{\text{Rate}_1} = 2^n$$

$$\ln\left(\frac{\text{Rate}_2}{\text{Rate}_1}\right) = \ln 2^n$$

$$\ln\left(\frac{\text{Rate}_2}{\text{Rate}_1}\right) = n \ln 2$$

$$n = \frac{\ln\left(\frac{\text{Rate}_2}{\text{Rate}_1}\right)}{\ln 2}$$

Using the same argument to derive  $m$  from reactions 1 and 3:

$$m = \frac{\ln\left(\frac{\text{Rate}_3}{\text{Rate}_1}\right)}{\ln 2}$$

The values of the order of the reactants,  $m$  and  $n$ , then will be used to determine  $k_1$ , using Equation 3.

## Pre-Lab Activity

### Setting the stage for the activity

You will perform the three reactions described above and monitor the change of pressure. This allows the respective rates to be calculated. From this calculation the rate constant of the rate determining step  $k_1$  can be calculated. To calculate  $n$  and  $m$ , it is not necessary to calculate the actual rates because both  $n$  and  $m$  depend only on the ratio of the rates. Therefore, the ratio of the slopes of the pressure versus time graph is sufficient to determine  $n$  and  $m$ .

### Example calculation to try

A commercially available 3% hydrogen peroxide solution was analyzed. After mixing the potassium iodide and hydrogen peroxide solutions, the volume of the solution  $V_s$  was 60.0 mL. The volume  $V$  that the  $O_2$  gas could occupy was 242 mL, and the reaction temperature  $T$  was carried out at 303 K. Table 2 shows the results.

Table 2: Results of the 3 reactions

Reaction	$[H_2O_2]$ (M)	$[I^-]$ (M)	$\Delta p/\Delta t$ (Pa/s)	$n$	$m$
1	0.147	0.0250	29.3	1	1
2	0.294	0.0250	60.6		
3	0.147	0.0500	61.6		

The following calculations determine  $n$  and  $m$ :

$$n = \frac{\ln\left(\frac{6.06 \times 10^{-2}}{2.93 \times 10^{-2}}\right)}{\ln(2)} = 1.050 \approx 1$$

$$m = \frac{\ln\left(\frac{6.16 \times 10^{-2}}{2.93 \times 10^{-2}}\right)}{\ln(2)} = 1.070 \approx 1$$

Both  $n$  and  $m$  must be integers; the closest integer for each is 1.

Now we can calculate the rate constant. For instance, we can use reaction 1:

$$k_1 = \frac{(2)(2.42 \times 10^{-4} \text{ m}^3)}{\left(0.147 \frac{\text{mol}}{\text{L}}\right)\left(0.0250 \frac{\text{mol}}{\text{L}}\right)(0.0600 \text{ L})\left(8.314 \frac{\text{Nm}}{\text{mol K}}\right)(303 \text{ K})} \left(29.3 \frac{\text{N}}{\text{m}^2 \text{ s}}\right) \quad (4)$$

$$k_1 = 2.55 \times 10^{-2} \frac{1}{\text{Ms}}$$

**1. Why is the unit for the volume of the solution inserted as “L” instead of “m<sup>3</sup>”, the SI unit for volume?**

The rate of a chemical reaction is given as the change of the concentration of a substance in mol/L divided by the time for that change. Therefore, the volume of the solution has to be in liters.

**2. Why do both *n* and *m* have to be rounded to the closest integer, in this case 1?**

Since both *n* and *m* are stoichiometric coefficients of the chemical equation, they must be integers.

---

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

- 1. 0.1000 M KI:** Dissolve 8.300 g KI in some water in a 500-mL volumetric flask and then fill it to the mark with distilled water.
- 2.** Commercially available 3% hydrogen peroxide can be used directly.

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## Safety

Follow all standard laboratory procedures.

### Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

2	5	6	4
Add the $\text{H}_2\text{O}_2$ solution quickly to the solution in the Erlenmeyer flask.	Perform the reaction two more times with different $\text{H}_2\text{O}_2$ and KI concentrations.	Calculate the rate constant of the limiting reaction.	Monitor the pressure until it stabilizes.
1	3		
Set up the data collection system. Pipet the specified amount of KI solution and water into a flask and the specified amount of $\text{H}_2\text{O}_2$ into a small beaker.	Immediately plug the flask with a stopper with a pressure sensor and temperature sensor.		

### Procedure with Inquiry

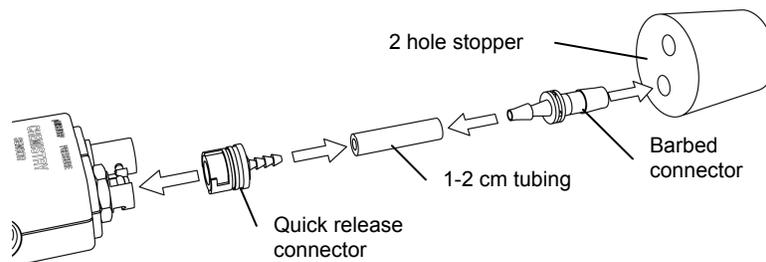
After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "♦" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

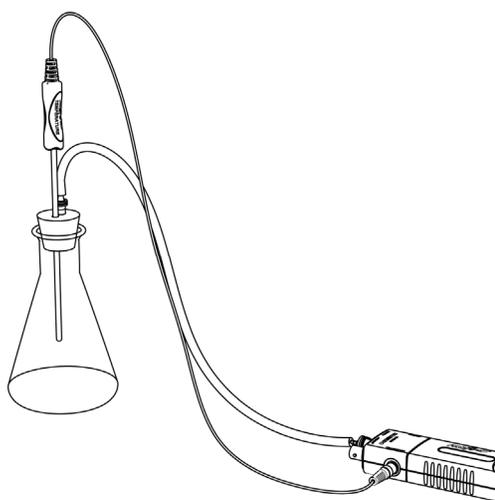
#### Set Up

- Start a new experiment on the data collection system. ♦<sup>(1,2)</sup>

2.  Place the barbed connector of the pressure sensor tightly into the rubber stopper and connect it to the pressure port of the sensor with a piece of tubing. If necessary, add a drop of glycerin onto the end of the connector that goes into the hole in the rubber stopper.



3.  Insert the temperature sensor into the other hole in the rubber stopper. If necessary, add a drop of glycerin.
4.  If electrical tape is available, wrap the Erlenmeyer flask with 10 to 15 rounds of electric tape. This is a preventive measure in case the flask cracks. The tape keeps the glass pieces together.
5.  Connect the absolute pressure sensor to the data collection system using a sensor extension cable.  $\diamond^{(2.2)}$



6.  Display Pressure on the y-axis with Time on the x-axis.  $\diamond^{(7.1.1)}$

### Collect Data

Table 3: Reactant amounts to use for the three reactions

Reaction	3% H <sub>2</sub> O <sub>2</sub> (mL)	0.1 M KI (mL)	Water (mL)
1	10.00	15.00	35.00
2	20.00	15.00	25.00
3	10.00	30.00	20.00

7.  Perform each of the three reactions, using the measured amounts given in Table 3, according the steps listed below.
- With graduated pipets, measure and transfer the water and potassium iodide solution into the 250-mL Erlenmeyer flask.
  - With a graduated pipet, measure and transfer the prescribed amount of H<sub>2</sub>O<sub>2</sub> solution into the 50-mL beaker.

## Lab 12: Determination of the Rate of the Decomposition of Hydrogen Peroxide

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- c. Pour the  $\text{H}_2\text{O}_2$  solution into the Erlenmeyer flask and immediately insert the rubber stopper into the flask.

**Important:** Make sure that the stopper is sitting firmly in the flask. Pressure is building in the flask and a loose stopper might pop out. If that happens, you will need to repeat the experiment.

- d. Start recording data.  $\diamond^{(6.2)}$
- e. Continue to record the data for three minutes. Gently shake the Erlenmeyer flask constantly during data collection. Hold the stopper firmly during the experiment.
- f. Why do you think it is necessary to shake the solution (Hint: what is the product of the reaction)?

We need to shake the solution to make sure that the oxygen generated quantitatively leaves the solution.

- g. Stop recording data.  $\diamond^{(6.2)}$

**Note:** The initial portion of the pressure versus time graph is not straight, which is attributed to the fact that the reaction does not begin immediately.

8.  Display the three data runs on a graph.  $\diamond^{(7.1.3)}$
9.  Print the graph.  $\diamond^{(11.2)}$
10.  Save your experiment and clean up according to your teacher's instructions.  $\diamond^{(11.1)}$

### Data Analysis

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1.  Convert the percent by mass concentration to molarity for the different volumes of  $\text{H}_2\text{O}_2$ . Record the values in Table 4.

For Reaction 1 the 60.00 mL reaction mixture had

$$\left(\frac{3.00 \text{ g H}_2\text{O}_2}{100 \text{ mL}}\right)(10 \text{ mL}) = 0.300 \text{ g H}_2\text{O}_2$$

The number of moles of the  $\text{H}_2\text{O}_2$  in the 60.00 mL solution is

$$\left(\frac{0.300 \text{ g}}{34 \frac{\text{g}}{\text{mol}}}\right) = 8.82 \times 10^{-3} \text{ mol H}_2\text{O}_2$$

The concentration of  $\text{H}_2\text{O}_2$  is

$$\left(\frac{8.82 \times 10^{-3} \text{ mol}}{0.0600 \text{ L}}\right) = 0.147 \text{ M}$$

2.  Find the slope of the best-fit line for each data run on the Absolute Pressure versus Time graph and enter the value below. <sup>(9.6)</sup> Record the values in Table 4 using the appropriate units.

$$\text{Slope}_1 (\text{kPa/s}): \quad \underline{\quad 0.0244 \quad}$$

$$\text{Slope}_2 (\text{kPa/s}): \quad \underline{\quad 0.0444 \quad}$$

$$\text{Slope}_3 (\text{kPa/s}): \quad \underline{\quad 0.0482 \quad}$$

Converting the units for the slope of Reaction 1:

$$2.44 \times 10^{-2} \text{ kPa/s} = 24.4 \text{ Pa/s}$$

3.  Calculate the order of the two reactants from the respective rates.

$$n = \frac{\ln\left(\frac{44.4}{24.4}\right)}{\ln(2)} = 0.864$$

$$m = \frac{\ln\left(\frac{48.2}{24.4}\right)}{\ln(2)} = 0.982$$

4.  Calculate the rate constants, using Equation 3, by substituting your respective experimental data (refer to Equation 4 as an example). Record the values in Table 4 and determine the average value of  $k_1$ .

Calculating the rate constant for Reaction 1:

$$k_1 = \frac{(2)(2.42 \times 10^{-4} \text{ m}^3)}{\left(0.147 \frac{\text{mol}}{\text{L}}\right)\left(0.0250 \frac{\text{mol}}{\text{L}}\right)(0.06000 \text{ L})\left(8.314 \frac{\text{Nm}}{\text{mol K}}\right)(303 \text{ K})} \left(24.4 \frac{\text{N}}{\text{m}^2 \text{ s}}\right)$$

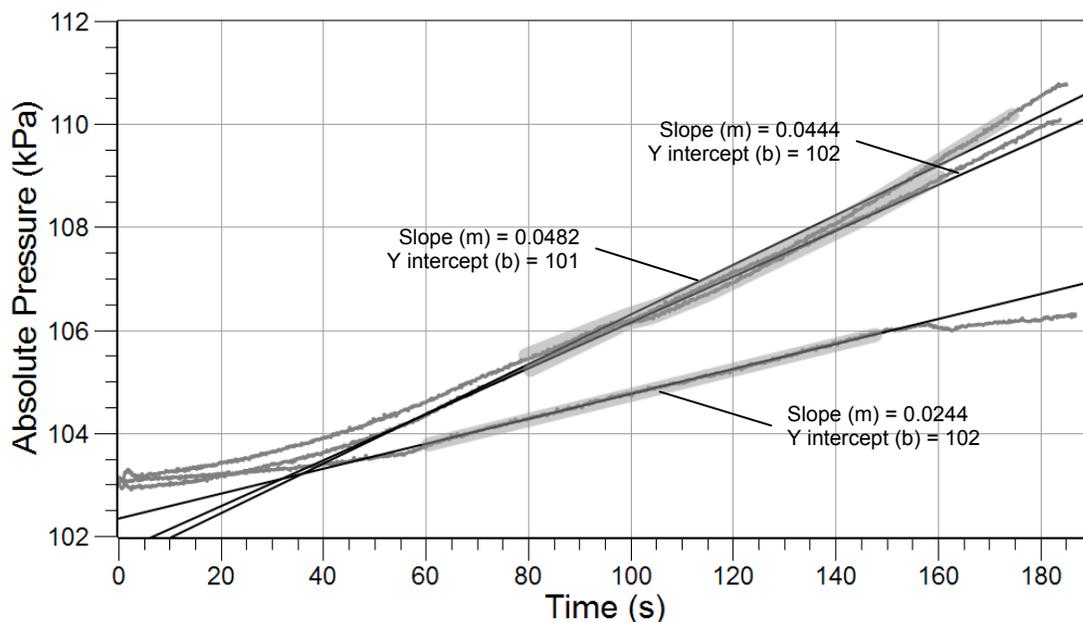
$$k_1 = 2.13 \times 10^{-2} \frac{1}{\text{Ms}}$$

Table 4: Experimental data and data analysis

Reaction	[H <sub>2</sub> O <sub>2</sub> ] (M)	[I <sup>-</sup> ] (M)	$\Delta p/\Delta t$ (Pa/s)	$n$	$m$	$k_1$ (M <sup>-1</sup> s <sup>-1</sup> )
1	0.147	0.025	24.4	0.864	0.982	$2.13 \times 10^{-2}$
2	0.294	0.025	44.4			$1.93 \times 10^{-2}$
3	0.147	0.050	48.2			$2.10 \times 10^{-2}$

Average  $k_1$ :  $\underline{\quad 2.05 \times 10^{-2} \text{ M}^{-1} \text{ s}^{-1} \quad}$

5.  Sketch or attach the graph showing the curves and specifying the slopes of the three reactions.



### Analysis Questions

1. Why can we use the rate of change of pressure directly instead of the rate of change of concentration to calculate  $n$  and  $m$ ?

We need to calculate only the ratio of the two rates. The conversion of rate of change of pressure to rate of change of concentration is achieved by multiplication and division with the same numbers for both rates (in the numerator and denominator). Therefore, they don't change the ratio and cancel out.

2. What might the reason be if the pressure starts to decrease instead of increase?

This would indicate there is a leak in the system. Most likely the stopper is not firmly placed.

3. Did the temperature increase? Why or why not?

Because the reaction is exothermic, it releases heat. This increases the temperature of the solution.

4. As you probably observed, the initial portion of the graphs may have curved a little due to the fact that there is an induction time for this reaction. Did that introduce any error into your measurement?

No, it did not introduce errors because we calculated the rate from the slope of the linear portion of the graph.

5. Based on the values of  $n$  and  $m$ , what is the overall order of the reaction?

The reaction is a second order reaction.

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## Synthesis Questions

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Use available resources to help you answer the following questions.

- 1. How would the slope for the second reaction have changed (when we doubled the concentration of  $\Gamma$ ), if  $n$  were 2?**

Instead of doubling, it would have been increased 4-fold.

- 2. How would the slope have changed if  $n$  were 2 and we doubled both the  $\Gamma$  and  $\text{H}_2\text{O}_2$  concentrations?**

The slope would have increased 4-fold as well.

- 3. If you check the equations for the two consecutive reaction steps, you will notice that the  $\Gamma$  ions are recovered and not consumed in the reaction. How is that possible?**

The  $\Gamma$  ions are catalysts and therefore do not change during the reaction.

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## Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

- 1. The order of  $\text{H}_2\text{O}_2$  in the reaction is:**

- A. The slope of the pressure versus time graph.
- B. 1
- C. 2
- D. The value of the rate constant,  $k_1$

- 2. The rate constant of the decomposition was determined from:**

- A. The concentration of the reactants.
- B. The ratio of the slopes of the pressure versus time graphs.
- C. The slope of the pressure versus time graph.
- D. The temperature versus time graph.

- 3. Since  $n$  or  $m$ , or both, probably did not come out to be an integer, which of the following statements is correct regarding this fact?**

- A. There is an experimental error.
- B. You must round to the nearest integer.
- C. Both were determined experimentally.
- D. All three statements are correct.

### Extended Inquiry Suggestions

The reaction can be performed at different temperatures to determine the activation energy of the reaction:

$$k = Ae^{-E_a/RT}$$

$$\ln k = \ln A - \frac{E_a}{R} \frac{1}{T}$$

The activation energy ( $E_a$ ) can be determined from the slope of the  $\ln k$  versus  $1/T$  plot.

Experimental data from an experiment performed at 298 K.

T = 298 K	[H <sub>2</sub> O <sub>2</sub> ] (M)	[I <sup>-</sup> ] (M)	$\Delta p/\Delta t$ (Pa/s)	<i>n</i>	<i>m</i>	$k_1$ (M <sup>-1</sup> s <sup>-1</sup> )	
1	0.147	0.025	$2.44 \times 10^{-2}$	0.864	0.902	0.0216	$2.08 \times 10^{-2}$
2	0.294	0.025	$4.44 \times 10^{-2}$			0.0197	
3	0.147	0.050	$4.82 \times 10^{-2}$			0.0213	

Experimental data from an experiment performed at 303 K.

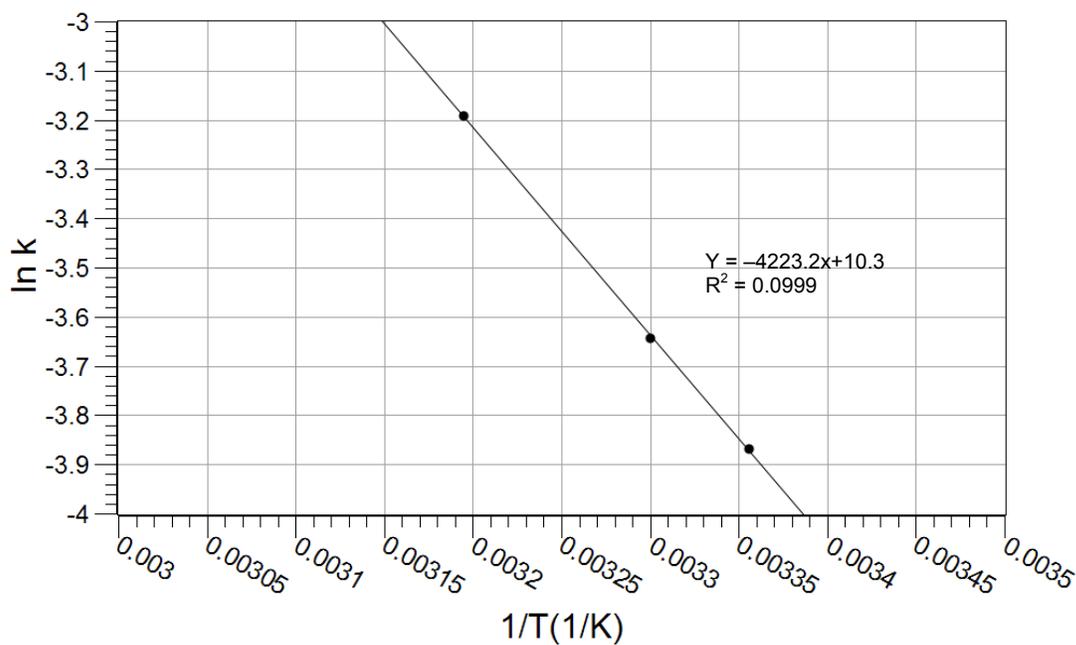
T = 303 K	[H <sub>2</sub> O <sub>2</sub> ] (M)	[I <sup>-</sup> ] (M)	$\Delta p/\Delta t$ (Pa/s)	<i>n</i>	<i>m</i>	$k_1$ (M <sup>-1</sup> s <sup>-1</sup> )	
1	0.147	0.025	$2.93 \times 10^{-2}$	1.07	1.04	0.0255	$2.62 \times 10^{-2}$
2	0.294	0.025	$6.16 \times 10^{-2}$			0.0268	
3	0.147	0.05	$6.01 \times 10^{-2}$			0.0262	

Experimental data from an experiment performed at 313 K.

T = 313 K	[H <sub>2</sub> O <sub>2</sub> ] (M)	[I <sup>-</sup> ] (M)	$\Delta p/\Delta t$ (Pa/s)	<i>n</i>	<i>m</i>	$k_1$ (M <sup>-1</sup> s <sup>-1</sup> )	
1	0.147	0.025	$4.81 \times 10^{-2}$	1.08	0.971	0.0406	$4.11 \times 10^{-2}$
2	0.294	0.025	$1.02 \times 10^{-1}$			0.0430	
3	0.147	0.05	$9.43 \times 10^{-2}$			0.0398	

Temperature dependence of  $k_1$ .

$k$ ( $M^{-1} s^{-1}$ )	$\ln k$	T (K)	$1/[T$ (K)]
$2.08 \times 10^{-2}$	-3.86941	298	$3.36 \times 10^{-3}$
$2.62 \times 10^{-2}$	-3.64302	303	$3.30 \times 10^{-3}$
$4.11 \times 10^{-2}$	-3.19165	313	$3.20 \times 10^{-3}$



The activation energy is

$$E_a = -(-4223.2 \text{ K}) \left( 8.314 \frac{\text{J}}{\text{mol K}} \right) = 35,112 \frac{\text{J}}{\text{mol}} = 35.1 \frac{\text{kJ}}{\text{mol}}$$



## Lab 13: Enthalpy of a Chemical Reaction

### Objectives

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Students use Hess's Law to calculate the enthalpy change of a reaction between ammonia and hydrochloric acid in aqueous solution.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Carrying out and monitoring the temperature change of 3 reactions
- ◆ Comparing the results of the enthalpy calculated from the heat of reaction to that calculated using Hess's Law

### Time Requirement

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◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	30 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

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#### *For each student or group:*

- ◆ Data collection system
- ◆ Stainless steel temperature sensor
- ◆ Polystyrene cup, 8 oz.
- ◆ Clamp, utility
- ◆ Beaker, 250-mL
- ◆ Graduated cylinder, 50-mL or 100-mL
- ◆ Ring stand
- ◆ 2.00 M Sodium hydroxide (NaOH), 50 mL<sup>1</sup>
- ◆ 2.00 M Hydrochloric acid (HCl), 50 mL<sup>2</sup>
- ◆ 2.00 M Ammonium chloride (NH<sub>4</sub>Cl), 50 mL<sup>3</sup>
- ◆ 2.00 M Ammonia (NH<sub>3</sub>), 50 mL<sup>4</sup>

<sup>1-4</sup>To prepare the solutions, refer to the Lab Preparation section

## **Concepts Students Should Already Know**

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Students should be familiar with the following concepts:

- ◆ Stoichiometric calculations
- ◆ Hess's Law
- ◆ First Law of Thermodynamics
- ◆ Balancing chemical equations

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 12: Determination of the Rate of the Decomposition of Hydrogen Peroxide

## **Using Your Data Collection System**

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting sensors to the data acquisition device ◆<sup>(2.1)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Saving your experiment ◆<sup>(11.1)</sup>

## **Background**

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### **Hess's Law**

Hess's Law states that if a chemical reaction is the combination of other chemical reactions, then the heat of reaction is the sum of the heat of reactions of the combined reactions. There are two important components of Hess's Law. First, the heat of reaction is independent of the path of the reaction. This means the heat of reaction is the same if the reaction is performed in one step or through multiple reactions.

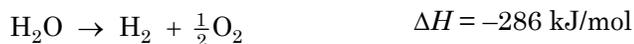
Second, if we know the heat of reaction for each step except one and we know the heat of reaction for the complete reaction, we can calculate the heat of reaction for the unknown step. This is an important tool for obtaining the heat of reaction for reactions that are difficult to perform.

To use Hess's Law, we often use the theoretical form of the equation even though it might not accurately represent how the reaction takes place. For example, Equation 1 is the theoretical representation for the production of water.



In the actual process, each O<sub>2</sub> molecule reacts with two H<sub>2</sub> molecules.

Often we must use the reverse of an equation when we combine multiple equations in order to get a final equation. The reverse of Equation 1 is



While this reaction does not occur naturally, the energy change of this theoretical process is accurate. (It is the same as the forward reaction but it has the opposite sign.)

### The First Law of Thermodynamics

Thermochemistry studies are based on measuring the heat that is released or absorbed in a chemical process. The First Law of Thermodynamics states that energy is conserved in a process; therefore, any energy released or absorbed as heat can be measured by its direct effect on the environment. For example, if a reaction is performed in an aqueous solution, released heat will increase the temperature of the solution and we can determine the amount of heat involved in the reaction from the temperature change. The heat  $q$  lost or gained by the system can be calculated as follows:

$$q = mc\Delta T$$

where

$q$  = heat lost or gained (J)

$m$  = mass of the solution (g)

$c$  = specific heat of the solution [J/(g °C)]

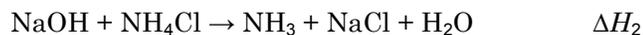
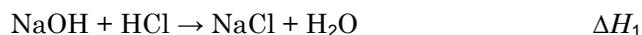
$\Delta T$  = change of temperature due to the reaction.

(The product of  $m$  and  $c$  is the "heat capacity"  $C$  (J/°C) of the solution.)

### Pre-Lab Activity

#### Setting the stage for the activity

In order to calculate the enthalpy change in a reaction, you will perform the following reactions:



## Lab 13: Enthalpy of a Chemical Reaction

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Based on the heat of reaction of these reactions, you will predict how to calculate the heat of reaction for the following reaction:



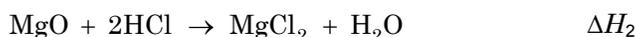
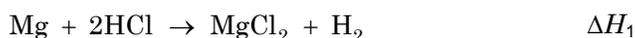
You will perform the third reaction to confirm your prediction.

You will perform the reactions in a polystyrene cup placed inside a beaker. The polystyrene cup is a good heat insulator and insulation is enhanced by the beaker around it. Therefore, the escape of heat is reduced. The temperature change of the solution in the cup will be monitored with a temperature sensor.

### Example calculation to try

The heat of reaction between magnesium (Mg) and oxygen (O<sub>2</sub>) is to be measured. While this reaction occurs directly (quite spectacularly, actually), it would be very difficult to measure the heat of reaction directly. Instead, use Hess's Law to determine the heat of reaction indirectly.

The following reactions will be used:



We measured the heat of reactions for the first two reactions and used the published value for the third.

To obtain the desired equation, the second equation has to be reversed, so that the sign of  $\Delta H_2$  is changed:



The heat of reaction will be:  $\Delta H_4 = \Delta H_1 + (-\Delta H_2) + \Delta H_3$

The reactions were carried out in a calorimeter made from a polystyrene cup with 50.0 mL of a 2 M HCl solution, set in a beaker. The heat capacity of the calorimeter was:

$$C = (50.0 \text{ g}) \left( 4.18 \frac{\text{J}}{\text{g} \cdot ^\circ\text{C}} \right) + \left( 15.0 \frac{\text{J}}{^\circ\text{C}} \right) = 224 \frac{\text{J}}{^\circ\text{C}}$$

where 4.18 J/(g °C) is the specific heat of water and 15.0 J/°C is the heat capacity of the cup. The heat capacity of the calorimeter, including the material of the calorimeter and the solution, is the amount of the heat that increases the temperature of the entire calorimeter by 1 °C.

In the calorimeter, the magnesium sample was added to the 50 mL of 2 M HCl and the change in temperature obtained. In the second experiment in the calorimeter, the sample of MgO was added to another 50 mL of 2 M HCl solution and the change in temperature also obtained. The experimental data was recorded in Table 1.

Table 1: Experimental and calculated data to determine the heat of reaction

Parameters	Reaction 1 (Mg + HCl)	Reaction 2 (MgO + HCl)
$C$ (J/°C)	224	224
$m$ (g)	0.4654	1.055
$T_i$ (°C)	23.5	22.5
$T_f$ (°C)	66.9	38.0
$\Delta T$ (°C)	43.4	15.5
$q$ (J)	$9.72 \times 10^3$	$3.47 \times 10^3$
$\Delta H$ (kJ/mol)	508	133

The heat gain of Reaction 1 was

$$q = C\Delta T = \left(224 \frac{\text{J}}{^\circ\text{C}}\right)(43.4 \text{ }^\circ\text{C}) = 9.72 \times 10^3 \text{ J}$$

This heat was produced by 0.4654 g of Mg, which is

$$\frac{(0.4654 \text{ g})}{\left(24.3 \frac{\text{g}}{\text{mol}}\right)} = 1.92 \times 10^{-2} \text{ mol Mg}$$

Therefore, the molar heat of reaction is

$$\Delta H_1 = \frac{(-9721.6 \text{ J})}{\left(1.92 \times 10^{-2} \text{ mol}\right)} = -508 \frac{\text{kJ}}{\text{mol}}$$

The negative sign indicates the reaction was exothermic.

The value of  $\Delta H_2$  can be calculated the same way. Note that the sign of  $q$  is reversed because  $\Delta H_4$  is calculated with the second reaction reversed.

The heat of reaction for the desired reaction is:

$$\Delta H_4 = \Delta H_1 - \Delta H_2 + \Delta H_3 = \left(-508 \frac{\text{kJ}}{\text{mol}}\right) - \left(-133 \frac{\text{kJ}}{\text{mol}}\right) + \left(-286 \frac{\text{kJ}}{\text{mol}}\right) = -640 \frac{\text{kJ}}{\text{mol}}$$

## Lab 13: Enthalpy of a Chemical Reaction

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**1.** Refer to the equations you will be studying during this activity. Combine the first two reactions in such a way that the resulting combination will result in the third reaction. (Hint: If you reverse a reaction, the heat of reaction has the same value, but the opposite sign.)

The second reaction would have to be reversed:



**2.** How do you calculate the heat of reaction for the third reaction in terms of the heat of reactions for the first two reactions?

$$\Delta H_3 = \Delta H_1 + (-\Delta H_2) \text{ or } \Delta H_3 = \Delta H_1 - \Delta H_2$$

### Lab Preparation

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These are the materials and equipment to set up prior to the lab:

- 1. 2.00 M NaOH:** Dissolve 160 g of NaOH in some water in a 2-L volumetric flask. After the solution cools, fill the flask to the mark. (Do not store NaOH in a volumetric flask. Over time the NaOH reacts with the glass and will cause you to be unable to open the flask.)
- 2. 2.00 M HCl:** Under a hood, slowly add 330 mL of 36% HCl solution to about 500 mL of water in a 2-L volumetric flask. Fill the flask to the mark.
- 3. 2.00 M NH<sub>4</sub>Cl:** Dissolve 214 g NH<sub>4</sub>Cl in some water in a 2-L volumetric flask and fill it to the mark.
- 4. 2.00 M NH<sub>3</sub>:** Under a hood add 260 mL of 30% ammonia solution to some water in a 2-L volumetric flask. Fill the flask to the mark. If a hood is not available, obtain pre-made 2 M ammonia solution.

### Safety

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Add these important safety precautions to your normal laboratory procedures:

- ◆ Avoid contact with the ammonia solution. Ammonia can irritate your nose or eyes. If irritation occurs, breathe fresh air.
- ◆ If the NaOH or HCl solutions come in contact with your skin or eyes, rinse immediately with a large amount of running water.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

3	1	5	2	4
Perform the first reaction (HCl with NaOH) in the calorimeter and record the temperature.	Determine how you would calculate the heat of reaction of the 3 <sup>rd</sup> reaction using the heat of reaction of the 1 <sup>st</sup> and 2 <sup>nd</sup> reaction.	Calculate $\Delta H$ for the 3 <sup>rd</sup> reaction two ways, including the way determined at the beginning of the activity. Compare results.	Assemble your experimental setup with a polystyrene cup, beaker, and temperature sensor.	Perform reactions between NaOH and NH <sub>4</sub> Cl and between NH <sub>3</sub> and HCl, recording the temperature for each.

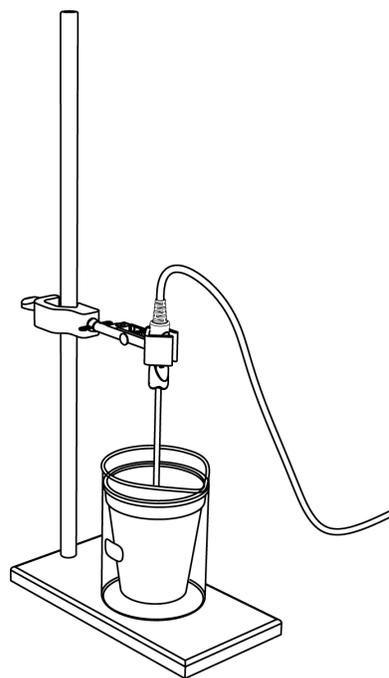
## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "♦" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

- Start a new experiment on the data collection system. ♦<sup>(1.2)</sup>
- Connect a temperature sensor to the data collection system. ♦<sup>(2.1)</sup>
- Place the polystyrene cup in the 250-mL beaker.
- Mount the temperature sensor on the ring stand and set it into the cup about half an inch from the bottom.
- Why do you place the cup in a beaker?



The air trapped between the wall of the cup and the beaker serves as further heat insulation to lower the heat loss.

## Lab 13: Enthalpy of a Chemical Reaction

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### Collect Data

6.  Perform the three reactions in the calorimeter using the reactants shown in Table 2 following the steps below.

Table 2: Reactant volumes

Reaction	Solution 1	Solution 2
1	50.0 mL 2.00 M HCl	50.0 mL 2.00 M NaOH
2	50.0 mL 2.00 M NaOH	50.0 mL 2.00 M NH <sub>4</sub> Cl
3	50.0 mL 2.00 M HCl	50.0 mL 2.00 M NH <sub>3</sub>

For each reaction,

- Measure Solution 1 and transfer it into the cup.
- Rinse the graduated cylinder with deionized water.
- Measure 50 mL of Solution 2.
- Start recording data.  $\diamond^{(6.2)}$
- When the temperature readings stabilize, quickly add Solution 2 to the cup. Continue monitoring the temperature until the temperature starts to drop, then stop recording data.  $\diamond^{(6.2)}$
- Dispose of the solutions properly, wash the cup and graduated cylinder, and rinse them with deionized water.

**Note:** Please handle the ammonia solution with extra care. Allow the solution as little as possible exposure to air and make sure you do not breathe near the ammonia solution. If ammonia irritates your nose or eyes, go where there is fresh air.

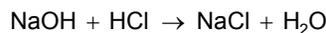
7.  Record the initial and maximum temperature for each data run in Table 3 in the Data Analysis section.  $\diamond^{(7.2.1)}$
8.  Save your experiment and clean up according to your teacher's instructions  $\diamond^{(11.1)}$

## Data Analysis

Use the following value for the heat capacity of the calorimeter:

$$C = (100.0\text{g})\left(4.18\frac{\text{J}}{\text{g}\cdot^{\circ}\text{C}}\right) + \left(15.0\frac{\text{J}}{^{\circ}\text{C}}\right) = 433\frac{\text{J}}{^{\circ}\text{C}}$$

1.  Write the equations for the three reactions you conducted.



2.  Calculate the change of temperature due to each reaction. Record your results in Table 3.

Calculating the change of temperature for Reaction 1:

$$31.8\text{ }^{\circ}\text{C} - 21.9\text{ }^{\circ}\text{C} = 9.9\text{ }^{\circ}\text{C}$$

3.  Calculate the change of heat based on the change of temperature and the heat capacity of the reactor for each reaction. Record your results in Table 3.

Calculating the change of heat for Reaction 1

$$q = C\Delta T = \left(433\frac{\text{J}}{^{\circ}\text{C}}\right)(9.9\text{ }^{\circ}\text{C}) = 4.3 \times 10^3 \text{ J}$$

4.  Calculate the amount of each reactant present in each reaction. Record your results in Table 3.

Since the volume and the concentration of the two solutions were the same and the stoichiometric ratio between them is 1:1, the amount of the two reactants is the same. They are present in stoichiometric ratio.

For Reaction 1:

$$\left(2.00\frac{\text{mol}}{\text{L}}\right)(0.0500\text{ L}) = 0.100\text{ mol NaOH and HCl}$$

5.  Calculate the molar change of heat for each reaction. Record your results in Table 3.

For Reaction 1:

$$\left(\frac{4.3 \times 10^3 \text{ J}}{0.100 \text{ mol}}\right) = 4.3 \times 10^4 \frac{\text{J}}{\text{mol}}$$

## Lab 13: Enthalpy of a Chemical Reaction

Table 3: Experimental and calculated data to determine the heat of reaction

Parameters	Reaction 1	Reaction 2	Reaction 3
Initial temperature (°C)	21.9	21.8	21.8
Maximum temperature (°C)	31.8	22.8	31.4
Change of temperature (°C)	9.9	1.0	9.0
$q$ (J)	$4.3 \times 10^3$	$4.3 \times 10^2$	$3.9 \times 10^3$
Amount of reactant (mol)	0.100	0.100	0.100
Molar heat of reaction (J)	$4.3 \times 10^4$	$4.3 \times 10^3$	$3.9 \times 10^4$

**6. Verify that the heat of Reaction 3 can be calculated from Reactions 1 and 2 the way you proposed earlier.**

Answers will depend on student's results. In the sample data set:

$$\Delta H_3 = \Delta H_1 - \Delta H_2 = (4.3 \times 10^4 \text{ J}) - (4.3 \times 10^3 \text{ J}) = 3.9 \times 10^4 \text{ J}$$

The experimental value was  $3.9 \times 10^4$  J.

### Analysis Questions

**1. Identify potential sources of experimental error and propose solutions.**

Imperfect heat insulation results in heat loss and, as a result, experimental error. To minimize heat loss, use a lid on the cup or use two cups (one placed inside the other) to double the wall thickness.

**2. All solutions used the heat capacity of water ( $4.18 \text{ J g}^{-1} \text{ }^\circ\text{C}^{-1}$ ) to calculate the heat capacity of the calorimeter. Did this introduce an error? Explain!**

The error introduced is negligible because the heat capacity of diluted solutions is very close to the heat capacity of pure water.

**3. Suppose all of the temperature readings were off by  $0.5 \text{ }^\circ\text{C}$ . How would that change your results?**

A consistent deviation in temperature readings would not have any effect on the results because the difference between temperature readings is used to calculate the heat of reaction. That difference would be the same.

## Synthesis Questions

Use available resources to help you answer the following questions.

**1.** Use Hess's Law to obtain  $\Delta H_4$  for the following reactions.



Reversing the third reaction yields the right combination:

$$\Delta H_4 = \Delta H_1 + \Delta H_2 - \Delta H_3.$$

**2.** Another way of performing calorimetric experiments is in a “bomb” calorimeter. A sample is placed in a “bomb,” which is a stainless steel container with heavy duty walls to withstand high pressure. It is pressurized with  $\text{O}_2$  and the sample is ignited. The temperature increase is monitored. From this the heat of reaction can be obtained. Consider the following:

A food sample (2.200 g) was burnt in an experiment with a bomb calorimeter. The heat capacity of the calorimeter was  $5.754 \text{ kJ}^\circ\text{C}$ . The temperature changed from  $24.56^\circ\text{C}$  to  $32.33^\circ\text{C}$ . What is the heat of reaction per gram of this food sample?

$$\Delta H = \frac{-\left(5.754 \frac{\text{kJ}}{^\circ\text{C}}\right)(32.33^\circ\text{C} - 24.56^\circ\text{C})}{(2.200 \text{ g})} = -20.3 \frac{\text{kJ}}{\text{g}}$$

## Multiple Choice Questions

Select the best answer or completion to each of the questions or incomplete statements below.

**1.** Hess's Law:

- A.** Gives the heat of reaction for a reaction obtained by combining the heat of reaction of other reactions.
- B.** Provides evidence that the heat of reaction is independent of the path of the reaction.
- C.** Can be used to obtain the heat of reaction for reactions not possible to perform.
- D.** All of the above.

## Lab 13: Enthalpy of a Chemical Reaction

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**2.** The heat capacity of the calorimeter is:

- A.** The amount of heat necessary to increase the temperature of the calorimeter, including the solutions, 1 °C.
- B.** The same as the heat of reaction per mole of reactant.
- C.** Can be obtained from the heat of reaction of the combined reactions.
- D.** Is usually negligible.

**3.** The heat change that accompanies the dissolution processes can be measured with calorimetry as well. In an experiment, 5.00 g of  $\text{NH}_4\text{NO}_3$  was dissolved in 50 mL of water in a polystyrene cup calorimeter. The temperature dropped from 23.50 °C to 17.57 °C. What is the heat of reaction for the dissolution of  $\text{NH}_4\text{NO}_3$ ?

- A.**  $\Delta H = + 1330 \text{ J/mol}$ .
- B.**  $\Delta H = + 21.3 \text{ kJ/mol}$ .
- C.**  $\Delta H = - 21.3 \text{ kJ/mol}$ .
- D.**  $\Delta H = + 2130 \text{ J/mol}$ .

### Extended Inquiry Suggestions

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Students can perform the experiment presented in the Pre-Lab Activity section in a calorimeter. This experiment demonstrates that the heat of reaction for unmeasurable reactions can be obtained.

## Lab 14a: Separation and Analysis of Cations

### Objectives

Students use chemical properties to qualitatively classify and identify the cations present in a solution.

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Organizing and tracking the results of multiple reactions
- ◆ Performing basic laboratory separation processes

### Time Requirement

◆ Preparation time	50 minutes
◆ Pre-lab discussion and activity	30 minutes
◆ Lab activity	90 minutes

### Materials and Equipment

#### *For each student or group:*

- |                            |   |
|----------------------------|---|
| ◆ Test tube, 10-mL (10)    | ◆ 6 M Sodium hydroxide (NaOH), 20 mL <sup>1</sup>   |
| ◆ Test tube rack           | ◆ 6 M Ammonia (NH <sub>3</sub> ), 20 mL <sup>2</sup>                                      |
| ◆ Pipet, graduated, 10-mL  | ◆ 0.1 M Potassium chromate (K <sub>2</sub> CrO <sub>4</sub> ), 20 mL <sup>3</sup>         |
| ◆ Rubber bulb              | ◆ 1% Aluminon dye, 2 mL <sup>4</sup>  |
| ◆ Pipet, plastic (7), 1-mL | ◆ 6 M Hydrochloric acid (HCl), 20 mL <sup>5</sup>   |
| ◆ Centrifuge               | ◆ Dimethylglyoxime (DMG) reagent, 5 drops <sup>6</sup>                                    |
| ◆ Beaker, 250-mL           | ◆ 0.2 M Potassium ferrocyanide (K <sub>4</sub> [Fe(CN) <sub>6</sub> ]), 2 mL <sup>7</sup> |
| ◆ Evaporating dish         | ◆ 3 M Sulfuric acid (H <sub>2</sub> SO <sub>4</sub> ), 3 mL <sup>8</sup>                  |
| ◆ Stirring rod             | ◆ 3 % Hydrogen peroxide (H <sub>2</sub> O <sub>2</sub> ), 2 mL <sup>9</sup>               |
| ◆ Hot plate                | ◆ Unknown cation solution, 20 mL <sup>10</sup>  |
| ◆ Litmus paper (10)        | ◆ Deionized water, 5 mL   |
| ◆ pH paper (1 roll)        | ◆ Marking pen   |

<sup>1-8</sup>To prepare the solutions, refer to the Lab Preparation section.

<sup>6</sup>This reagent is available premade from chemical suppliers

<sup>9</sup>3% hydrogen peroxide is readily available in pharmacies.

<sup>10</sup>The “unknown” cation solution can contain 2 to 4 of the cations. The unknown solutions can be prepared by combining any 2 to 4 of the cation solutions used in this lab. Refer to the Lab Preparation section for the list of cation solutions.

## **Concepts Students Should Already Know**

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Students should be familiar with the following concepts:

- ◆ Balancing chemical equations
- ◆ Acid-base reactions
- ◆ Stoichiometry of chemical reactions

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 14b: Analysis of Anions
- ◆ Lab 15b: Analysis of a Coordination Compound

## **Using Your Data Collection System**

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

As this lab activity does not use a data collection system, no Tech Tips (indicated by the symbol "◆" and a superscripted number following a step) are needed.

## **Background**

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The primary goal of a qualitative analysis is to identify the substances present in a mixture. In the qualitative analysis procedure, chemical properties of an unknown substance are determined by systematically reacting the unknown with a number of different reagents. By knowing what a particular reaction will produce if a specific ion is present, the ions in the solution can be identified.

In some cases the products of a reaction cannot definitively confirm the presence of a specific ion. In these cases it is necessary to perform tests for a particular ion as well as to separate the components of the mixture.

## **Pre-Lab Activity**

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### ***Setting the stage for the activity***

In this exercise you will be given a solution containing 2 to 4 unknown cations. The analysis of cations uses a flowchart of reactions in the procedure. The flowchart is given at the end of the Procedure section.

It is important to perform the reactions in the procedure with great care so that complete precipitation and separation occur. Otherwise, unreacted ions may interfere with subsequent steps in the procedure and cause false positive results.

### Example calculation to try

The analysis of a sample gave the following results. For procedural details refer to the Collect Data section.

Table 1: Identifying cations in a solution

Step	Action	Observation	Possible explanation
1	Add NaOH solution (Procedure 1)	No ammonia production	$\text{NH}_4^+$ is ruled out.
2	Add 6M HCl (Procedure 2)	White precipitate	Potential presence of $\text{Ag}^+$ or $\text{Pb}^{2+}$ or both
3	Separate the precipitate from the supernatant. Add deionized water to the precipitate and heated it. (Procedure 3)	No change	The precipitate is $\text{AgCl}$ , which confirms the presence of $\text{Ag}^+$ .
4	Add NaOH solution (in excess) to the supernatant from Step 3. Separate the precipitate from the supernatant. (Procedure 6)	Green precipitate	The presence of $\text{Ni}^{2+}$ is likely because of the color; $\text{Mn}^{2+}$ , $\text{Fe}^{3+}$ , $\text{Al}^{3+}$ are possible
5	Add $\text{H}_2\text{SO}_4$ in excess to the supernatant from Step 4. (Procedure 11)	No change	$\text{Al}^{3+}$ can be ruled out.
6	Add $\text{NH}_3$ solution to the precipitate from Step 4. (Procedure 7)	Blue solution forms. All precipitate dissolves.	The presence of $\text{Ni}^{2+}$ is confirmed, $\text{Mn}^{2+}$ and $\text{Fe}^{3+}$ are ruled out.

The analysis confirmed the presence of  $\text{Ag}^+$  and  $\text{Ni}^{2+}$  ions.

#### 1. How would the result be different if, in Step 3, the precipitate did dissolve?

The dissolving precipitate in Step 3 would indicate the presence of  $\text{Pb}^{2+}$ .

#### 2. Why was there no need to check for $\text{Mn}^{2+}$ and $\text{Fe}^{3+}$ after Step 6?

If either  $\text{Mn}^{2+}$  or  $\text{Fe}^{3+}$  were present, the precipitate would not have dissolved completely in the  $\text{NH}_3$  solution.

## Lab Preparation

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These are the materials and equipment to set up prior to the lab:

### Test solutions

1. **6 M NaOH:** Dissolve 240 g of NaOH in water in a 1-L volumetric flask and fill it to the mark.
2. **6 M NH<sub>3</sub>:** Dissolve 340 mL of a 30% NH<sub>3</sub> solution in water in a 1-L volumetric flask and fill it to the mark.
3. **0.1 M K<sub>2</sub>CrO<sub>4</sub>:** Dissolve 19.4 g of K<sub>2</sub>CrO<sub>4</sub> in water in a 1-L volumetric flask and fill it to the mark.
4. **Aluminon dye:** Dissolve 4.734 g of Aluminon dye (aurin tricarboxylic acid) in water in a 100 mL volumetric flask and fill it to the mark to make a 1% solution.
5. **6 M HCl:** Under a hood, slowly add 500 mL of 36% HCl solution to about 400 mL of water in a 1-L volumetric flask and fill it to the mark.
6. **Dimethylglyoxime reagent:** Dissolve 1 g of dimethylglyoxime in ethanol in a 100-mL volumetric flask and fill it to the mark.
7. **0.2 M K<sub>4</sub>[Fe(CN)<sub>6</sub>]:** Dissolve 84.5 g of K<sub>4</sub>[Fe(CN)<sub>6</sub>] in water in a 1-L volumetric flask and fill it to the mark.
8. **3 M H<sub>2</sub>SO<sub>4</sub>:** Combine 167 mL of 98% H<sub>2</sub>SO<sub>4</sub> solution *slowly* with at least 500 mL of water in a 1-L volumetric flask and fill it to the mark.
9. **3% H<sub>2</sub>O<sub>2</sub>:** Commercially available 3% hydrogen peroxide can be used directly.

### For the unknown cation solution

10. **0.1 M AlCl<sub>3</sub>:** Dissolve 24.1 g of AlCl<sub>3</sub> · 6H<sub>2</sub>O in water in a 1-L volumetric flask and fill it to the mark.
11. **0.1 M NiCl<sub>2</sub>:** Dissolve 23.8 g of NiCl<sub>2</sub> · 6H<sub>2</sub>O in water in a 1-L volumetric flask and fill it to the mark.
12. **0.1 M Pb(NO<sub>3</sub>)<sub>2</sub>:** Dissolve 33.1 g of Pb(NO<sub>3</sub>)<sub>2</sub> in water in a 1-L volumetric flask and fill it to the mark.
13. **0.1 M AgNO<sub>3</sub>:** Dissolve 17.0 g of AgNO<sub>3</sub> in water in a 1-L volumetric flask and fill it to the mark.
14. **0.1 M MnSO<sub>4</sub>:** Dissolve 22.3 g of MnSO<sub>4</sub> · 4H<sub>2</sub>O in water in a 1-L volumetric flask and fill it to the mark.
15. **0.1 M (NH<sub>4</sub>)<sub>2</sub>Fe(SO<sub>4</sub>)<sub>2</sub>:** Dissolve 39.2 g of (NH<sub>4</sub>)<sub>2</sub>Fe(SO<sub>4</sub>)<sub>2</sub> · 6H<sub>2</sub>O in water in a 1-L volumetric flask and fill it to the mark.

## Safety

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Follow all standard laboratory procedures.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

1	2	3	4
Prepare 10 clean test tubes and the hot water bath.	Perform the prescribed procedures to analyze the cations in the unknown sample. Record your observations	Based on your results, complete the balanced chemical equations.	Identify the cations in your unknown sample.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** This activity does not use a data collection system, so there are no Tech Tips (indicated by the symbol "♦" and a superscripted number following a step).

**You should have a solution containing 2 to 4 unknown cations. It is important to perform the reactions in the procedure so complete precipitation and separation occur. Otherwise, unreacted ions may interfere with subsequent steps in the procedure and cause false positive results.**

### Set Up

- Make sure you have at least 10 clean test tubes in a test tube rack.
- Read the various procedures in Table 2 and devise a method to organize and label the test tubes and other test materials.

**Note:** Label the disposable pipets before they are used, to avoid contamination.

- Record the number of your unknown solution: \_\_\_\_\_.
- Prepare a hot water bath (approximately 80 °C).

### Collect Data

- Perform the reactions with your unknown solution according to Table 2. You can also refer to the Cation Flowchart at the end of this section.

## Lab 14a: Separation and Analysis of Cations

6.  After each procedure, record your observations of each test performed in Table 3 in the Data Analysis section.

**Note:** Use a fresh pipet for each procedure

Table 2: Procedural steps to analyze cations

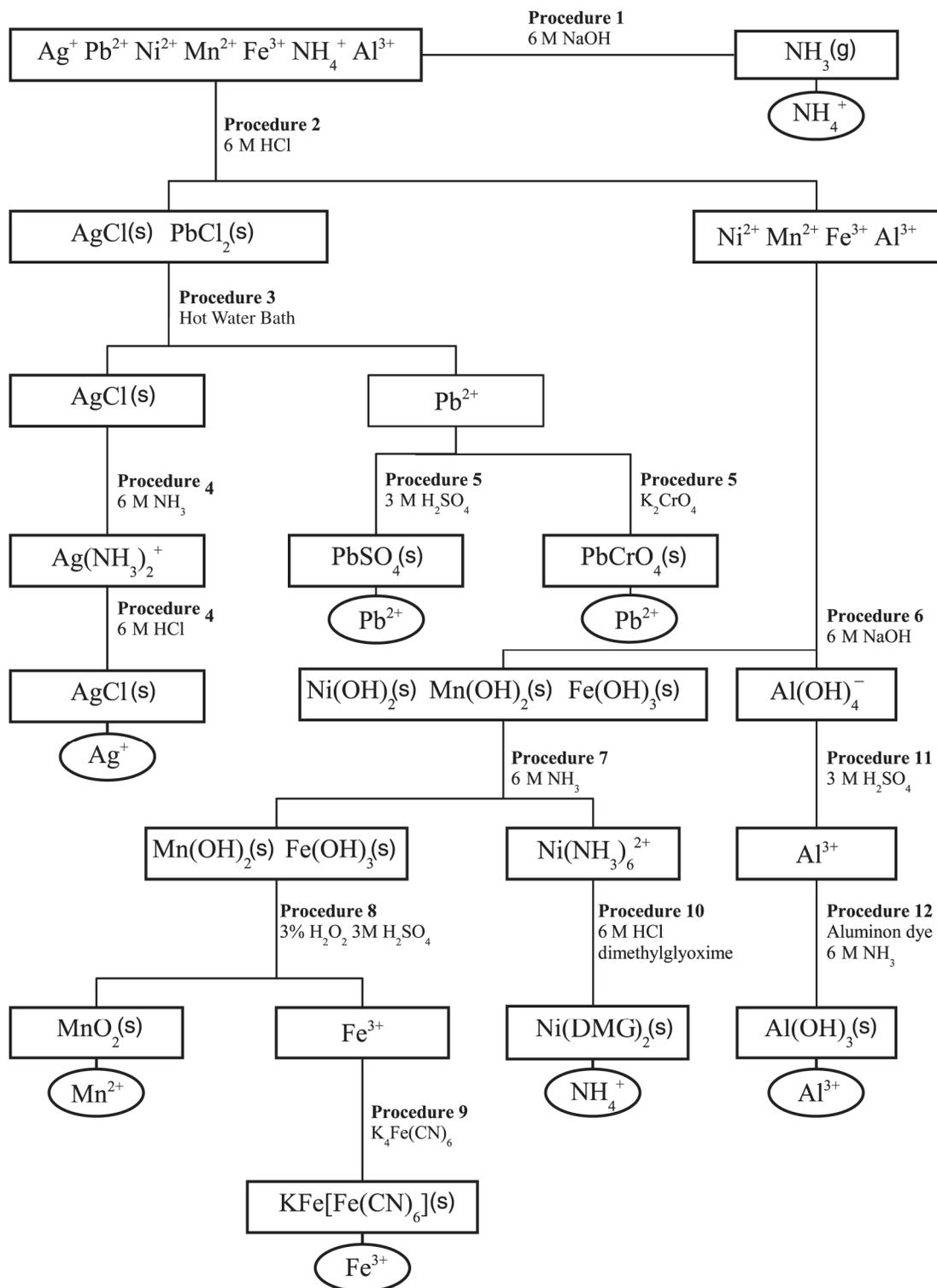
<b>Procedure 1:</b> Detection of ammonium ion	<ul style="list-style-type: none"><li><input type="checkbox"/> Measure 1 mL of the unknown solution into an evaporating dish.</li><li><input type="checkbox"/> Add approximately 2 mL of 6 M NaOH with a plastic pipet and cover with a watch glass with a piece of moist red litmus paper attached.</li><li><input type="checkbox"/> Observe the litmus paper for a color change from red to blue, indicating the presence of ammonia.</li></ul>
<b>Procedure 2:</b> Separation of silver and lead as chlorides	<ul style="list-style-type: none"><li><input type="checkbox"/> With a clean pipet transfer 2 mL of the unknown solution to a test tube.</li><li><input type="checkbox"/> Add 2 to 3 drops of 6 M HCl. If a precipitate forms, continue adding 6 M HCl solution drop by drop while stirring until no more precipitate forms. If no precipitate forms, save the solution for <i>Procedure 6</i>.</li><li><input type="checkbox"/> Centrifuge the mixture in the test tube and then add one drop of 6 M HCl.</li><li><input type="checkbox"/> If no additional precipitate appears, pour the supernatant into another test tube.</li><li><input type="checkbox"/> If more precipitate forms, repeat the last two steps until no new precipitate forms.</li><li><input type="checkbox"/> Label the samples and save the supernatant for <i>Procedure 6</i>. Use the precipitate for <i>Procedure 3</i>.</li></ul>
<b>Procedure 3:</b> Separation of lead ion from silver ion	<p><i>Continue from Procedure 2.</i></p> <ul style="list-style-type: none"><li><input type="checkbox"/> With a clean pipet add 3 to 4 mL of deionized water to the precipitate from <i>Procedure 2</i>.</li><li><input type="checkbox"/> Heat the test tube, swirling it while in a hot water bath for several minutes while stirring.</li><li><input type="checkbox"/> Centrifuge the test tube to separate the supernatant from any remaining solid.</li><li><input type="checkbox"/> Save the supernatant for use in <i>Procedure 5</i>. Use the precipitate for <i>Procedure 4</i>.</li></ul>

<p><b>Procedure 4:</b> Confirmation of silver ion</p>	<p><i>Continue from Procedure 3.</i></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> Add 10 drops of 6 M ammonia solution to the precipitate from <i>Procedure 3</i> and stir.</li> <li><input type="checkbox"/> Add 6 M HCl drop by drop until the precipitate reappears or litmus paper indicates the solution is acidic.</li> </ul>
<p><b>Procedure 5:</b> Confirmation of lead ion</p>	<p><i>Continue from Procedure 3.</i></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> If the solution from <i>Procedure 3</i> contains any precipitation, reheat it in the water bath until the precipitate is completely dissolved.</li> <li><input type="checkbox"/> Divide the sample into two test tubes. Add several drops of 3 M H<sub>2</sub>SO<sub>4</sub> to one of the test tubes.</li> <li><input type="checkbox"/> Add several drops of potassium chromate to the second test tube and stir.</li> </ul>
<p><b>Procedure 6:</b> Precipitation of nickel, manganese and iron ions as hydroxides</p>	<p><i>Continue from Procedure 2.</i></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> Add 6 M NaOH to the supernatant from <i>Procedure 2</i> until precipitation is complete.</li> <li><input type="checkbox"/> Add 0.5 mL more of the NaOH and stir.</li> <li><input type="checkbox"/> Separate the supernatant from any precipitate. Save the supernatant for <i>Procedure 11</i>. Use the precipitate for <i>Procedure 7</i>.</li> </ul>
<p><b>Procedure 7:</b> Separation of nickel ion from manganese and iron ion</p>	<p><i>Continue from Procedure 6.</i></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> Add 3 mL of 6 M ammonia and stir well.</li> <li><input type="checkbox"/> Separate the supernatant from any precipitate. Save the supernatant for <i>Procedure 10</i>. Use the precipitate for <i>Procedure 8</i>.</li> </ul>
<p><b>Procedure 8:</b> Confirmation of manganese ion</p>	<p><i>Continue from Procedure 7.</i></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> Add 1 mL of 3% hydrogen peroxide solution to the precipitate from <i>Procedure 7</i>.</li> <li><input type="checkbox"/> Stir, then heat the sample in the water bath until no more gas is generated.</li> <li><input type="checkbox"/> Add 3 M H<sub>2</sub>SO<sub>4</sub> by drops until the mixture is acidic.</li> <li><input type="checkbox"/> Separate the supernatant from any precipitate and use it for <i>Procedure 9</i></li> </ul>

## Lab 14a: Separation and Analysis of Cations

<b>Procedure 9:</b> Confirmation of iron ion	<i>Continue from Procedure 8.</i> <input type="checkbox"/> Add two drops of 0.2 M $\text{K}_4[\text{Fe}(\text{CN})_6]$ to the solution from <i>Procedure 8</i> .
<b>Procedure 10:</b> Confirmation of nickel ion	<i>Continue from Procedure 7.</i> <input type="checkbox"/> Add 1 drop of 6 M HCl. Then add 5 drops of dimethylglyoxime reagent to the solution from <i>Procedure 7</i> .
<b>Procedure 11:</b> Detection of aluminum ion	<i>Continue from Procedure 6.</i> <input type="checkbox"/> Neutralize the solution from <i>Procedure 6</i> by adding 3 M $\text{H}_2\text{SO}_4$ . Use the pH paper to test the pH after adding each drop. <input type="checkbox"/> Add 2 more drops of 3M $\text{H}_2\text{SO}_4$ after obtaining a neutral pH. Use the supernatant for <i>Procedure 12</i> .
<b>Procedure 12:</b> Confirmation of aluminum ion	<i>Continue from Procedure 11.</i> <input type="checkbox"/> Add 2 drops of aluminon dye to the solution from <i>Procedure 11</i> . <input type="checkbox"/> Add the 6 M ammonia by drops while stirring. Use litmus paper to detect when the solution is basic and then stop adding the ammonia.

# Cation Flowchart



**Data Analysis**

1.  Enter the equations and identified cations in Table 3 that conform to your observations.

Table 3: Complete the table

Procedure	Observation	Proposed Equations	Identified Cations
1	No change in color of litmus paper.	$\text{Ag}^+(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Ag}_2\text{O}(\text{s}) + \text{H}_2\text{O}$ $\text{Ni}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Ni}(\text{OH})_2(\text{s})$ $\text{Cu}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Cu}(\text{OH})_2(\text{s})$ $\text{Mn}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Mn}(\text{OH})_2(\text{s})$ $\text{Pb}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Pb}(\text{OH})_2(\text{s})$ $\text{Fe}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Fe}(\text{OH})_3(\text{s})$ $\text{Al}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Al}(\text{OH})_3(\text{s})$	$\text{Ag}^+$ , $\text{Pb}^{2+}$ , $\text{Ni}^{2+}$ , $\text{Mn}^{2+}$ , $\text{Fe}^{3+}$ , $\text{Al}^{3+}$
	Change in color	$\text{NH}_4^+(\text{aq}) + \text{OH}^-(\text{aq}) \rightarrow \text{NH}_3(\text{g}) + \text{H}_2\text{O}$	$\text{NH}_4^+$
2	White precipitate	$\text{Ag}^+(\text{aq}) + \text{Cl}^-(\text{aq}) \rightarrow \text{AgCl}(\text{s})$ $\text{Pb}^{2+}(\text{aq}) + 2\text{Cl}^-(\text{aq}) \rightarrow \text{PbCl}_2(\text{s})$	$\text{Ag}^+$ , $\text{Pb}^{2+}$
	No precipitate		$\text{Ni}^{2+}$ , $\text{Mn}^{2+}$ , $\text{Fe}^{3+}$ , $\text{Al}^{3+}$
3	Solid did not return into solution		$\text{Ag}^+$
	Solid returned into solution	$\text{PbCl}_2(\text{s}) \rightarrow \text{Pb}^{2+}(\text{aq}) + 2\text{Cl}^-(\text{aq})$	$\text{Pb}^{2+}$
4	Precipitate disappears	$\text{AgCl}(\text{s}) + 2\text{NH}_3(\text{aq}) \rightarrow \text{Ag}(\text{NH}_3)_2^+(\text{aq}) + \text{Cl}^-(\text{aq})$	$\text{Ag}^+$
	Precipitate reappears	$\text{Ag}(\text{NH}_3)_2^+(\text{aq}) + 2\text{H}^+(\text{aq}) + \text{Cl}^-(\text{aq}) \rightarrow$ $\text{AgCl}(\text{s}) + 2\text{NH}_4^+$	
5	Precipitate forms	$\text{Pb}^{2+}(\text{aq}) + \text{SO}_4^{2-}(\text{aq}) \rightarrow \text{PbSO}_4(\text{s})$	$\text{Pb}^{2+}$
	Precipitate forms	$\text{Pb}^{2+}(\text{aq}) + \text{CrO}_4^{2-}(\text{aq}) \rightarrow \text{PbCrO}_4(\text{s})$	

Procedure	Observation	Proposed Equations	Identified Cations
6	Precipitate forms	$\text{Ag}^+(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Ag}_2\text{O}(\text{s}) + \text{H}_2\text{O}$ $\text{Ni}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Ni}(\text{OH})_2(\text{s})$ $\text{Cu}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Cu}(\text{OH})_2(\text{s})$ $\text{Mn}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Mn}(\text{OH})_2(\text{s})$ $\text{Pb}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Pb}(\text{OH})_2(\text{s})$ $\text{Fe}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Fe}(\text{OH})_3(\text{s})$ $\text{Al}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Al}(\text{OH})_3(\text{s})$	$\text{Ni}^{2+}$ , $\text{Mn}^{2+}$ , $\text{Fe}^{3+}$
	Precipitate dissolves	$\text{Ag}^+(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Ag}_2\text{O}(\text{s}) + \text{H}_2\text{O}$ $\text{Ni}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Ni}(\text{OH})_2(\text{s})$ $\text{Cu}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Cu}(\text{OH})_2(\text{s})$ $\text{Mn}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Mn}(\text{OH})_2(\text{s})$ $\text{Pb}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{Pb}(\text{OH})_2(\text{s})$ $\text{Fe}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Fe}(\text{OH})_3(\text{s})$ $\text{Al}^{3+}(\text{aq}) + 3\text{OH}^-(\text{aq}) \rightarrow \text{Al}(\text{OH})_3(\text{s})$	$\text{Al}^{3+}$
7	Precipitate remains		$\text{Mn}^{2+}$ , $\text{Fe}^{3+}$
	Precipitate disappears	$\text{Ni}^{2+}(\text{aq}) + 6\text{NH}_3 \rightarrow \text{Ni}(\text{NH}_3)_6^{2+}(\text{aq})$	$\text{Ni}^{2+}$
8	Precipitate remains	$\text{Mn}(\text{OH})_2(\text{s}) + \text{H}_2\text{O}_2 \rightarrow \text{MnO}_2(\text{s}) + 2\text{H}_2\text{O}$	$\text{Mn}^{2+}$
	Precipitate disappears	$\text{Fe}(\text{OH})_3(\text{s}) + 3\text{H}^+(\text{aq}) \rightarrow \text{Fe}^{3+}(\text{aq}) + 3\text{H}_2\text{O}$	$\text{Fe}^{3+}$
9	Precipitate forms	$4\text{Fe}^{3+}(\text{aq}) + 3[\text{Fe}(\text{CN})_6]^{4-}(\text{aq}) \rightarrow$ $\text{Fe}_4[\text{Fe}(\text{CN})_6]_3(\text{s})$	$\text{Fe}^{3+}$
10	Precipitate forms	$\text{Ni}(\text{NH}_3)_6^{2+}(\text{aq}) + 2\text{DMG} \rightarrow$ $\text{Ni}(\text{DMG})_2(\text{s}) + 6\text{NH}_3(\text{aq})$	$\text{Ni}^{2+}$
11	No precipitate	$\text{Al}(\text{OH})_4^-(\text{aq}) + 4\text{H}^+(\text{aq}) \rightarrow$ $\text{Al}^{3+}(\text{aq}) + 4\text{H}_2\text{O}$	$\text{Al}^{3+}$
12	Precipitate forms		$\text{Al}^{3+}$

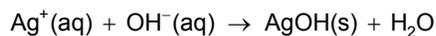
## Analysis Questions

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**1.** If you had  $\text{Mn}^{2+}$  ions in your unknown solution, the reaction mixture with NaOH would eventually turn brown. Propose an explanation for that phenomenon (Hint: see Procedure 8).

The  $\text{O}_2$  in the air can oxidize  $\text{Mn}^{2+}$  to manganese ions with a higher oxidation number, which are mostly brown.

**2.** NaOH reacts with  $\text{Ag}^+$  to form  $\text{Ag}_2\text{O}$ . The reaction is based on the fact that one of the products of the expected exchange reaction is not stable and loses water. Propose an equation for the exchange reaction and an equation for one of the products losing water.



**3.** The reaction mixture described above has a brown precipitate. What do you think it is?

$\text{Ag}_2\text{O}$

## Synthesis Questions

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Use available resources to help you answer the following questions.

**1.** How would you use the reactions described in Questions 2 and 3 above to identify  $\text{Ag}^+$  and distinguish it from  $\text{Pb}^{2+}$ ?

The color of the precipitate of  $\text{Ag}^+$  with NaOH is unique. Using NaOH in Procedure 2 would reveal the presence of  $\text{Ag}^+$ .

**2.** The reaction of a sample solution with NaOH forms a white precipitate. Which ions could be present? (Hint: one possible ion could have been present in Procedure 6, the other could not have.) You may have to perform a reaction to answer this question.

$\text{Al}^{3+}$  ion and  $\text{Pb}^{2+}$  ion.

**3.** Propose a reaction to distinguish between the ions named in the above question.

Excess NaOH would dissolve the  $\text{Al}(\text{OH})_3$  precipitate (Procedure 6) but not the  $\text{Pb}(\text{OH})_2$ . Also,  $\text{Pb}^{2+}$  forms a precipitate when reacted with  $\text{H}_2\text{SO}_4$  (Procedure 5) and  $\text{Al}^{3+}$  does not.

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**Multiple Choice Questions**

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Select the best answer or completion to each of the questions or incomplete statements below.

1. A white precipitate with NaOH could indicate the presence of:
  - A.  $\text{Al}^{3+}$
  - B.  $\text{Fe}^{3+}$
  - C.  $\text{Mn}^{2+}$
  - D. None of these
2. A yellow precipitate can be:
  - A. AgCl
  - B.  $\text{Ni}(\text{OH})_2$
  - C.  $\text{PbCrO}_4$
  - D.  $\text{PbCl}_2$
3. A green solution forms a precipitate when mixed with NaOH. The solution must:
  - A. React with excess NaOH.
  - B. Not react with  $\text{NH}_3$  solution.
  - C. Give precipitate with  $\text{H}_2\text{SO}_4$ .
  - D. Give a red precipitate with dimethylglyoxime.

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**Extended Inquiry Suggestions**

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You can challenge your students by giving them a number of unknown solutions in test tubes (TT) and have them perform reactions among only those solutions, without using any other reagents. Allow them to identify the content of the test tubes using a reaction matrix and what they have learned in this experiment. An example of a reaction matrix is shown below.

The following solutions are used:  $\text{AgNO}_3$ ,  $\text{Na}_2\text{SO}_4$ , HCl,  $\text{Pb}(\text{NO}_3)_2$ ,  $\text{Al}(\text{NO}_3)_3$ , NaOH and put in test tubes numbered "1" through "6." (The dash indicates there was no change when the solutions were combined.)

## Lab 14a: Separation and Analysis of Cations

Reaction Matrix

TT	1	2	3	4	5	6
1		White precipitate, does not dissolve with heat.	Brown precipitate	—	—	—
2			—	—	White precipitate that dissolves with heat.	—
3				White precipitate	White precipitate	—
4					—	—
5						White precipitate
6						

The following strategy can be followed:

1. A brown precipitate forms when solution from test tubes #1 and #3 are mixed, resulting in  $\text{Ag}_2\text{O}$  (this has a characteristic precipitate). Therefore, #1 and #3 are  $\text{AgNO}_3$  and  $\text{NaOH}$ .
2. The precipitate that dissolves when heated is  $\text{PbCl}_2$ . Therefore, #2 and #5 are  $\text{HCl}$  and  $\text{Pb}(\text{NO}_3)_2$ .
3. To identify the specific test tube, first assume #3 is  $\text{AgNO}_3$ . If #3 is  $\text{AgNO}_3$  and #2 is  $\text{HCl}$ , then mixing #3 and #2 would yield  $\text{AgCl}$ , a white precipitate. This did not happen.
4. If #3 is  $\text{AgNO}_3$  then #1 has to be  $\text{NaOH}$ .
5. If #3 is  $\text{AgNO}_3$  then #2 has to be  $\text{Pb}(\text{NO}_3)_2$  (no reaction) and #5 has to be  $\text{HCl}$  (white precipitate).
6. The results of steps 3 to 5 cannot be possible since they resulted in #1 being  $\text{NaOH}$  and #2 being  $\text{HCl}$ . These mixed would not yield a visible reaction. This is inconsistent with our observations (there was a white precipitate between #1 and #2). Therefore, our assumption that #3 is  $\text{AgNO}_3$  is wrong.
7. If #1 is  $\text{AgNO}_3$ , then #3 has to be  $\text{NaOH}$ .
8. If #1 is  $\text{AgNO}_3$  then #5 has to be  $\text{Pb}(\text{NO}_3)_2$  (no reaction) and #2 has to be  $\text{HCl}$  (#5 mixed with #2 formed a white precipitate).
9. #5 ( $\text{Pb}(\text{NO}_3)_2$ ) reacts with #6 but not with #4. Therefore, #6 has to be  $\text{Na}_2\text{SO}_4$  ( $\text{PbSO}_4$  precipitate) and #4 has to be  $\text{Al}(\text{NO}_3)_3$ .

10. #4 ( $\text{Al}(\text{NO}_3)_3$ ) reacts with #3 ( $\text{NaOH}$ ) yielding white  $\text{Al}(\text{OH})_3$  precipitate. This is consistent with our results (and can be further confirmed by adding an excess of  $\text{NaOH}$ , which would dissolve the  $\text{Al}(\text{OH})_3$ ).

Therefore the solutions are as follows:

<b>Test Tube</b>	<b>Solution</b>
1	$\text{AgNO}_3$
2	$\text{HCl}$
3	$\text{NaOH}$
4	$\text{Al}(\text{NO}_3)_3$
5	$\text{Pb}(\text{NO}_3)_2$
6	$\text{Na}_2\text{SO}_4$



## Lab 14b: Analysis of Anions

### Objectives

Students analyze solutions of known anions and apply the knowledge gained to identify the anions in an unknown solution.

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Comparing the results of test tube reactions of known anions with the results of the solution containing unknown anions.
- ◆ Performing basic laboratory separation processes.

### Time Requirement

◆ Preparation time	60 minutes
◆ Pre-lab discussion and activity	50 minutes
◆ Lab activity	60 minutes

### Materials and Equipment

#### *For each student or group:*

- |   |  |
|---|--|
| ◆ Test tube, 10-mL (13)   | ◆ 0.2 M Barium nitrate ( $\text{Ba}(\text{NO}_3)_2$ ), 5 mL <sup>6</sup> |
| ◆ Test tube rack  | ◆ Saturated iron(II) sulfate ( $\text{FeSO}_4$ ), 2 mL <sup>7</sup>      |
| ◆ Pipets (13), 1 mL, disposable   | ◆ 0.1 M Silver nitrate ( $\text{AgNO}_3$ ), 5 mL <sup>8</sup>            |
| ◆ Stirring rods (several)   | ◆ 6 M Nitric acid ( $\text{HNO}_3$ ), 5 mL <sup>9</sup>                  |
| ◆ Litmus paper (15)   | ◆ 5 M Ammonia ( $\text{NH}_3$ ), 5 mL <sup>10</sup>                      |
| ◆ 0.2 M Sodium sulfate ( $\text{Na}_2\text{SO}_4$ ), 10 mL <sup>1</sup>         | ◆ 3 M Sulfuric acid ( $\text{H}_2\text{SO}_4$ ), 5 mL <sup>11</sup>      |
| ◆ 0.2 M Monopotassium phosphate ( $\text{KH}_2\text{PO}_4$ ), 5 mL <sup>2</sup> | ◆ Concentrated $\text{H}_2\text{SO}_4$ , 2 mL                            |
| ◆ 0.2 M Sodium nitrate ( $\text{NaNO}_3$ ), 5 mL <sup>3</sup>                   | ◆ Distilled water  |
| ◆ 0.2 M Sodium chloride ( $\text{NaCl}$ ), 5 mL <sup>4</sup>                    | ◆ Centrifuge   |
| ◆ Unknown anion solution, 20 mL <sup>5</sup>                                    | ◆ Marking pen  |

<sup>1-11</sup> To prepare the solutions, refer to the Lab Preparation section.

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Balancing chemical equations
- ◆ Acid-base reactions
- ◆ Stoichiometry of chemical reactions

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 14a: Separation and Analysis of Cations
- ◆ Lab 15b: Analysis of a Coordination Compound

## Using Your Data Collection System

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

As this lab activity does not use a data collection system, no Tech Tips (indicated by the symbol "◆" and a superscripted number following a step) are needed.

## Background

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Anion analysis is somewhat simpler than cation analysis because separations are not usually required. In this exercise, known solutions of four common anions are analyzed. The anions are:

- ◆  $\text{PO}_4^{3-}$  (phosphate)
- ◆  $\text{Cl}^-$  (chloride)
- ◆  $\text{SO}_4^{2-}$  (sulfate)
- ◆  $\text{NO}_3^-$  (nitrate)

After the reactions used to identify the individual anions have been completed, you will identify the anions present in an unknown solution.

## Pre-Lab Activity

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### *Setting the stage for the activity*

In this exercise you will be given a solution containing 2 to 4 unknown anions. The analysis of anions does not need a flowchart of reactions because it is much simpler than that of cations.

You must perform the reactions in the procedure with great care so that complete precipitation and separation occur. Otherwise, unreacted ions might interfere with subsequent steps in the procedure and cause false positive results.

### Example calculation to try

The table below summarizes results obtained from the analysis of a sea water sample. For procedural details refer to the Procedure section of this activity.

Table 1: Steps of anion identification of a sea water sample

Step	Action	Observation	Possible Explanation
1	Added $\text{Ba}(\text{NO}_3)_2$ solution by drops until precipitation occurs.	White precipitate formed	Possibly $\text{SO}_4^{2-}$ and $\text{PO}_4^{3-}$ are present.
2	Separated the precipitate from the supernatant. Added $\text{HNO}_3$ solution to the precipitate.	Some precipitate dissolved	The remaining precipitate is $\text{BaSO}_4$ , indicating the presence of $\text{SO}_4^{2-}$ .
3	Separated the precipitate from the supernatant. Added $\text{AgNO}_3$ to the supernatant.	Yellow precipitate formed	The precipitate is $\text{Ag}_3\text{PO}_4$ , which confirms the presence of $\text{PO}_4^{3-}$ .
4	Added $\text{AgNO}_3$ solution to the supernatant from Step 2, and then added $\text{NH}_3$ solution.	White precipitate formed. Adding $\text{NH}_3$ solution dissolved the precipitate.	The precipitate that dissolves in $\text{NH}_3$ is $\text{AgCl}$ , indicating the presence of $\text{Cl}^-$ .
5	Tested for $\text{NO}_3^-$ .	No change.	$\text{NO}_3^-$ can be excluded.

The analysis confirmed the presence of  $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$ , and  $\text{PO}_4^{3-}$  ions.

#### 1. What would be another method to identify the $\text{Cl}^-$ ions? (Hint: Remember the activity dealing with the reactions of cations.)

$\text{Cl}^-$  ions result in a  $\text{PbCl}_2$  precipitate when reacted with  $\text{Pb}^{2+}$ . The  $\text{PbCl}_2$  can be dissolved by heating.

#### 2. Why is $\text{Ba}^{2+}$ used in Step 1 in the above table instead of $\text{Pb}^{2+}$ ?

While  $\text{Pb}^{2+}$  would precipitate with both,  $\text{SO}_4^{2-}$  and  $\text{PO}_4^{3-}$  anions, the latter substance would not dissolve in  $\text{HNO}_3$ . This means you would not be able to detect  $\text{PO}_4^{3-}$ .

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

### Anion solutions

- 0.2 M  $\text{Na}_2\text{SO}_4$ :** Dissolve 28 g of  $\text{Na}_2\text{SO}_4$  in some distilled water in a 1-L volumetric flask and fill it to the mark.

## Lab 14b: Analysis of Anions

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- 0.2 M  $\text{KH}_2\text{PO}_4$ :** Dissolve 27 g of  $\text{KH}_2\text{PO}_4$  in some distilled water in a 1-L volumetric flask and fill it to the mark.
- 0.2 M  $\text{NaNO}_3$ :** Dissolve 17 g of  $\text{NaNO}_3$  in some distilled water in a 1-L volumetric flask and fill it to the mark.
- 0.2 M  $\text{NaCl}$ :** Dissolve 12 g of  $\text{NaCl}$  in some distilled water in a 1-L volumetric flask and fill it to the mark.
- Unknown solution:** Mix equal volumes of  $\text{NaNO}_3$ ,  $\text{KH}_2\text{PO}_4$ ,  $\text{NaCl}$ , and  $\text{Na}_2\text{SO}_4$  solution.

### Test solutions

- 0.2 M  $\text{Ba}(\text{NO}_3)_2$ :** Dissolve 52 g of  $\text{Ba}(\text{NO}_3)_2$  in some distilled water in a 1-L volumetric flask and fill it to the mark.
- Saturated  $\text{FeSO}_4$ :** Add solid  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  to distilled water until no more will dissolve.
- 0.1 M  $\text{AgNO}_3$ :** Dissolve 16 g of  $\text{AgNO}_3$  in some distilled water in a 1-L volumetric flask and fill it to the mark.
- 3 M  $\text{HNO}_3$ :** Combine 190 mL of 60%  $\text{HNO}_3$  solution *slowly* with some distilled water in a 1-L volumetric flask and fill it to the mark.
- 0.5 M  $\text{NH}_3$ :** Combine 340 mL of 30%  $\text{NH}_3$  solution with some distilled water in a 1-L volumetric flask and fill it to the mark.
- 3 M  $\text{H}_2\text{SO}_4$ :** Combine 167 mL of 98%  $\text{H}_2\text{SO}_4$  solution *slowly* with some distilled water in a 1-L volumetric flask and fill it to the mark.

**Note:** Add the 98%  $\text{H}_2\text{SO}_4$  solution slowly to some distilled water while mixing and not the other way around.

### Safety

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Add these important safety precautions to your normal laboratory procedures:

- ◆ Wear safety goggles throughout this activity.
- ◆ Concentrated acids are extremely dangerous. Pay extra attention to avoid contact with skin and eyes.
- ◆ Wash acids off with plenty of water in case of skin contact.
- ◆ Add concentrated acids to water or solutions *very slowly*.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

4	2	1	3
Determine the constituents of your unknown and complete the balanced chemical equations.	Perform the test for phosphate and sulfate ions, the test for chloride ions, and the test for nitrate ions.	Prepare a fresh set of test tubes with the known anions to begin the first test.	After each test using known anions, run the test on your unknown and record all observations.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** The procedure section of this set of labs typically comes with Tech Tips (indicated by the symbol "♦" and a superscripted number following a step). No Tech Tips apply to this particular lab.

### Set Up

1.  To prepare a fresh set of test tubes: Procedures 1 and 2 should be performed with a set of test tubes in which each test tube contains a known anion. To set up your test tubes:
  - a. Label 4 clean test tubes "1" through "4" and place them in the test tube rack.
  - b. Label the disposable pipets to prevent contamination of the solutions.
  - c. Add 4 to 5 drops of  $\text{KH}_2\text{PO}_4$ ,  $\text{Na}_2\text{SO}_4$ ,  $\text{NaCl}$ , and  $\text{NaNO}_3$  to the test tubes in the order shown in Table 2.

Table 2: Solutions for Step 1.

Test tube	Known anion
1	$\text{PO}_4^{3-}$ , phosphate
2	$\text{SO}_4^{2-}$ , sulfate
3	$\text{Cl}^-$ , chloride
4	$\text{NO}_3^-$ , nitrate
5	Unknown solution

## Lab 14b: Analysis of Anions

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- d. Add 5 M ammonia to each solution by drop until it is basic when tested with litmus paper: After thoroughly mixing, touch the solution in the test tube with a clean stirring rod and then touch a piece of litmus paper with the stirring rod for testing.

**Note:** Use different stirring rods for each solution or, if using a single stirring rod, thoroughly rinse and dry the stirring rod before mixing the next solution.

2.  In a fifth test tube, prepare the unknown solution as follows:
- a. Add 10 to 15 drops of the unknown solution. The unknown solution has some of the anions you are testing.
- b. Add 5 M ammonia to the unknown solution by drops until it is basic when tested with litmus paper: After thoroughly mixing, touch the solution in the test tube with a clean stirring rod and then touch a piece of litmus paper with the stirring rod for testing.

### Collect Data

#### Procedure 1: Ba(NO<sub>3</sub>)<sub>2</sub> test

3.  Prepare a fresh set of test tubes as described in the Set Up section.
4.  Add 2 to 3 drops of Ba(NO<sub>3</sub>)<sub>2</sub> to test tubes 1 through 4 to form precipitates between Ba<sup>2+</sup> and some of the anions.
5.  Which anions precipitate with Ba<sup>2+</sup> ions?

Precipitation occurs with SO<sub>4</sub><sup>2-</sup> and PO<sub>4</sub><sup>3-</sup> ions.

6.  Record your observations in Table 3 in the Data Analysis section.
7.  **Unknown:** Add Ba(NO<sub>3</sub>)<sub>2</sub> solution drop by drop to your unknown until precipitation occurs.
- Note:** Precipitation should occur after adding 1-2 drops of Ba(NO<sub>3</sub>)<sub>2</sub>. If there is no precipitate after adding 1-2 drops, stop adding Ba(NO<sub>3</sub>)<sub>2</sub>.
8.  **Unknown:** Centrifuge the mixture in the test tube. Using a clean test tube, separate the supernatant (label it Supernatant 1) from the precipitate (label it Precipitate 1) and save them both.
9.  **Unknown:** Record your observations in Table 4.
10.  To each test tube (from 1 through 4), that contains a precipitate formed with Ba(NO<sub>3</sub>)<sub>2</sub>, add 6 M HNO<sub>3</sub> drop by drop until the solution is acidic when tested with litmus paper after stirring.

11.  Which precipitate dissolved in  $\text{HNO}_3$ ?

The precipitate formed with  $\text{PO}_4^{3-}$  ions dissolves.

12.  Record your observations in Table 3.

13.  **Unknown:** Add 6 M  $\text{HNO}_3$  drop by drop to the precipitate obtained (if any) from your unknown (Precipitate 1).

14.  **Unknown:** Observe if there is any partial or complete dissolution. Record your observations in Table 4.

15.  **Unknown:** Using a clean test tube, separate the supernatant from the precipitate, if there is any, and label them as Precipitate 2 and Supernatant 2.

16.  **Unknown:** Was there any dissolution? If so, which anion was present? If not or if there was precipitate left, that would prove the presence of which ion?

Partial dissolution is evidence of the presence of  $\text{PO}_4^{3-}$  ions. Any remaining precipitate is  $\text{BaSO}_4$ , which indicates the presence of  $\text{SO}_4^{2-}$  ions in the original solution.

17.  **Unknown:** Record your observations in Table 4.

### Procedure 2: $\text{AgNO}_3$ test

18.  Prepare a fresh set of test tubes labeled 1 through 4 as described in the Set Up section.

19.  Using the graduated cylinder, dilute each solution with 1 mL of distilled water.

20.  Add 2 drops of  $\text{AgNO}_3$  to each of the four test tubes. Some of the anions should combine with the silver ion to form insoluble precipitates.

21.  Add  $\text{NH}_3$  solution by drops to the solution that contained the  $\text{Cl}^-$  ions until the precipitate dissolves.

22.  Record your observations in Table 3 in the Data Analysis section.

23.  **Unknown:** Which anions have been removed with  $\text{Ba}(\text{NO}_3)_2$  and which might be present in Supernatant 1?

$\text{SO}_4^{2-}$  and  $\text{PO}_4^{3-}$  ions have been removed and  $\text{Cl}^-$  and  $\text{NO}_3^-$  ions could be present.

24.  **Unknown:** Add 2 drops of  $\text{AgNO}_3$  solution to Supernatant 1.

25.  **Unknown:** If there is a precipitate, what anion had to be present?

Since there only  $\text{Cl}^-$  and  $\text{NO}_3^-$  ions could be present, precipitation indicates the presence of  $\text{Cl}^-$ .

## Lab 14b: Analysis of Anions

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26.  **Unknown:** Record your observations in Table 4.

27.  **Unknown:** Which anions might be present in Supernatant 2?

$\text{SO}_4^{2-}$  ions were removed and remain removed.  $\text{PO}_4^{3-}$  ions have been removed but dissolved again with  $\text{HNO}_3$ . Therefore  $\text{PO}_4^{3-}$  ions could be present.

28.  **Unknown:** Add 2 drops of  $\text{AgNO}_3$  solution to Supernatant 2. Add  $\text{NH}_3$  solution until the precipitate dissolves.

29.  **Unknown:** If there is precipitation, what anion has to be present?

Since only  $\text{PO}_4^{3-}$  and  $\text{NO}_3^-$  ions could be present, precipitation indicates the presence of  $\text{Cl}^-$ .

30.  **Unknown:** Record your observations in Table 4.

### Procedure 3: The brown ring test for nitrate ion

31.  Add 10 drops of 0.2 M  $\text{NaNO}_3$  solution into a clean test tube.

32.  Add 3 M  $\text{H}_2\text{SO}_4$  drop by drop while stirring until the solution is acidic when tested with litmus paper.

33.  Add 5 drops of freshly prepared saturated iron(II) sulfate ( $\text{FeSO}_4$ ) solution.

34.  Gently tilt the test tube to a  $45^\circ$  angle and very carefully add 5 drops of concentrated  $\text{H}_2\text{SO}_4$  so the drops flow down the side of the test tube and float on top of the solution.

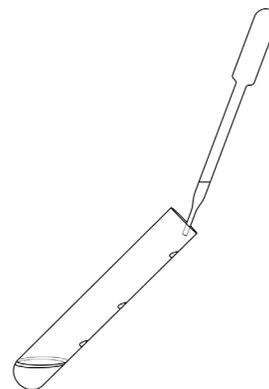
**Note:** Do not mix the solutions.

35.  Observe the indication of the presence of  $\text{NO}_3^-$ . Record your observations in Table 3.

**Note:** A very faint brown ring will appear at the interface between the liquid layers in the test tube.

36.  **Unknown:** Add a few drops of fresh unknown in a clean test tube and perform the analysis for nitrate ions.

37.  **Unknown:** Record your observations in Table 4.



## Data Analysis

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1.  Enter the equations and identified cations in Table 3 that conform to your observations.

Table 3: Test observations and results with known anions

Procedure	Anion	Actions	Observations	Proposed Equations
1: Ba(NO <sub>3</sub> ) <sub>2</sub> test for PO <sub>4</sub> <sup>3-</sup> and SO <sub>4</sub> <sup>2-</sup> ions	PO <sub>4</sub> <sup>3-</sup>	Ba(NO <sub>3</sub> ) <sub>2</sub> added	white precipitate formed	$2\text{PO}_4^{3-}(\text{aq}) + 3\text{Ba}^{2+}(\text{aq}) \rightarrow \text{Ba}_3(\text{PO}_4)_2(\text{s})$
		HNO <sub>3</sub> added	precipitate disappears	$\text{Ba}_3(\text{PO}_4)_2(\text{s}) + 3\text{H}^+(\text{aq}) \rightarrow 3\text{Ba}^{2+}(\text{aq}) + \text{H}_3\text{PO}_4(\text{aq})$
	SO <sub>4</sub> <sup>2-</sup>	Ba(NO <sub>3</sub> ) <sub>2</sub> added	white precipitate formed	$\text{Ba}^{2+}(\text{aq}) + \text{SO}_4^{2-}(\text{aq}) \rightarrow \text{BaSO}_4(\text{s})$
		HNO <sub>3</sub> added	precipitate remains	
	Cl <sup>-</sup>	Ba(NO <sub>3</sub> ) <sub>2</sub> added	no precipitate formed	
	NO <sub>3</sub> <sup>-</sup>	Ba(NO <sub>3</sub> ) <sub>2</sub> added	no precipitate formed	
2: AgNO <sub>3</sub> test for Cl <sup>-</sup> ions and PO <sub>4</sub> <sup>3-</sup> ions	PO <sub>4</sub> <sup>3-</sup>	AgNO <sub>3</sub> added	yellow precipitate formed	$3\text{Ag}^+(\text{aq}) + \text{PO}_4^{3-}(\text{aq}) \rightarrow \text{Ag}_3(\text{PO}_4)(\text{s})$
		NH <sub>3</sub> added	no change—precipitate didn't dissolve	
	SO <sub>4</sub> <sup>2-</sup>	AgNO <sub>3</sub> added	no precipitate formed	
	Cl <sup>-</sup>	AgNO <sub>3</sub> added	white precipitate formed	$\text{Ag}^+(\text{aq}) + \text{Cl}^-(\text{aq}) \rightarrow \text{AgCl}(\text{s})$
		NH <sub>3</sub> added	precipitate dissolves	
	NO <sub>3</sub> <sup>-</sup>	AgNO <sub>3</sub> added	no precipitate formed	
3: Brown ring test for NO <sub>3</sub> <sup>-</sup>	NO <sub>3</sub> <sup>-</sup>	NaNO <sub>3</sub> , H <sub>2</sub> SO <sub>4</sub> , FeSO <sub>4</sub> , and H <sub>2</sub> SO <sub>4</sub> added	Very faint brown ring	

## Lab 14b: Analysis of Anions

Table 4: Analysis of unknown solution

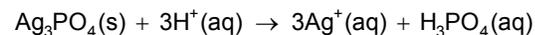
Step	Action	Observations	Anion Present
1	Add Ba(NO <sub>3</sub> ) <sub>2</sub> solution by drops until precipitation occurs.	White precipitate	Possibly SO <sub>4</sub> <sup>2-</sup> and PO <sub>4</sub> <sup>3-</sup> are present.
2	Separate the precipitate (Precipitate 1) from the supernatant (Supernatant 1). Add HNO <sub>3</sub> solution to the precipitate. Separate supernatant (Supernatant 2) from precipitate (Precipitate 2)	Some precipitate dissolved	The remaining precipitate is BaSO <sub>4</sub> , indicating the presence of SO <sub>4</sub> <sup>2-</sup> .
3	Add AgNO <sub>3</sub> to Supernatant 2.	Yellow precipitate is observed.	The precipitate is Ag <sub>3</sub> PO <sub>4</sub> , which confirms the presence of PO <sub>4</sub> <sup>3-</sup> .
4	Add AgNO <sub>3</sub> solution Supernatant 1 and then added NH <sub>3</sub> solution.	White precipitate. Adding NH <sub>3</sub> solution dissolves the precipitate.	The precipitate that dissolves in NH <sub>3</sub> is AgCl, indicating the presence of Cl <sup>-</sup> .
5	Test for NO <sub>3</sub> <sup>-</sup> .	Ring should be observed.	Presence of NO <sub>3</sub> <sup>-</sup> can be confirmed.

2.  List the anions present in your unknown.

In this example, PO<sub>4</sub><sup>3-</sup>, SO<sub>4</sub><sup>2-</sup>, Cl<sup>-</sup>, and NO<sub>3</sub><sup>-</sup> were present.

### Analysis Questions

1. Ag<sup>+</sup> ions in a solution with PO<sub>4</sub><sup>3-</sup> ions resulted in a precipitate. HNO<sub>3</sub> was then added. Provide an equation for the reaction between the precipitate and HNO<sub>3</sub>.



2. Consider the result of the Ba(NO<sub>3</sub>)<sub>2</sub> test with SO<sub>4</sub><sup>2-</sup> ions. What happened when you added HCl to the reaction mixture? Also consider that adding HNO<sub>3</sub> would not change the precipitate. What conclusion can you draw about the product of BaCl<sub>2</sub> and SO<sub>4</sub><sup>2-</sup>?

The product, BaSO<sub>4</sub>, is rather stable because it is insoluble in HCl and HNO<sub>3</sub>.

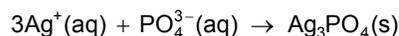
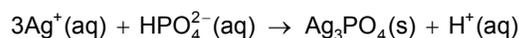
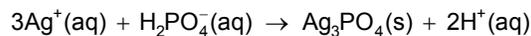
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**Synthesis Questions**

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Use available resources to help you answer the following questions.

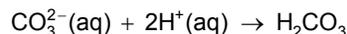
- 1.**  $\text{Ag}^+$  reacts with all three types of phosphate ions. Show the equation for each type.



- 2.** Propose a method to distinguish between the  $\text{PO}_4^{3-}$  and  $\text{H}_2\text{PO}_4^-$  ions.

After the reaction is completed, the reaction mixture with the  $\text{H}_2\text{PO}_4^-$  ion is acidic because of the released  $\text{H}^+$  ions that can be detected with an acid-base indicator such as litmus paper.

- 3.**  $\text{CO}_3^{2-}$  ions behave very much like  $\text{SO}_4^{2-}$  ions with  $\text{Ag}^+$  and  $\text{Ba}^{2+}$ . However, they react with  $\text{HCl}$  differently. Propose a possible equation for the reaction between  $\text{CO}_3^{2-}$  and  $\text{HCl}$ . (Hint: One of the products is unstable and decomposes to form a gas.)



$\text{CO}_2$  leaves the test tube as gas and fizzing can be observed.

---

**Multiple Choice Questions**

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Select the best answer or completion to each of the questions or incomplete statements below.

- 1.**  $\text{AgNO}_3$  added to a solution formed a white precipitate. This could indicate the presence of:

- A.  $\text{Cl}^-$
- B.  $\text{NO}_3^-$
- C.  $\text{SO}_4^{2-}$
- D. None of these

- 2.** A yellow precipitate can be:

- A.  $\text{Ag}_3\text{PO}_4$
- B.  $\text{AgCl}$
- C.  $\text{BaSO}_4$
- D.  $\text{Ba}_3(\text{PO}_4)_2$

### Lab 14b: Analysis of Anions

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**3.** A  $\text{PO}_4^{3-}$  ion yields a white precipitate when mixed with an unknown cation. The resulting precipitate can then be dissolved in HCl. The cation could be:

- A.**  $\text{Ag}^+$
- B.**  $\text{Pb}^{2+}$
- C.**  $\text{Ba}^{2+}$
- D.**  $\text{Ni}^{2+}$

### Extended Inquiry Suggestions

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Challenge your students by giving them a number of unknown solutions in test tubes and have them perform the reactions among only those solutions without using any other reagents. Allow your students to identify the contents of the test tubes using a reaction matrix and what they have learned in this experiment. An example of a reaction matrix is shown below.

The following solutions are included:  $\text{BaCl}_2$ ,  $\text{AgNO}_3$ ,  $\text{HCl}$ ,  $\text{Na}_3\text{PO}_4$ ,  $\text{Na}_2\text{SO}_4$ , and  $\text{Pb}(\text{NO}_3)_2$  and are put in test tubes (TT) numbered “1” through “6.” (The dash indicates there was no change when the solutions were combined.)

Reaction Matrix

TT	1	2	3	4	5	6
1		—	White precipitate that does not dissolve with heat or by adding HCl	—	—	Yellow precipitate
2			—	White precipitate that does not dissolve with heat or by adding HCl	White precipitate that does not dissolve with heat or by adding HCl	—
3				White precipitate that dissolves with heat	—	—
4					White precipitate that dissolves with heat	White precipitate that does not dissolve with heat or by adding HCl
5						White precipitate that does not dissolve with heat. It does dissolve by adding HCl
6						

Your students can apply the following strategy:

1. The white precipitate that dissolves by heat has to be  $\text{PbCl}_2$ . Therefore #3 and #4 are  $\text{Pb}^{2+}$  and  $\text{HCl}$ . Since 3 has fewer reactions, let's assume it is  $\text{HCl}$ , and then 4 has to be  $\text{Pb}^{2+}$ . Indeed, among the solutions,  $\text{Pb}^{2+}$  reacts with four of the solutions, which is the case with solution #4. Therefore solution #3 is  $\text{HCl}$  and solution #4 is  $\text{Pb}(\text{NO}_3)_2$ .
2. The white precipitate which dissolves in  $\text{HCl}$  has to be  $\text{Ba}_3(\text{PO}_4)_2$ . Therefore, either #5 is  $\text{BaCl}_2$  and #6 is  $\text{Na}_3\text{PO}_4$  or the other way around.

### Lab 14b: Analysis of Anions

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- As  $\text{Ag}^+$  and  $\text{PO}_4^{3-}$  are among the cations and anions, there should be a yellow precipitate,  $\text{Ag}_3\text{PO}_4$ . The only yellow precipitate is from the reaction between #1 and #6. In the light of the previous point, #6 must be  $\text{Na}_3\text{PO}_4$  then and #1 should be  $\text{AgNO}_3$ . Also in the light of the previous point, #5 must be  $\text{BaCl}_2$ .
- $\text{AgNO}_3$  forms a white precipitate with  $\text{HCl}$ . The only white precipitate that #1 gives is with #3, therefore #3 is confirmed as  $\text{HCl}$  and #1 is  $\text{AgNO}_3$ .
- #2 must then be  $\text{Na}_2\text{SO}_4$  which can be confirmed with the reaction with #4 (white precipitate forms:  $\text{PbSO}_4$ ) and #5 (white precipitate forms:  $\text{BaSO}_4$ ).

Therefore, the solutions are as shown in the following table.

Answers for the Reaction Matrix

Test Tube	Solution
1	$\text{AgNO}_3$
2	$\text{Na}_2\text{SO}_4$
3	$\text{HCl}$
4	$\text{Pb}(\text{NO}_3)_2$
5	$\text{BaCl}_2$
6	$\text{Na}_3\text{PO}_4$

## Lab 15a: Synthesis of a Coordination Compound

### Objectives

Students synthesize a coordination compound, potassium aluminum sulfate dodecahydrate (alum), and calculate the theoretical and percent yields.

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Carrying out a series of reactions that result in the synthesis of alum
- ◆ Practicing laboratory techniques
- ◆ Performing basic laboratory separation processes such as filtration, decanting, and recrystallization

### Time Requirement

◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

#### *For each student or group:*

- |   |   |
|---|---|
| ◆ Balance (one per class)                     | ◆ Beaker tongs  |
| ◆ Hot plate                                   | ◆ Filter paper (3)  |
| ◆ Fume hood                                   | ◆ Wire gauze  |
| ◆ Beaker, 400-mL                              | ◆ 3 M Sulfuric acid (H <sub>2</sub> SO <sub>4</sub> ), 35 mL <sup>1</sup> |
| ◆ Beaker (2), 250-mL                          | ◆ 3 M Potassium hydroxide (KOH), 25 mL <sup>2</sup>                       |
| ◆ Beaker, 100-mL                              | ◆ 50% Ethanol, 50 mL <sup>3</sup>   |
| ◆ Graduated cylinder, 50-mL                   | ◆ 100% Ethanol, 50 mL   |
| ◆ Büchner funnel                              | ◆ Acetone (C <sub>3</sub> H <sub>6</sub> O), 50 mL                        |
| ◆ Filter flask (also called a Büchner filter) | ◆ Aluminum foil, 1.1 g  |
| ◆ Stirring rod, glass                         | ◆ Distilled water for rinsing equipment                                   |
| ◆ Watch glass                                 | ◆ Ice, 400 mL   |
| ◆ Scissors                                    |   |

<sup>1-3</sup>To prepare the solutions, refer to the Lab Preparation section.

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Acid-base reactions
- ◆ Amphoteric behavior
- ◆ Balancing chemical equations
- ◆ Reduction-oxidation reactions
- ◆ Stoichiometry of chemical reactions

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 15b: Analysis of a Coordination Compound
- ◆ Lab 22a: Organic Synthesis I—Preparation

## Using Your Data Collection System

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

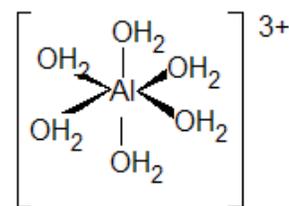
As this lab activity does not use a data collection system, no Tech Tips (indicated by the symbol "◆" and a superscripted number following a step) are needed.

## Background

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Synthesis is an especially important procedure for manufacturing chemicals we use in everyday life. Along with naturally available chemicals, synthetic chemicals help make our lives more comfortable. This lab activity is an excellent example of the synthesis process.

Alum is the name for a group of crystallized hydrated double sulfates. The following represents a general formula for alum, where  $M^+$  refers to an alkali metal or ammonium ion, and  $M^{3+}$  refers to one of the trivalent metal ions:



Hydrated aluminum ion

These salts are used in dyeing, water purification, and food preservation. They are also used as fire retardants and in various industrial processes. Potassium aluminum sulfate dodecahydrate, or potassium alum as it is known, is the commonly used form of alum. Although the aluminum ion exists in the  $\text{Al}(\text{H}_2\text{O})_6^+$  form in aqueous solution, we just usually omit writing the water molecules, since they play no role in the chemistry of the aluminum ion. The crystal of potassium alum forms with twelve water molecules.

Potassium alum,  $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ , can be synthesized through the following reactions: Potassium hydroxide (KOH) oxidizes aluminum to form soluble aluminum ions. The amphoteric aluminum ions form  $\text{Al}(\text{OH})_4^-$ , which forms insoluble aluminum hydroxide,  $\text{Al}(\text{OH})_3$ , when treated with sulfuric acid. Applying excess sulfuric acid, however, dissolves the aluminum hydroxide into  $\text{Al}^{3+}$  ions. As the nearly saturated solution cools, octahedral crystals of alum form.

## Pre-Lab Activity

### Setting the stage for the activity

Synthesizing alum starts with dissolving a piece of aluminum foil in a KOH solution. This forms  $\text{Al}(\text{OH})_4^-$  which reacts to create  $\text{Al}^{3+}$  ions (as one of the products) with  $\text{H}_2\text{SO}_4$ . After evaporating most of the water, crystals of  $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  form.

To facilitate the precipitation process, the reaction mixture needs to cool and is then transferred into a filtration funnel. Filtration is a common technique to separate solid components from a mixture.

A chilled mixture of ethanol and water washes out the contaminating ions but minimizes the loss of the crystal product.

### Example calculation to try

A sample of 1.012 g of aluminum foil was dissolved in 25 mL of 3 M KOH. The solution was filtered and 3 M  $\text{H}_2\text{SO}_4$  was added to the supernatant (the clear, filtered solution). A sufficient amount of sulfuric acid was added so that the  $\text{Al}(\text{OH})_3$  that precipitated when sulfuric acid was first added, dissolved again.

Next, the reaction mixture was heated until the volume was reduced to about half of the original volume. After the reaction mixture was cooled, precipitated alum crystals were filtered and washed with 50% chilled ethanol. The crystals were dried and their mass measured. The mass of the crystals was 10.50 g.

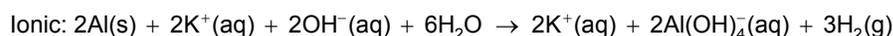
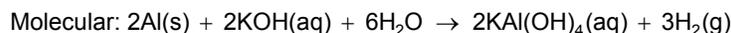
The theoretical yield is:

$$(1.012 \text{ g Al}) \left( \frac{474.1 \text{ g KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}}{26.98 \text{ g Al}} \right) = 17.77 \text{ g KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$$

The actual yield was

$$\left( \frac{10.50 \text{ g}}{17.77 \text{ g}} \right) \times 100 = 59.09\%$$

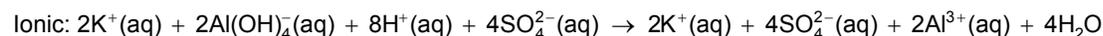
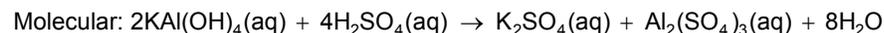
### 1. What are the molecular, ionic, and net ionic equations of the dissolution of aluminum in KOH?



## Lab 15a: Synthesis of a Coordination Compound

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**2. What are the molecular, ionic, and net ionic equations converting the  $\text{KAl(OH)}_4$  solution to  $\text{Al}_2(\text{SO}_4)_3$  solution?**



### Lab Preparation

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These are the materials and equipment to set up prior to the lab:

- 1. 3 M  $\text{H}_2\text{SO}_4$ :** Very slowly add 330 mL of 98%  $\text{H}_2\text{SO}_4$  to at least 500 mL water in a 2-L volumetric flask. Once the solution has cooled, fill the flask to the mark.
- 2. 3 M  $\text{KOH}$ :** Dissolve 336 g  $\text{KOH}$  in water in a 2-L volumetric flask and fill it to the mark.
- 3. 50% Ethanol:** Combine 500 mL ethanol and some water in a 1-L volumetric flask and fill it to the mark.

### Safety

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Add these important safety precautions to your normal laboratory procedures:

- ◆ Both  $\text{KOH}$  and  $\text{H}_2\text{SO}_4$  can cause serious burns. Handle them carefully. Wash off  $\text{KOH}$  or  $\text{H}_2\text{SO}_4$  with plenty of water in case of skin contact.

### Sequencing Challenge

---

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

4	3	2	1	5
Set the reduced solution in an ice bath to facilitate crystal formation. Isolate the crystals through filtration.	Boil the acidic solution until about half of the solvent evaporates and then cool the solution to room temperature.	Add $\text{H}_2\text{SO}_4$ solution slowly until the precipitate that forms is dissolved.	Under a fume hood, dissolve a piece of aluminum foil in a $\text{KOH}$ solution. Filter the solution.	Obtain the mass of the alum crystals and calculate the yield.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

The procedure section of this set of labs typically comes with Tech Tips (indicated by the symbol "♦" and a superscripted number following a step). No Tech Tips apply to this particular lab.

### Set Up

- Measure the mass of a piece of aluminum foil. Trim the piece or add small pieces until the mass of the aluminum is about 1 g.
- Record the mass of the aluminum, to two decimal places, in Table 1 in the Data Analysis section.
- Cut the aluminum foil into small pieces and place them into a 250-mL beaker.
- Set up the Büchner funnel and flask and insert a piece of filter paper into the funnel.

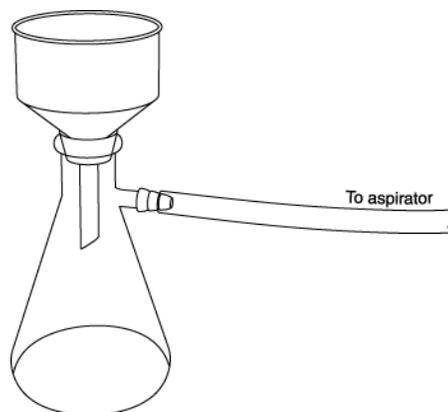
### Collect Data

- Under a fume hood, use the 50-mL graduated cylinder to slowly add 25 mL of 3 M KOH solution to the 250-mL beaker with the aluminum foil.
- When all the aluminum has dissolved and the solution has cooled, carefully filter the solution through the Büchner funnel into the filter flask with the vacuum on.
- Rinse the 250-mL beaker with distilled water while the vacuum is still on, and pour the filtered solution back into the beaker.

- Why do you need to rinse the beaker?

The beaker needs to be rinsed to ensure all of the product is transferred into the funnel.

- Clean the Büchner funnel and filter flask, and place a piece of filter paper into the funnel.



- Allow the solution to cool to room temperature.
- Rinse the graduated cylinder with distilled water.
- Pour 5 mL of the 3 M H<sub>2</sub>SO<sub>4</sub> into the graduated cylinder.

## Lab 15a: Synthesis of a Coordination Compound

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- 13.**  Stirring constantly with the glass rod, slowly add the 5 mL portion of the 3 M  $\text{H}_2\text{SO}_4$  from the graduated cylinder to the beaker of reaction mixture with constant stirring. A precipitate should form as the  $\text{OH}^-$  ions are neutralized.

**CAUTION:** This reaction is exothermic—it generates heat.

- 14.**  Continue to add 5 mL portions of sulfuric acid until the precipitate dissolves.
- 15.**  What happens after enough  $\text{H}_2\text{SO}_4$  is added to neutralize the KOH? What happens if excess  $\text{H}_2\text{SO}_4$  is added?

When all the KOH is neutralized,  $\text{Al}(\text{OH})_3$  precipitates and then dissolves when additional  $\text{H}_2\text{SO}_4$  is added and the solution becomes acidic, forming  $\text{Al}^{3+}$  ions.

- 16.**  Place the beaker on the hot plate and gently boil the solution until about 40 to 45 mL of solution remain and all the precipitate is dissolved.
- 17.**  Why is it necessary to evaporate a little more than half of the solvent?

Evaporating some of the solvent increases the concentration of the product, which promotes the precipitation.

- 18.**  Using beaker tongs, remove the beaker from the hot plate and place it on a piece of wire gauze. Allow the solution to cool to room temperature.
- 19.**  Prepare an ice bath, using the 400-mL beaker, for the 250-mL beaker and carefully place the beaker inside it. Do not disturb the ice bath or the beaker.

**Note:** Ice should be packed around the beaker above the level of the solution.

- 20.**  Why is it necessary to cool the mixture?

The solubility decreases as the temperature decreases. At lower temperature more product precipitates.

- 21.**  While the alum solution is in the ice bath, prepare a second ice bath in a 250-mL beaker.
- 22.**  Put 50 mL of 50% ethanol in a 100-mL beaker and place it carefully in the second ice bath.

**Note:** Ice should be packed around the beaker above the level of the solution.

- 23.**  After about 15 minutes, check the contents of the first beaker for the presence of alum crystals.

**Note:** If no crystals are visible, scratch the bottom of the beaker with a glass stirring rod to promote crystal formation.

- 24.**  Turn on the filtration system.
- 25.**  Pour the mixture through the funnel.

- 26.**  Wash the product remaining on the filter paper with the 50 mL of cold 50% ethanol solution.
- 27.**  Rinse the product with 50 mL of pure ethanol while continuing the filtration.
- 28.**  Rinse the product with 50 mL of acetone in order to dry the product.
- 29.**  After the acetone has drained from the Büchner funnel, continue the suction for another five minutes to ensure that the product is dry and then turn off the vacuum.
- 30.**  Carefully remove the filter paper and crystals and place them on a watch glass.
- 31.**  Allow the crystals to dry at room temperature.
- 32.**  Measure and record the mass of alum in Table 1 in the Data Analysis section.
- 33.**  Clean up according to your teacher's directions.

## Data Analysis

Record the necessary measurements and complete the calculations.

- 1.**  Calculate the amount of aluminum that was dissolved.

$$\frac{1.009 \text{ g Al}}{26.98 \frac{\text{g}}{\text{mol}} \text{ Al}} = 0.03740 \text{ mol Al}$$

- 2.**  Calculate the theoretical number of moles of alum considering the stoichiometric ratio between aluminum and alum.

$$0.03740 \text{ mol Al} = 0.03740 \text{ mol alum}$$

- 3.**  Calculate the theoretical mass of alum that can be synthesized from the aluminum foil used for this procedure.

$$(0.03740 \text{ mol KAl(SO}_4)_2 \cdot 12\text{H}_2\text{O}) \left( \frac{474.3 \text{ g KAl(SO}_4)_2 \cdot 12\text{H}_2\text{O}}{1 \text{ mol KAl(SO}_4)_2 \cdot 12\text{H}_2\text{O}} \right) = 17.73 \text{ g KAl(SO}_4)_2 \cdot 12\text{H}_2\text{O}$$

- 4.**  Calculate the yield from the theoretical amount and experimental amount of alum.

$$\left( \frac{15.33 \text{ g}}{17.73 \text{ g}} \right) \times 100 = 86.46\%$$

## Lab 15a: Synthesis of a Coordination Compound

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Table1: Determination of the yield of synthesized alum

Parameter	Value
Mass of aluminum foil (g)	1.009
Amount of aluminum (mol)	0.03740
Theoretical amount of alum (mol)	0.03740
Theoretical mass of alum (g)	17.73
Actual mass of alum (g)	15.33
Yield (%)	86.46

### Analysis Questions

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**1. How could you increase the yield?**

Reducing the volume of the reaction mixture by boiling as well as lowering the temperature of the ice bath could promote more precipitation.

**2. How would contamination, such as dust, affect the formation of the crystals?**

Contamination such as dust particles would serve as centers to initiate precipitation. Because of this, more but smaller crystals would form.

**3. During crystallization, byproducts can also crystallize. What are two possible byproducts that can crystallize along with alum?**

$K_2SO_4$  and  $Al_2(SO_4)_3$  can crystallize along with alum.

**4. How could you purify the crystals further? What is the drawback of purifying the crystals?**

Recrystallization from water would purify the crystals. However, it would lower the yield.

### Synthesis Questions

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Use available resources to help you answer the following questions.

**1. The term "alum" is a collective term for a group of salts. Briefly, how can you make alum with  $NH_4^+$  instead of  $K^+$ ?**

The combination of solutions with stoichiometric quantities of  $(NH_4)_2SO_4$  and  $Al_2(SO_4)_3$  would yield the  $(NH_4)Al(SO_4)_2$  product. The rest of the process would be the same as the one performed in this activity.

**2. What is another metal ion that has a +3 oxidation state and would form an alum?**

$Cr^{3+}$  would be a good candidate to form  $KCr(SO_4)_2$ .

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**Multiple Choice Questions**

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Select the best answer or completion to each of the questions or incomplete statements below.

- 1. What property of aluminum explains that it dissolves in KOH?**
  - A.** It is a metal.
  - B.** Since it is amphoteric, it can react with acids and bases.
  - C.** Anything would dissolve in a strong base like KOH.
  - D.** None of the above.
  
- 2. What can be a potential problem if the volume of the solution is too large after boiling?**
  - A.** No alum is produced.
  - B.** Too much of the product remains in the solution, decreasing the yield.
  - C.** A significant amount of the contaminants precipitates and necessitates recrystallization.
  - D.** Undesired byproducts form.
  
- 3. What happens if the alum solution isn't cooled sufficiently?**
  - A.** No alum is produced.
  - B.** Too much of the product remains in the solution, decreasing the yield.
  - C.** A significant amount of the contaminants precipitates and necessitates recrystallization.
  - D.** Undesired byproducts form.

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**Extended Inquiry Suggestions**

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Suggest that students prepare more than one kind of alum. Combining saturated  $\text{K}_2\text{SO}_4$  and  $\text{Cr}_2(\text{SO}_4)_3$  solutions yields  $\text{KCr}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ . This forms amazing-looking purple crystals.

**CAUTION:** Be sure to use excess  $\text{K}_2\text{SO}_4$  to avoid contamination by  $\text{Cr}_2(\text{SO}_4)_3$ .



## Lab 15b: Analysis of a Coordination Compound

### Objectives

Students confirm the identity of a sample of alum by conducting both qualitative and quantitative analyses.

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Conducting substance identification tests such as a melting point analysis and a flame test
- ◆ Performing a stoichiometric analysis, using a crucible and Bunsen burner, to determine the amount of water in a hydrated compound
- ◆ Conducting qualitative analyses for sulfate and aluminum

### Time Requirement

◆ Preparation time	30 minutes
◆ Pre-lab discussion and activity	30 minutes
◆ Lab activity	80 minutes

### Materials and Equipment

#### *For each student or group:*

- |                                      |  |
|--------------------------------------|--|
| ◆ Data collection system             | ◆ Centrifuge (1 per class)                                     |
| ◆ Stainless steel temperature sensor | ◆ Wire with a loop on the end, 4 in.                           |
| ◆ Ring stand with ring               | ◆ Hot plate  |
| ◆ Clay triangle                      | ◆ Bunsen burner  |
| ◆ Clamp, buret                       | ◆ Striker  |
| ◆ Clamp, utility                     | ◆ 0.2 M Barium chloride ( $\text{BaCl}_2$ ), 1 mL <sup>1</sup> |
| ◆ Crucible with lid                  | ◆ 6 M Sodium hydroxide ( $\text{NaOH}$ ), 5 mL <sup>2</sup>    |
| ◆ Tongs                              | ◆ 6 M Hydrochloric acid ( $\text{HCl}$ ), 5 mL <sup>3</sup>    |
| ◆ Test tubes (2), 10 mL              | ◆ Borax, 0.5 g   |
| ◆ Beaker, 250-mL                     | ◆ Alum from previous experiment, 3 g                           |
| ◆ Capillary tube                     | ◆ Rubber band  |
| ◆ Stirring rod                       | ◆ Water, 200 mL  |
| ◆ Watch glass (2), 100-mm            | ◆ Distilled water, 10 mL                                       |
| ◆ Balance (1 per class)              |  |

<sup>1-3</sup>To prepare the solutions refer to the Lab Preparation section.

## **Concepts Students Should Already Know**

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Students should be familiar with the following concepts:

- ◆ Stoichiometry of chemical reactions
- ◆ Reactions of anions

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 9: Mole Relationships in a Chemical Reaction
- ◆ Lab 14a: Separation and Analysis of Cations
- ◆ Lab 14b: Analysis of Anions
- ◆ Lab 15a: Synthesis of a Coordination Compound
- ◆ Lab 22b: Organic Synthesis II—Analysis

## **Using Your Data Collection System**

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1,2)</sup>
- ◆ Connecting a sensor to the data collection system ◆<sup>(2,1)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6,2)</sup>

## **Background**

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Once the synthesis of a compound has been completed, the identity of the compound produced should be confirmed. A number of tests can be used to confirm the identity of potassium alum,  $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ .

To verify that the substance is potassium alum, you will perform a melting point test and a flame test; you will measure the water of hydration, and you will verify the presence of aluminum and sulfate in the compound.

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**Pre-Lab Activity**

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**Setting the stage for the activity**

In this activity, you will analyze the alum you synthesized in the previous activity. The analysis includes quantitatively measuring the melting point and the amount of water of crystallization and qualitatively identifying potassium with a flame test, detecting sulfate ions in a reaction with  $\text{Ba}^{2+}$ , and detecting aluminum by dissolving the compound in both an acid and base.

**Example calculation to try****Test 1**

An alum sample prepared by a student was analyzed. First, a capillary tube was filled with some of the sample and mounted on a temperature sensor. The sensor was placed into a water bath and the water bath was heated slowly. The temperature was monitored. The crystals started to melt at 90 °C and melted completely at 91 °C.

**Test 2**

To determine the amount of water in the synthesized alum, an empty crucible, with an initial mass of 35.443 g, was heated to a constant mass (35.222 g). After a sample of the alum was placed in the crucible, the mass was found to be 35.791 g, so the mass of the alum was

$$35.791 \text{ g} - 35.222 \text{ g} = 0.569 \text{ g}$$

The crucible was gently heated until no more vapor was released. After reheating until the mass of the crucible and remaining product was constant, the mass was 35.542 g. The amount of water lost was

$$35.791 \text{ g} - 35.542 \text{ g} = 0.249 \text{ g}$$

0.249 g of water is

$$0.249 \text{ g H}_2\text{O} \left( \frac{1 \text{ mol H}_2\text{O}}{18.01 \text{ g H}_2\text{O}} \right) = 1.38 \times 10^{-2} \text{ mol H}_2\text{O}$$

The mass and corresponding amount of the anhydrous alum was

$$0.569 \text{ g} - 0.249 \text{ g} = 0.320 \text{ g}$$

$$0.320 \text{ g alum} \left( \frac{1 \text{ mol alum}}{258.1 \text{ g alum}} \right) = 1.24 \times 10^{-3} \text{ mol alum}$$

The molar ratio between the water of crystallization and the anhydrous alum is

$$1.38 \times 10^{-2} : 1.24 \times 10^{-3} = 11.1:1$$

## Lab 15b: Analysis of a Coordination Compound

The theoretical value is 12, therefore the percent error is

$$\text{Percent Error} = \frac{|\text{Theoretical Value} - \text{Experimental Value}|}{\text{Theoretical Value}} \times 100$$

$$\text{Percent Error} = \frac{12 - 11.1}{12} \times 100 = 7.50\%$$

Table 1: Synthesized alum analysis results

Measured Quantity	Values
Melting point (°C)	91.0
Mass of crucible and lid (g)	35.222
Mass of crucible, lid, and alum sample (g)	35.791
Mass of alum sample (g)	0.569
Mass of crucible, lid, and alum after final heating (g)	35.542
Water of hydration (g)	0.249
Water of hydration (mol)	$1.38 \times 10^{-2}$
Mass of anhydrous alum (g)	0.320
Amount of anhydrous alum (FW: 258.1 g/mol) (mol)	$1.24 \times 10^{-3}$
Experimental stoichiometric ratio between $\text{KAl}(\text{SO}_4)_2$ and water	11.1:1
% Error	7.50

### Test 3

Another sample of the alum was dissolved in distilled water and reacted with  $\text{BaCl}_2$  solution. A white precipitate was observed, indicating the presence of  $\text{SO}_4^{2-}$  ions.

### Test 4

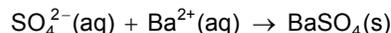
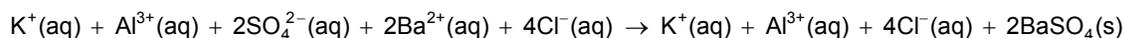
A piece of iron wire with a small loop on the end was heated in a Bunsen burner flame until it was red hot, then dipped into borax crystals on a watch glass and reheated until the borax formed a small bead on the tip of the wire. Then the borax bead on the tip was dipped into the alum sample and placed back into the Bunsen burner flame. Purple flames were observed, indicating the presence of  $\text{K}^+$  ions.

### Test 5

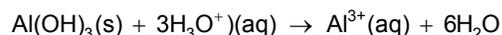
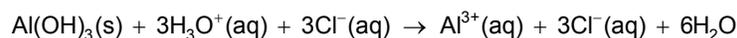
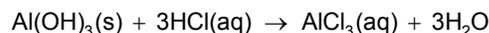
Another sample of the alum was dissolved in distilled water. Then  $\text{NaOH}$  solution was added, which formed a white precipitate. The precipitate was separated from the supernatant and split into two portions.

To one portion of the precipitate, HCl solution was added until the precipitate dissolved. To the other portion of the precipitate, NaOH solution was added until the precipitate dissolved. The result of these tests (the precipitate dissolved when both NaOH and HCl were added) substantiates the presence of  $\text{Al}^{3+}$ . (Aluminum hydroxide is amphoteric—it dissolves in both acid and base.)

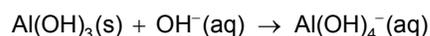
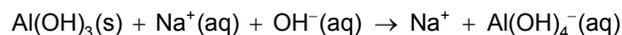
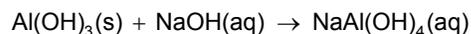
**1. What are the molecular, ionic, and net ionic equations of the reaction of alum with  $\text{BaCl}_2$ ?**



**2. What are the molecular, ionic, and net ionic equations of the reactions of the precipitate obtained from reacting alum with NaOH and then with HCl?**



**3. What are the molecular, ionic, and net ionic equations of the reactions between NaOH and the precipitate obtained from the alum solution with NaOH ?**



## Lab Preparation

These are the materials and equipment to set up prior to the lab:

- 0.2 M  $\text{BaCl}_2$ :** Dissolve 4.88 g of  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  in some water in a 100 mL volumetric flask and fill it to the mark.
- 6 M NaOH:** Dissolve 24 g of NaOH in some water in a 100 mL volumetric flask and fill it to the mark.
- 6 M HCl:** Under a hood, slowly add 128.5 mL of 36% HCl solution to about 100 mL of water in a 250 mL volumetric flask and fill it to the mark.

## Safety

Add these important safety precautions to your normal laboratory procedures:

- ◆ If the NaOH or HCl solutions come in contact with your skin or eyes, rinse immediately with a large amount of running water.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

1	4	2	3	5
Set up the data collection system for the first test to measure the melting point of the alum sample.	Observe the results of adding NaOH and HCl to alum samples dissolved in NaOH, once you've completed the flame test.	Then determine the amount of water in the alum sample and check the result of reacting dissolved alum with BaCl <sub>2</sub> .	After checking the results of the test for SO <sub>4</sub> <sup>2-</sup> , hold an alum sample in a flame to observe the color of the flame when it burns.	Determine what each of the five tests tells you about the alum sample.

## Procedure with Inquiry

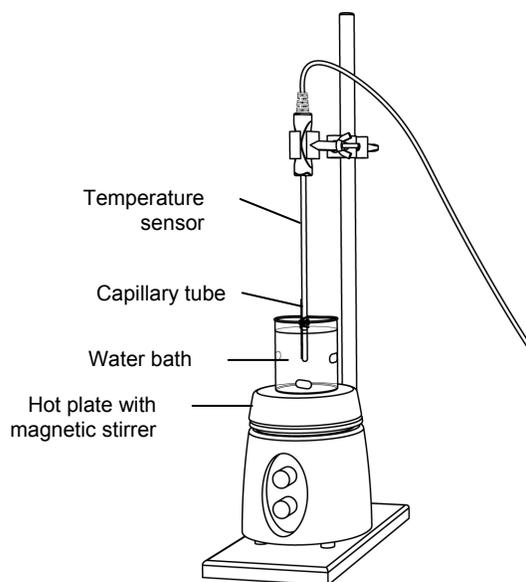
After you complete a step (or answer a question), place a check mark in the box () next to that step.

### Part 1 – Melting point determination

#### Set Up

- Set up the ring stand and hot plate as shown in the illustration.
- Fill a 250-mL beaker three-quarters full with water to use as a water bath. Place the beaker on the hot plate.
 

**Note:** Do not turn on the hot plate yet.
- Start a new experiment on the data collection system. ♦<sup>(1,2)</sup>
- Connect a stainless steel temperature sensor to the data collection system. ♦<sup>(2,1)</sup>
- Crush about 0.5 g of alum crystals to a fine powder in a watch glass using a glass stirring rod. Scrape the powdered alum into a pile in the center of the watch glass.



6.  Push the open end of a capillary into the alum, then invert the capillary tube and tap the closed end on the bench top to pack the alum into the end. Repeat this procedure until the capillary tube contains about 1 cm of firmly packed alum.
7.  Attach the capillary tube to the end of the temperature sensor with a small rubber band so that the bottom of the capillary tube is even with the end of the temperature sensor.
8.  Why is it important to have the capillary attached to the end of the temperature sensor?

The tip of the sensor measures the temperature. Therefore, to measure the temperature accurately around the capillary the tip of the sensor should be close to the capillary.

9.  Clamp the temperature sensor to the support stand using a utility clamp. Position the clamp so the tip of the temperature sensor and the portion of the capillary tube containing the alum are immersed in the water bath.

**Note:** Be sure that the open end of the capillary tube is above the water level so no water is able to get into the capillary.

### Collect Data

10.  Turn on the hot plate to heat the water bath at a gradual rate. Stir the water continuously while it heats.
11.  Start data recording. <sup>◆(6.2)</sup>
12.  Observe the alum in the capillary tube until it has completely melted. Record the temperature in Table 4.
13.  Stop data recording. <sup>◆(6.2)</sup>

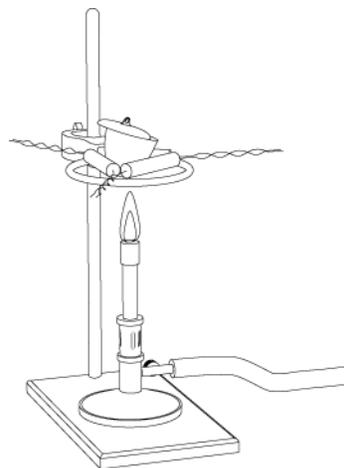
## Part 2 – Water of hydration determination

### Set Up

14.  Clean and dry a porcelain crucible and lid.
15.  Measure the mass of the crucible and lid. Record the measurement in Table 2.

## Lab 15b: Analysis of a Coordination Compound

- 16.**  To obtain a constant mass, prepare the crucible as follows:
- Place the crucible and lid on the clay triangle over the Bunsen burner.
  - Heat the crucible with a gentle flame for 5 minutes by moving the burner around the bottom of the crucible.
  - After the bottom of the crucible has become red-hot, increase the flame by allowing more air into the burner.
  - Continue moving the burner around the bottom of the crucible.
  - Heat the crucible for 10 to 12 minutes.
  - Turn off the burner and allow the crucible to cool to room temperature.



**Note:** For the rest of the experiment, handle the crucible and lid using only crucible tongs. Also, do not set the crucible on the lab bench or it may crack or become contaminated.

- 17.**  Why do you have to use tongs to hold the lid?

Touching the lid with bare hands contaminates the lid and changes its mass, introducing error in the mass measurement. Also, the crucible may still be hot and cause injury.

- 18.**  After the crucible has cooled, measure and record the mass of the “fired” crucible, together with its lid, in Table 2.

Table 2: Mass of the empty crucible measured to the nearest milligram

Measurement	Mass
Crucible and lid before heating (g)	29.851
Crucible and lid after the first heating (g)	29.835
Crucible and lid after the second heating (g)	29.833
Crucible and lid after the third heating (g)	
Crucible and lid after the fourth heating (g)	

- 19.**  Repeat the steps above for heating, cooling, and measuring the mass of the crucible and lid until you have two readings for the mass that are within 3 mg of each other.
- 20.**  Copy the last measurement in Table 2 into Table 4.
- 21.**  Why is it important to heat the crucible before using it?

It is important to remove contamination and moisture that would change the mass of the crucible when heating the sample.

- 22.**  Put about 2 g of alum into the crucible and measure the mass of the crucible, alum, and lid. Record the mass to the nearest 0.001 g in Table 3.
- 23.**  Place the crucible on the ring stand using the tongs and place the lid slightly ajar so that water vapor can escape.

**Collect Data**

- 24.**  Begin heating the crucible slowly with the Bunsen burner.

- 25.**  Why do you think it is important to heat the crucible slowly?

It is important to apply heat slowly because if the crucible is heated too quickly, the alum may splatter, resulting in error due to the loss of mass.

- 26.**  When vapor can no longer be seen escaping from the crucible, continue to move the burner around for 2 minutes, and gradually increase the size of the flame so that all parts of the crucible are heated.
- 27.**  Allow the crucible to cool for a few minutes and then place it into a desiccator, if available, using crucible tongs. Allow it to cool to room temperature. Measure and record the mass of the crucible, lid, and alum to the nearest milligram (0.001 g) in Table 3.

Table 3: Mass of the crucible and sample, measured to the nearest milligram

Measurement	Mass
Crucible, lid, and alum before heating (g)	31.813
Crucible, lid, and alum after the first heating (g)	30.972
Crucible, lid, and alum after the second heating (g)	30.972
Crucible, lid, and alum after the third heating (g)	
Crucible, lid, and alum after the fourth heating (g)	

- 28.**  Repeat the heating, cooling, and measuring procedure until two successive mass measurements are within 3 mg of each other.
- 29.**  Record all measurements in Table 3. Copy the final measurement into Table 4.

**Part 3 – BaCl<sub>2</sub> test****Set Up**

- 30.**  Dissolve a few alum crystals in 5 mL of distilled water

## Lab 15b: Analysis of a Coordination Compound

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### Collect Data

31.  Add a few drops of  $\text{BaCl}_2$  solution to the alum solution. Record your observations in Table 4.
32.  Add 3 to 5 drops of the HCl solution to the precipitate. Record your observations in Table 4.

### Part 4 – Flame test

#### Set Up

33.  Place a few crystals of borax on a watch glass. On a separate watch glass place a few crystals of alum.

#### Collect Data

34.  Place the tip of a wire with a little loop on the end in the Bunsen burner flame until the loop turns red hot.
35.  Place the glowing loop into the borax crystals. The crystals will melt on the hot tip. Place the tip again into the flame until the borax forms a melted “pearl” on the tip.
36.  Dip the hot tip into the alum crystals and back into the flame. Record your observations in Table 4.

### Part 5 – Test for aluminum

#### Set Up

37.  In one of the 10-mL test tubes, dissolve a few alum crystals in 5 mL of deionized water.

#### Collect Data

38.  Add, by drops, 6 M NaOH while a white precipitate forms. Separate the supernatant from the precipitate. (If the solution does not separate, use a centrifuge.) Divide the precipitate into two 10-mL test tubes.
39.  Slowly add 20 drops of 6 M NaOH solution to one test tube. Record your observations in Table 4.
40.  Slowly add 20 drops of 6 M HCl solution to the other test tube. Record your observations in Table 4.
41.  Clean up according to your teacher’s directions.

**Data Analysis**

1.  Calculate the error in the melting point between the literature value and from your experimental value. Record your results in Table 4.

$$\left( \frac{93.5\text{ }^{\circ}\text{C} - 92.5\text{ }^{\circ}\text{C}}{92.5\text{ }^{\circ}\text{C}} \right) \times 100 = 1.1\%$$

2.  Determine the mass of the alum from the mass of the empty crucible and the mass of the crucible with the alum sample (before heating). Record your results in Table 4.

$$31.813\text{ g} - 29.833\text{ g} = 1.980\text{ g}$$

3.  Determine the mass of the evaporated water from the mass of the crucible with the alum sample before heating and after heating. Record your results in Table 4.

$$31.8127\text{ g} - 30.9722\text{ g} = 0.840\text{ g}$$

4.  Calculate the amount of water that evaporated. Record your results in Table 4.

$$\left( \frac{0.840\text{ g H}_2\text{O}}{18.02\frac{\text{g}}{\text{mol}}\text{ H}_2\text{O}} \right) = 4.66 \times 10^{-2}\text{ mol H}_2\text{O}$$

5.  Calculate the mass of the anhydrous alum from the mass of the crucible with the alum after heating and the mass of the empty crucible. Record your results in Table 4.

$$30.972\text{ g} - 29.833\text{ g} = 1.139\text{ g}$$

6.  Calculate amount of the anhydrous alum from its mass and formula weight. Record your results in Table 4.

$$\left( \frac{1.139\text{ g alum}}{258.1\frac{\text{g}}{\text{mol}}\text{ alum}} \right) = 4.413 \times 10^{-3}\text{ mol alum}$$

7.  Calculate molar ratio between the water and the alum. Record your results in Table 4.

$$\left( \frac{4.664 \times 10^{-2}\text{ mol H}_2\text{O}}{4.413 \times 10^{-3}\text{ mol alum}} \right) = 10.57$$

## Lab 15b: Analysis of a Coordination Compound

8.  Calculate the error in the ratio between the water and alum due to the difference between the literature value and the experimental value. Record your results in Table 4.

$$\left( \frac{12 - 10.57}{12} \right) \times 100 = 11.92\%$$

Table 4: Synthesized alum analysis results

<b>Part 1 – Melting point</b>	
Theoretical melting point (°C)	92.5
Experimental melting point (°C)	93.5
Percent error (%)	1.08
<b>Part 2 – Water of Hydration</b>	
Mass of crucible and lid after heating (g)	29.833
Mass of alum (g)	1.980
Mass of crucible, cover, and alum before heating (g)	31.813
Mass of crucible, lid, and alum after heating (g)	30.972
Water of hydration (g)	0.840
Water of hydration (mol)	$4.66 \times 10^{-2}$
Amount of anhydrous alum (g)	1.139
Amount of anhydrous alum (FW: 258.1 g/mol) (mol)	$4.413 \times 10^{-3}$
Experimental stoichiometric ratio between $\text{KAl}(\text{SO}_4)_2$ and water	1:10.57
Percent error (%)	11.92
<b>Part 3 – <math>\text{BaCl}_2</math> Test</b>	
$\text{BaCl}_2$	White precipitate forms
HCl	No change
<b>Part 4 – Flame Test</b>	
Flame turns purple	
<b>Part 5 – NaOH Test</b>	
Excess NaOH	Turns solution clear
HCl	Turns solution clear

## Analysis Questions

**1. How did the theoretical melting point compare to your experimental value? What is the reason for the deviation from the theoretical value (if there was any)?**

The melting points were close—there was a difference of 1.08%. The deviation is probably due to contaminations such as  $K_2SO_4$  and  $Al_2(SO_4)_3$ .

**2. How did the theoretical water of hydration compare to your experimental value? What is the reason for the deviation from the theoretical value (if there was any)?**

The difference was 10.50%. The deviation is probably due to contaminations such as  $K_2SO_4$  and  $Al_2(SO_4)_3$ .

**3. Why did we add HCl solution to the precipitate obtained with  $BaCl_2$  from the alum solution? (Hint: Check the reactions of other possible anions.)**

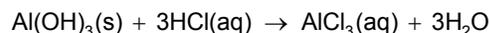
HCl was added to rule out the presence of  $PO_4^{3-}$  ions since  $Ba_3(PO_4)_2$  dissolves in HCl.

**4. Potassium ions color the Bunsen burner flame purple. Did you observe that color? Was there any other color you observed?**

Sodium contamination may cause the flame to be yellow.

**5. How does the reaction with NaOH, followed by the reactions with excess NaOH and HCl, prove the presence of aluminum? Write equations!**

There is an  $Al(OH)_3$  precipitate formed with NaOH. That precipitate is soluble in both NaOH and HCl:



## Synthesis Questions

Use available resources to help you answer the following questions.

**1. Ionic compounds usually melt at very high temperatures. How do you explain that the alum melts below the boiling point of water? (Hint: What else, other than  $KAl(SO_4)_2$ , is present in the crystals?)**

The alum does not actually melt; it dissolves in the water that is captured in the crystals.

**2. What experiment would support your answer to the previous question? (Hint: Think back to the experiments you did in this activity.)**

You could continue heating the alum sample after the water evaporated in the melting point experiment. The anhydrous alum will not melt even when the crucible is glowing red hot.

**3. What characteristics of aluminum does the reaction of  $Al(OH)_3$  with NaOH and HCl underline? (Hint: To what category of substances do NaOH and HCl belong?)**

The fact that  $Al(OH)_3$  dissolves in both acids and bases demonstrates the amphoteric characteristic of aluminum.

## Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

- 1. The melting point of alum is low because:**
  - A.** Alum is a molecular compound.
  - B.** Alum actually dissolves in its water of hydration and does not melt.
  - C.** There are always contaminations present to lower the melting point.
  - D.** None of the above.
  
- 2. The experimental result for water of hydration does not show a ratio of integer numbers between  $KAl(SO_4)_2$  and  $H_2O$ . What can you do?**
  - A.** There is nothing to do other than report the experimental value.
  - B.** One has to multiply the numbers with integer numbers until the ratio becomes a ratio between two integer numbers.
  - C.** Repeat the experiment until you do get a ratio of integer numbers.
  - D.** Round off the numbers to the nearest integer recognizing the experimental error of the process.
  
- 3. A solution of the alum that you synthesized was combined with an  $AgNO_3$  solution. There was no reaction observed. Does that contradict your claim that you synthesized alum?**
  - A.** Yes, a yellow precipitate should have been observed.
  - B.** Yes, a white precipitate should have been observed.
  - C.** No, no reaction was expected to occur.
  - D.** It depends on the temperature at which the experiment was performed.
  
- 4. A solution of the alum you synthesized was combined with a  $BaCl_2$  solution. There was no reaction observed. Does that contradict your claim that you synthesized alum?**
  - A.** Yes, a yellow precipitate should have been observed.
  - B.** Yes, a white precipitate should have been observed.
  - C.** No, there no was reaction expected to occur.
  - D.** It depends on the temperature at which the experiment was performed.

## Extended Inquiry Suggestions

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If other types of alums were prepared previously (e.g.  $KCr(SO_4)_2 \cdot 12H_2O$ ) they could be analyzed using the same methods found in this activity.

## Lab 16: Gravimetric Determination of a Precipitate

### Objectives

In this activity, students use gravimetric analysis to determine the amount of sulfate in a sample of an unknown alkali sulfate.

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Isolating sulfate from an unknown sample by precipitation with barium chloride.
- ◆ Collecting, drying, and obtaining the mass of the precipitate.
- ◆ Calculating the amount of sulfate in the unknown from collected data.

### Time Requirement

◆ Preparation time	30 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	120 minutes

### Materials and Equipment

#### *For each student or group:*

- |                                      |   |
|--------------------------------------|---|
| ◆ Data collection system             | ◆ Funnel  |
| ◆ Stainless steel temperature sensor | ◆ Dropper   |
| ◆ Ring stand with ring               | ◆ Hot plate   |
| ◆ Clamp, utility                     | ◆ Bunsen burner   |
| ◆ Clamp, buret                       | ◆ Clay triangle   |
| ◆ Crucible with lid                  | ◆ 0.5 M Barium chloride ( $\text{BaCl}_2$ ), 30-mL <sup>1</sup> |
| ◆ Tongs                              | ◆ 0.1 M Silver nitrate ( $\text{AgNO}_3$ ), 5-mL <sup>2</sup>   |
| ◆ Beaker, glass, 400-mL              | ◆ 6 M Hydrochloric acid (HCl), 5-mL <sup>3</sup>                |
| ◆ Beaker, glass, 250-mL              | ◆ Unknown alkali sulfate, 0.35 g <sup>4</sup>                   |
| ◆ Beaker (3), 25-mL                  | ◆ Filter paper, Whatman® Ashless, #42                           |
| ◆ Beaker or flask, 400-mL,           | ◆ Rubber policeman and stirring rod                             |
| ◆ Graduated cylinder, 100-mL         | ◆ Watch glass, 100-mm   |
| ◆ Graduated cylinder, 10-mL          | ◆ Distilled water, 100 mL                                       |
| ◆ Buret, 50 mL                       | ◆ Wash bottle with distilled water                              |

<sup>1-4</sup>To prepare the solutions and the unknown samples of  $\text{K}_2\text{SO}_4$  and  $\text{Na}_2\text{SO}_4$ , refer to the Lab Preparation section.

## **Concepts Students Should Already Know**

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Students should be familiar with the following concepts:

- ◆ Stoichiometry of chemical reactions
- ◆ Reactions of anions and cations
- ◆ Using the analytical balance

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 1: Determine the Empirical Formula of a Compound
- ◆ Lab 2: Determine the Percentage of Water in a Hydrate
- ◆ Lab 14a: Separation and Analysis of Cations
- ◆ Lab 14b: Analysis of Anions
- ◆ Lab 15b: Analysis of a Coordination Compound
- ◆ Lab 23: Determination of a Solubility Product

## **Using Your Data Collection System**

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a sensor to your data acquisition system ◆<sup>(2.1)</sup>
- ◆ Monitor live data without recording ◆<sup>(6.1)</sup>

## **Background**

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Gravimetric analysis is one of the oldest and most accurate quantitative methods for determining the amount of an analyte in a sample. Strategies usually involve transforming the analyte into a water insoluble form which precipitates out of solution and can be isolated by filtration and drying. The final stage of the analysis is obtaining the mass. From the measured mass and stoichiometric considerations, the mass, number of moles, and percentage of the sulfate content in the precipitate can be determined.

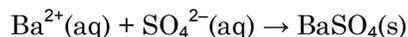
In this activity, the mass of a sample containing a sulfate of an alkali metal is measured and then dissolved in dilute hydrochloric acid. The sulfate is isolated by precipitation with barium chloride. The precipitate is digested in a heated solution to form coarser, easily filtered particles,

and to purify the precipitate. The precipitate is then collected by filtration, washed, dried, and its mass measured. The amount of sulfate in the original sample can be calculated from the mass of the precipitate and its chemical composition.

## Pre-Lab Activity

### Setting the stage for the activity

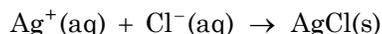
In this activity, you will analyze an unknown alkali sulfate sample to obtain the mass of the  $\text{SO}_4^{2-}$  content. You will remove the  $\text{SO}_4^{2-}$  ions with  $\text{Ba}^{2+}$  ions in the form of barium sulfate ( $\text{BaSO}_4$ ). By obtaining the mass of the dried precipitate, you can calculate the  $\text{SO}_4^{2-}$  content of the sample.



### Example calculation to try

0.3550 g of an alkali sulfate sample was dissolved in 50 mL of water in a 400-mL beaker. Five milliliters of 6 M HCl was added, followed by an additional 200 mL of water. The solution was heated to 90 °C and then 25 mL of 0.5 M barium chloride ( $\text{BaCl}_2$ ) solution was added. The solution was maintained at 90 °C for an hour.

The precipitate was filtered on an ashless filter paper and washed until the filtrate showed no reaction with  $\text{AgNO}_3$ . Any chloride contamination would yield white  $\text{AgCl}$  precipitate:



An empty crucible (35.446 g) was heated to a constant mass and was found to be 35.409 g. The wet precipitate was transferred into the crucible along with the filter paper and heated slowly until the paper was burned and the precipitate appeared to be dry. The mass of the cooled crucible with the precipitate was 36.055 g. After a second and third heating, the mass remained 36.020 g. The mass of the precipitate was

$$36.020 \text{ g} - 35.409 \text{ g} = 0.611 \text{ g}$$

which is

$$0.611 \text{ g BaSO}_4 \left( \frac{1 \text{ mol BaSO}_4}{233.39 \text{ g BaSO}_4} \right) = 2.62 \times 10^{-3} \text{ mol BaSO}_4$$

Therefore, there was  $2.62 \times 10^{-3}$  mol of  $\text{SO}_4^{2-}$ , which is

$$2.62 \times 10^{-3} \text{ mol SO}_4^{2-} \left( \frac{96.1 \text{ g SO}_4^{2-}}{1 \text{ mol SO}_4^{2-}} \right) = 0.252 \text{ g SO}_4^{2-}$$

The percent sulfate content was

$$\left( \frac{0.252 \text{ g}}{0.355 \text{ g}} \right) \times 100 = 71.0\%$$

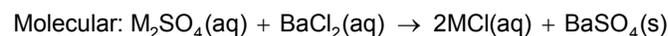
## Lab 16: Gravimetric Determination of a Precipitate

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Table 1: Determination of the amount of sulfate in the unknown

Parameter	Value
Mass of unknown sample (g)	0.355
Mass of precipitate (g)	0.611
Number of moles of precipitate (mol)	$2.62 \times 10^{-3}$
Number of moles of $\text{SO}_4^{2-}$ in precipitate (mol)	$2.62 \times 10^{-3}$
Mass of $\text{SO}_4^{2-}$ in precipitate (g)	0.252
Percent $\text{SO}_4^{2-}$ (%)	71.0

**1. What are the molecular, ionic, and net ionic equations of the reaction of the sample with  $\text{BaCl}_2$ ? Use “M<sup>+</sup>” to symbolize the metal ion.**



**2. Why do you think it is necessary to check the filtrate with  $\text{AgNO}_3$ ?**

We need to make sure that all  $\text{Cl}^-$  is washed out to prevent  $\text{Cl}^-$  contamination of the precipitate.

### Lab Preparation

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These are the materials and equipment to set up prior to the lab:

- 0.5000 M  $\text{BaCl}_2$ :** Dissolve 61.080 g of  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  in some water in a 500-mL volumetric flask and fill it to the mark.
- 0.1000 M  $\text{AgNO}_3$ :** Dissolve 1.700 g of  $\text{AgNO}_3$  in some water in a 100-mL volumetric flask and fill it to the mark.
- 6 M  $\text{HCl}$ :** Add 250 mL of 36%  $\text{HCl}$  solution slowly into about 200 mL of water in a 500-mL volumetric flask and fill it to the mark.
- Unknown samples:** Provide approximately 0.36 g of  $\text{K}_2\text{SO}_4$  or  $\text{Na}_2\text{SO}_4$  (or 0.80 g of  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ) per group in a weighing dish. Identify the unknowns by assigning each a number or use a preferred strategy.

## Safety

Add these important safety precautions to your normal laboratory procedures:

- ◆ If the HCl comes in contact with skin or eyes, rinse the contacted surface thoroughly with running water.
- ◆ Barium is a strong poison if ingested. Avoid contact with barium solution. In case of contact with skin, wash the barium off with plenty of water. If ingested, seek medical attention immediately.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

5	2	1	3	4
Transfer the precipitate into a crucible and heat it to constant mass. Identify the unknown and determine the percent sulfate in it.	Add BaCl <sub>2</sub> solution to cause the sulfate to precipitate.	Obtain the mass of a sample of an unknown alkali sulfate, dissolve it in 5.00 mL of water, then make the solution acidic.	Filter the precipitate with an ashless filter paper and wash it with hot water.	Check the filtered solution for Cl <sup>-</sup> ions with AgNO <sub>3</sub> solution. Wash the precipitate until no more forms.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box (☐) next to that step.

**Note:** When students see the symbol "◆" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

#### Prepare the unknown in solution

1. ☐ Make sure the 400-mL beaker, the stirring rod, rubber policeman, and the 100-mm watch glass are clean.
2. ☐ Obtain a weighing dish containing a sample of unknown substance from your instructor. Record the number below.

Sample number: \_\_\_\_\_

## Lab 16: Gravimetric Determination of a Precipitate

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3.  Carefully measure the mass by difference of approximately 0.350 g or 0.800 g of unknown, as instructed by your teacher, into the 400-mL beaker.

**Note:** You can achieve this by obtaining the mass of the unknown substance with the tared weighing dish and sample and then removing some of the sample directly into the beaker until the desired amount of the sample is transferred (that is, the remaining mass on the balance is less by the desired amount). Record the mass of the unknown substance to the nearest tenth of a milligram in Table 4.

Mass of unknown transferred to the beaker (g):           0.3580          

4.  Why do you measure the mass of the sample by difference rather than putting it straight into the beaker?

It is more accurate to measure from the original sample directly into the beaker (measuring by difference) than to measure the sample in the beaker (the beaker has rather large mass compared to the small sample) or to measure some of the sample into a weighing dish and transfer the sample into the beaker because, in that case, some of the sample is lost in the transfer.

5.  Add 50 mL of distilled water to the beaker and dissolve the sample with gentle swirling.

CAUTION: Be careful not to spill any of the solution.

6.  Slowly add 5 mL of 6 M HCl to the solution in the beaker.

7.  Add additional distilled water until there is about 250 mL of the solution in the beaker (use the scale on beaker).

8.  Cover the beaker with the 100-mm watch glass and, if necessary, store it in a safe place until you are ready to proceed with the determination.

### Form the precipitate

9.  Start a new experiment on the data collection system. ♦<sup>(1,2)</sup>

10.  Use the buret clamp to attach the buret to the ring stand.

11.  Connect the stainless steel temperature sensor to the data collection system. ♦<sup>(2,1)</sup>

- 12.**  Place the beaker with the solution on the hot plate and begin heating it to about 90 °C. Do not allow the solution to boil because some of the solution might be lost through spattering.

**Note:** Keeping the solution hot will promote the formation of large, filterable crystals and minimize the inclusion of impurities in the precipitate.

- 13.**  Immerse the temperature sensor in the solution so that it is not touching the beaker.

**Note:** When you remove the temperature sensor from the solution, be sure to rinse the solution adhering to the sensor back into the beaker with a small quantity of distilled water.

- 14.**  Why do you need to rinse any residue on the temperature sensor back into the beaker?

Rinsing is necessary to minimize the loss of the sample.

- 15.**  Set the data collection system to monitor live data without recording. <sup>(6.1)</sup>

- 16.**  When the solution has reached 90 °C, begin adding, drop by drop, the 0.5 M BaCl<sub>2</sub> solution from the buret into the beaker while you gently swirl the solution with the rubber policeman. Make sure you do not spill any of the solution.

- 17.**  When 25 to 35 mL of BaCl<sub>2</sub> solution has been added, stop the addition and let the precipitate settle.

- 18.**  When the precipitate has settled, test for completeness of precipitation by adding 1 to 2 drops of BaCl<sub>2</sub>. If you see any sign of precipitation, add 5 more milliliters of BaCl<sub>2</sub>.

- 19.**  Why is it necessary to test for completeness?

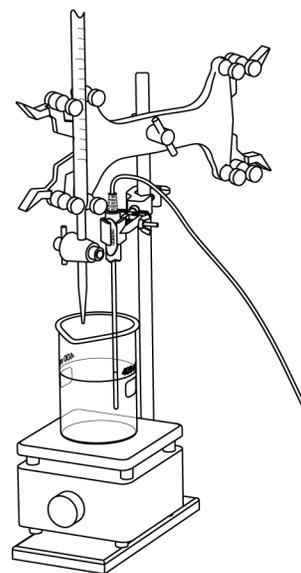
Testing for completeness assures that all sulfate ions are removed quantitatively from the solution.

- 20.**  After adding a sufficient amount of BaCl<sub>2</sub> to the beaker, rinse the rubber policeman over the beaker with the solution with a few drops of deionized water. Cover the beaker with a watch glass and continue heating at 90 °C for one hour. After this “digestion,” the precipitate should be coarse and the supernatant should be clear.

### Collect the precipitate

- 21.**  Fold a piece of Whatman® Ashless #42 filter paper by following the steps below.

- a. Fold the filter paper in half and fold that in half again.
- b. Hold three of the four parts of the filter paper on one side.



## Lab 16: Gravimetric Determination of a Precipitate

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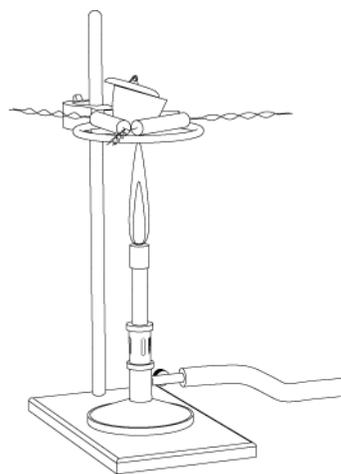
- c. Pull out the fourth part of the filter paper and form a cone.
  - d. Place the cone-shaped paper into the funnel.
  - e. Wet the paper with a few drops of distilled water so the paper sticks to the wall of the funnel.
22.  Support the funnel in the ring mounted on a ring stand over a beaker or flask of sufficient size (400-mL) to hold all of the supernatant.

### Collect Data

23.  In the 250-mL beaker, heat 100 mL of distilled water to about 80 °C for rinsing the precipitate.
24.  Pour the mixture with the precipitate through the funnel with the filter paper.
25.  Use a rubber policeman and small washes of hot distilled water to remove any remaining precipitate from the beaker.
26.  Wash the material in the funnel with three 5-mL portions of the hot, distilled water. Collect the washes separately in the small, 25-mL beakers.
27.  Add two drops of the  $\text{AgNO}_3$  solution to the last wash. If any cloudy white precipitate is observed, the precipitate still contains chloride ions and must be washed with hot distilled water a fourth time in a rinsed beaker.

### Dry the precipitate

28.  Clean and dry a porcelain crucible and lid. Then prepare the crucible as follows:
- a. Place the crucible and lid on the clay triangle over the Bunsen burner.
  - b. Heat the crucible with a gentle flame for 5 minutes by moving the burner around the bottom of the crucible.
  - c. After the bottom of the crucible has become red-hot, increase the flame by allowing more air into the burner.
  - d. Continue moving the burner around the bottom of the crucible.
  - e. Heat the crucible for 10 to 12 minutes.
  - f. Turn off the burner and allow the crucible to cool to room temperature.



**Note:** For the rest of the experiment, handle the crucible and lid using only crucible tongs. Also, do not set the crucible on the lab bench or it may crack or become contaminated.

29.  Why do you have to use tongs to hold the lid?

Touching the lid with bare hands contaminates the lid and changes its mass, introducing error in the mass measurement. Also, the crucible may still be hot and cause injury.

30.  After the crucible has cooled, measure and record the mass of the “fired” crucible, together with its lid, in Table 2.

Table 2: Mass of the empty crucible measured to the nearest tenth of a milligram

Measurement	Mass
Crucible and lid before heating (g)	31.3714
Crucible and lid after the first heating (g)	31.3576
Crucible and lid after the second heating (g)	31.3501
Crucible and lid after the third heating (g)	
Crucible and lid after the fourth heating (g)	

31.  Repeat the steps above for heating, cooling, and measuring the mass of the crucible and lid until you have two readings for the mass that are within 10 mg of each other.

32.  Copy the last measurement in Table 2 into Table 4.

33.  Remove the filter paper containing the precipitate from the funnel. *Carefully* fold the filter paper so that the precipitate is trapped inside and the final shape is small enough to be placed into the crucible.

**Note:** To prevent the loss of precipitate, avoid tearing or breaking the folded filter paper.

34.  Place the crucible on a wire triangle on a ring stand over a Bunsen burner.

35.  Heat the crucible with a small flame so the filter paper does not burst into flame.

**Note:** Keep the tongs and crucible lid ready to quickly cover the crucible and extinguish any flame from the paper before material is expelled from the crucible.

36.  Move the burner around and gradually increase the size of the flame so that all parts of the crucible are heated.

37.  When all of the carbon residue from the filter paper has been removed, the temperature should be maximized by bringing the tip of the blue cone of the flame to a point just below the crucible. Continue heating the crucible for ten minutes.

38.  Allow the crucible to cool for a few minutes and then place it into a desiccator, if available, using crucible tongs. Allow it to cool to room temperature. Measure and record the mass of the crucible, lid, and precipitate in Table 3.

## Lab 16: Gravimetric Determination of a Precipitate

Table 3: Mass of the crucible and sample, measured to the nearest tenth of a milligram

Measurement	Mass
Crucible, lid, and precipitate before heating (g)	32.8005
Crucible, lid, and precipitate after the first heating (g)	31.7656
Crucible, lid, and precipitate after the second heating (g)	31.7605
Crucible, lid, and precipitate after the third heating (g)	
Crucible, lid, and precipitate after the fourth heating (g)	

39.  Repeat the heating, cooling, and measuring procedure until two successive mass measurements are within 10 mg of each other.
40.  Record all measurements in Table 3. Copy the final measurement into Table 4.
41.  You do not need to save your experiment in this activity.
42.  Clean up according to your teacher's instructions.

### Data Analysis

1.  Calculate the mass of the dried precipitate from the mass of the empty crucible and the mass of the crucible with the dried precipitate. Record the value in Table 4.

$$31.7605 \text{ g} - 31.3501 \text{ g} = 0.4104 \text{ g}$$

2.  Calculate the number of moles of precipitate from its mass and formula weight. Record the value in Table 4.

$$(0.4104 \text{ g BaSO}_4) \left( \frac{1 \text{ mol BaSO}_4}{233.43 \text{ g BaSO}_4} \right) = 1.758 \times 10^{-3} \text{ mol BaSO}_4$$

3.  Calculate the number of moles of sulfate ions in the precipitate from the number of moles of precipitate. Record the value in Table 4.

The number of moles of sulfate ions is the same as the number of moles of precipitate since there is 1 mol of sulfate in 1 mol of precipitate. Therefore, there is  $1.758 \times 10^{-3}$  mol of sulfate ions in the precipitate.

4.  Calculate the mass of the sulfate ions from the number of moles of sulfate ions. Record the value in Table 4.

$$(1.758 \times 10^{-3} \text{ mol SO}_4^{2-}) \left( \frac{96.10 \text{ g SO}_4^{2-}}{1 \text{ mol SO}_4^{2-}} \right) = 0.1688 \text{ g SO}_4^{2-}$$

5. □ Calculate the theoretical percentage of sulfate content in the unknown sulfate sample based on the mass of the sulfate ions and the total mass of the sample. Record the value in Table 4.

$$\left( \frac{0.1688 \text{ g}}{0.3580 \text{ g}} \right) \times 100 = 47.15\%$$

Table 4: Determination of the amount of sulfate in the unknown

Parameter	Value
Mass of unknown sample (g)	0.3580
Mass of the crucible and lid (g)	31.3501
Mass of the crucible, lid, and precipitate (g)	31.7605
Mass of precipitate (g)	0.4104
Number of moles of precipitate (mol)	$1.758 \times 10^{-3}$
Number of moles of $\text{SO}_4^{2-}$ in precipitate (mol)	$1.758 \times 10^{-3}$
Mass of $\text{SO}_4^{2-}$ in precipitate (g)	0.1688
Percent $\text{SO}_4^{2-}$ (%)	47.15

## Lab 16: Gravimetric Determination of a Precipitate

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6.  Complete the table below and use it to identify the unknown. Consider that the unknown is the sulfate of either an alkali or alkaline earth metal. Consider also that alkaline earth sulfates are not very soluble in water with the exception of  $\text{MgSO}_4$ . Select the one that is closest to your experimental result.

Table 5: Possible unknowns

Formula	Formula Weight (g/mol)	$\text{SO}_4^{2-}$ Content (%)	Solubility in Water
$\text{Li}_2\text{SO}_4$	109.9	87.37	Soluble
$\text{Na}_2\text{SO}_4$	142.0	67.62	Soluble
$\text{Na}_2\text{SO}_4 \cdot \text{H}_2\text{O}$	322.0	29.81	Soluble
$\text{K}_2\text{SO}_4$	174.2	55.11	Soluble
$\text{CaSO}_4$	120.3	79.80	Slightly soluble
$\text{MgSO}_4$	136.1	70.54	Soluble
$\text{SrSO}_4$	183.6	52.29	Slightly soluble

Based on the data in Table 5, possible unknowns are  $\text{K}_2\text{SO}_4$  and  $\text{SrSO}_4$ . Considering the fact that the unknown is extremely soluble in water,  $\text{SrSO}_4$  can be ruled out since it is an alkaline earth metal sulfate. Therefore, most likely the unknown was  $\text{K}_2\text{SO}_4$ .

7.  Calculate the percent error based on the experimental value of the percent sulfate content of the sample and the percent sulfate content of the proposed unknown.

$$\text{Percent Error} = \frac{|\text{Theoretical Value} - \text{Experimental Value}|}{\text{Theoretical Value}} \times 100$$

$$\text{Percent Error} = \frac{|55.11 - 47.17|}{55.11} \times 100 = 14.41\%$$

### Analysis Questions

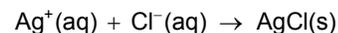
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1. If ordinary filter paper had been used instead of ashless paper, how would your results be affected? Explain.

The ash residue would have contributed to the mass, resulting in error.

2. Why are the washes of the precipitate tested with  $\text{AgNO}_3$ ?

$\text{AgNO}_3$  forms white  $\text{AgCl}$  precipitate with  $\text{Cl}^-$  ions:



To make sure all  $\text{Cl}^-$  ions are removed the filtrate needed to be tested.  $\text{Cl}^-$  ions would have introduced an error.

**3. What are the most likely sources of error in your procedure? Would they cause your result to be high or low?**

Loss of precipitate during transfer would have yielded less precipitate and a lower percentage of sulfate content. If, however, the precipitate was not dried completely, that would have yielded higher mass and higher percent sulfate content.

**4. Why do we have to make sure that the crystals are big?**

Small crystals clog the filter paper, making the filtration process much longer.

**5. Explain how you would determine the percent sulfate content in a  $\text{CaSO}_4$  sample, since it is not very water soluble?**

The sample would have to be converted to a water soluble form with, for example, HCl. This would produce  $\text{CaCl}_2$ , which is water soluble. Then the sulfate ions can be removed with  $\text{BaCl}_2$  solution in the form of  $\text{BaSO}_4$  as in this activity.

**Synthesis Questions**

Use available resources to help you answer the following questions.

**1. Which other cation, if any, could have been used instead of  $\text{Ba}^{2+}$  to form a precipitate with the  $\text{SO}_4^{2-}$  ions?**

$\text{Pb}^{2+}$  ions would have worked, except  $\text{Pb}(\text{NO}_3)_2$  solution would have been necessary because  $\text{PbCl}_2$  is not soluble in water at room temperature.

**2. What method might you use to analyze a sample that has both  $\text{SO}_4^{2-}$  and  $\text{PO}_4^{3-}$  ions? (Hint: Can you recall what is soluble in  $\text{Ba}_3(\text{PO}_4)_2$  that is not soluble in  $\text{BaSO}_4$ ?)**

The analysis would need to be performed one time as described above. This would provide the total amount of  $\text{SO}_4^{2-}$  and  $\text{PO}_4^{3-}$  content. For the second analysis, after the precipitate was made, excess HCl solution can be added to dissolve the  $\text{PO}_4^{3-}$  ions. Only  $\text{BaSO}_4$  remains as precipitate, therefore the  $\text{SO}_4^{2-}$  content can be obtained. The difference between the sulfate content and the total amount of  $\text{SO}_4^{2-}$  and  $\text{PO}_4^{3-}$  content provides the  $\text{PO}_4^{3-}$  content.

**Multiple Choice Questions**

Select the best answer or completion to each of the questions or incomplete statements below.

**1. Adding an excess amount of  $\text{BaCl}_2$  solution:**

- A.** Results in an error because the excess amount will show up in the precipitate.
- B.** Makes no difference if the precipitate is thoroughly washed with water.
- C.** Is necessary for completion of the precipitation.
- D.** Ruins the experiment.

**2. Incomplete precipitation results in:**

- A. Higher sulfate content than expected.
- B. Lower sulfate content than expected.**
- C. Accurate sulfate content.
- D. Unpredictable results.

**3. Incomplete transfer of precipitate from the beaker to the funnel results in:**

- A. Higher sulfate content than expected.
- B. Lower sulfate content than expected.**
- C. Accurate sulfate content.
- D. Unpredictable results.

**4. Incomplete washing of the precipitate and incomplete drying of the precipitate:**

- A. Have no effect on the results.
- B. Increases the sulfate content.
- C. Lowers the sulfate content.
- D. Have opposite effects, as the incomplete drying increases the mass while the incomplete washing decreases the expected sulfate content.**

### Extended Inquiry Suggestions

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The analysis of a mixture of two different alkali sulfates can be performed and the challenge is to determine the percentage of the components of the mixture:

$$n_1 + n_2 = n$$
$$(2AW_{M_1} + 96)n_1 + (2AW_{M_2} + 96)n_2 = m$$

where

$n_1$  = number of moles of one of the alkali sulfate in the sample that was measured (mol)

$n_2$  = number of moles of the other alkali sulfate in the sample that was measured (mol)

$AW_{M_1}$  = atomic weight of the first alkali metal (g/mol)

$2AW_{M_1} + 96$  = formula weight of first alkali sulfate (g/mol)

$AW_{M_2}$  = atomic weight of the second alkali metal (g/mol)

$2AW_{M_2} + 96$  = formula weight of second alkali sulfate (g/mol)

$m$  = mass of the sample that was measured (g)

$n$  = total number of moles of  $BaSO_4$  which is the same as the total number of moles of  $SO_4^{2-}$  in the sample (mol)

There are two equations with two unknowns to solve.

## Lab 17a: Absorption Spectra

### Objectives

Students learn about the composition of the electromagnetic radiation in the visible range, develop an understanding of how the interaction of objects and solutions with light result in the perception of color, and dispel their misconception of objects "having color".

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Using a simple spectrophotometer and making spectroscopic measurements
- ◆ Applying color charts to determine the wavelengths transmitted and absorbed that result in various colors

### Time Requirement

◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	30 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

#### *For each student or group:*

- |  |  |
|--|--|
| ◆ Data collection system <sup>1</sup>                              | ◆ 0.1 M Copper(II) chloride (CuCl <sub>2</sub> ), 10 mL <sup>3</sup> |
| ◆ Spectrometer   | ◆ 0.1 M Cobalt(II) chloride (CoCl <sub>2</sub> ), 10mL <sup>4</sup>  |
| ◆ Cuvette  | ◆ 0.1 M Nickel(II) chloride (NiCl <sub>2</sub> ), 10mL <sup>5</sup>  |
| ◆ Sensor extension cable   | ◆ 0.1 M Sodium chloride (NaCl), 10 mL <sup>6</sup>                   |
| ◆ Test tubes, large (6)  | ◆ Color chart <sup>7</sup>   |
| ◆ Test tube rack   | ◆ Wash bottle with distilled water                                   |
| ◆ Graduated cylinder, 10-mL  | ◆ Marking pen  |
| ◆ 0.1 M Iron(III) chloride (FeCl <sub>3</sub> ), 10mL <sup>2</sup> |  |

<sup>1</sup> Use either Xplorer GLX™ or a computer with Quantum software for the Amadeus spectrometer system.

<sup>2-6</sup> To prepare the solutions, refer to the Lab Preparation section.

<sup>7</sup> To present the color chart, refer to the Lab Preparation section.

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Moles
- ◆ Molarity
- ◆ Ionic nomenclature
- ◆ Electrolytes
- ◆ Absorbance, Beer's Law

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 10: Determine the Equilibrium Constant for a Chemical Reaction
- ◆ Lab 17b: Colorimetric Analysis
- ◆ Lab 18: Separation by Liquid Chromatography
- ◆ Lab 25: Order of Reaction

## Using Your Data Collection System

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting sensors to the data collection system ◆<sup>(2.1)</sup>
- ◆ Setting up the spectrometer ◆<sup>(4.4)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>
- ◆ Displaying all data runs ◆<sup>(7.1.3)</sup>
- ◆ Printing ◆<sup>(11.2)</sup>
- ◆ Saving your experiment ◆<sup>(11.1)</sup>

## Background

It is a common misconception that the apparent color of objects is, in fact, a property of the objects themselves. The perception of the color of the object is the color of the light that is *reflected* by the object or *transmitted* by a solution. Regular white light has the full visible spectrum, approximately 380–760 nanometers (nm).



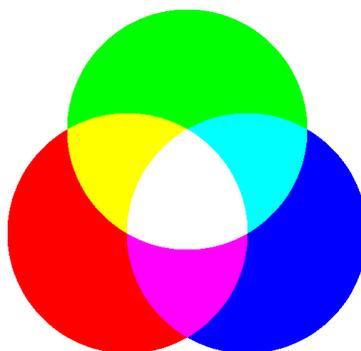
When light hits an object, the object absorbs some of the light waves and the rest of the light is reflected; this is the portion that we can see. If light is transmitted through a solution, photons with a certain wavelength can be absorbed by species in the solution, the rest of the light is transmitted through it, which we can then observe.

## Pre-Lab Activity

### Setting the stage for the activity

In actuality, it rarely happens that an object shows a color because all the other colors are absorbed. Instead, various components of the white light are absorbed to various extents, resulting in an array of visible colors. A thorough discussion of colors would be beyond this activity; we need, however, to touch upon the topic in order to help you understand how absorption spectra are made.

There are three primary colors when discussing light: red, green, and blue. The combination of all three of these colors results in white (see the color chart). Now, if all the blue is absorbed, only the green and red light get to your eyes. As a result, you will see yellow.



Also, if only one color is absorbed, then the mixture of the photons of the other two colors is visible. So if only the blue photons are absorbed, the red and green photons are observed as yellow. To make matters more complicated, not all the photons of a specifically-colored light necessarily have to be absorbed.

We can use a scale between 0 and 15 to describe the portion of photons of the particular color of the light that enters your eyes. For example, a light described by 15-red, 15-blue, and 15-green means that all the three colors are present in full; there was no absorption. Also, 10-red, 15-blue, and 5-green describes a light in which some of the red (5) photons and almost all of the green (10) photons were absorbed, but none of the blue photons were.

The chart below shows the different colors that result from various partial and full absorption of the primary colors. The top sequence of numbers represents (from left to right), the amount of blue, green, and red photons transmitted (present) on a 0 to 15 scale. The bottom sequence of numbers represents (from left to right) the amount of blue, green, and red photons absorbed on a 0 to 15 scale. For example, 10–15–5 in the top row means that the amount of blue present is 10, the amount of green is 15, and the amount of red is 5. The bottom set of numbers in the same block would be 5–0–10, indicating that 5 photons of blue, 0 photons of green, and 10 photons of red were absorbed.

Color chart

0-0-0	3-0-0	6-0-0	9-0-0	12-0-0	15-0-0	0-0-6	3-0-6	6-0-6	9-0-6	12-0-6	15-0-6	0-0-12	3-0-12	6-0-12	9-0-12	12-0-12	15-0-12
15-15-15	12-15-15	9-15-15	6-15-15	3-15-15	0-15-15	15-15-9	12-15-9	9-15-9	6-15-9	3-15-9	0-15-9	15-15-3	12-15-3	9-15-3	6-15-3	3-15-3	0-15-3
0-3-0	3-3-0	6-3-0	9-3-0	12-3-0	15-3-0	0-3-6	3-3-6	6-3-6	9-3-6	12-3-6	15-3-6	0-3-12	3-3-12	6-3-12	9-3-12	12-3-12	15-3-12
15-12-15	12-12-15	9-12-15	6-12-15	3-12-15	0-12-15	15-12-9	12-12-9	9-12-9	6-12-9	3-12-9	0-12-9	15-12-3	12-12-3	9-12-3	6-12-3	3-12-3	0-12-3
0-6-0	3-6-0	6-6-0	9-6-0	12-6-0	15-6-0	0-6-6	3-6-6	6-6-6	9-6-6	12-6-6	15-6-6	0-6-12	3-6-12	6-6-12	9-6-12	12-6-12	15-6-12
15-9-15	12-9-15	9-9-15	6-9-15	3-9-15	0-9-15	15-9-9	12-9-9	9-9-9	6-9-9	3-9-9	0-9-9	15-9-3	12-9-3	9-9-3	6-9-3	3-9-3	0-9-3
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15-0-15	12-0-15	9-0-15	6-0-15	3-0-15	0-0-15	15-0-9	12-0-9	9-0-9	6-0-9	3-0-9	0-0-9	15-0-3	12-0-3	9-0-3	6-0-3	3-0-3	0-0-3
0-0-3	3-0-3	6-0-3	9-0-3	12-0-3	15-0-3	0-0-9	3-0-9	6-0-9	9-0-9	12-0-9	15-0-9	0-0-15	3-0-15	6-0-15	9-0-15	12-0-15	15-0-15
15-15-12	12-15-12	9-15-12	6-15-12	3-15-12	0-15-12	15-15-6	12-15-6	9-15-6	6-15-6	3-15-6	0-15-6	15-15-0	12-15-0	9-15-0	6-15-0	3-15-0	0-15-0
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3-15-3	6-15-3	9-15-3	12-15-3	15-15-3	0-15-9	3-15-9	6-15-9	9-15-9	12-15-9	15-15-9	0-15-15	3-15-15	6-15-15	9-15-15	12-15-15	15-15-15	0-0-0
15-0-12	12-0-12	9-0-12	6-0-12	3-0-12	0-0-12	15-0-6	12-0-6	9-0-6	6-0-6	3-0-6	0-0-6	15-0-0	12-0-0	9-0-0	6-0-0	3-0-0	0-0-0

To characterize the ability of solutions to absorb light we use two physical quantities: transmittance  $T$  and absorbance  $A$ . Transmittance is defined as

$$T \equiv \frac{I}{I_0}$$

where  $I$  is the number of transmitted photons (the intensity) in unit time with the absorbing species present, and  $I_0$  is the number of transmitted photons in unit time with the absorbing species absent. As it is rather difficult to work with the number of photons, absorbance is used instead:

$$A \equiv -\log T = -\log \frac{I}{I_0}$$

It is important to keep in mind that absorption depends on wavelength, and therefore is color specific. That is, the ability of a substance to absorb light is different for photons with different wavelength.

In this activity, you will study five solutions of different colors and predict what the absorption spectra of the five solutions looks like.

**Example calculation to try**

We compared three solutions:  $\text{NaNO}_3$ ,  $\text{Cu(NO}_3)_2$ , and  $\text{Co(NO}_3)_2$ . The following table indicates the observed colors:

Table 1: Colors of various solutions

Solution	Color
$\text{NaNO}_3$	Colorless
$\text{Cu(NO}_3)_2$	Blue
$\text{Co(NO}_3)_2$	Pink/purple

Since all three substances are strong electrolytes, they all dissociate completely. As each solution has a different color, the color cannot be attributed to the anion,  $\text{NO}_3^-$ . Therefore, the different colors are associated with the different metal ions.

Since the  $\text{NaNO}_3$  solution is colorless, we can conclude that the sodium ion does not absorb any photons in the visible spectrum. Since the  $\text{Cu(NO}_3)_2$  solution is blue, we would guess that the  $\text{Cu}^{2+}$  ions don't absorb the blue photons and do absorb the red and green photons.

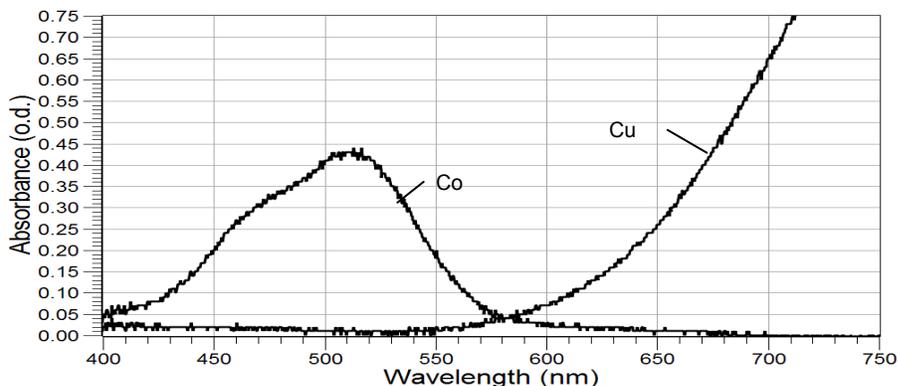
As predicted, in the absorption spectrum of the blue range (430–490 nm) we find no significant absorbance. Likewise, we assume that in the green range (510–550 nm) and in the red range (above 660 nm) there must be significant absorption. However, as you can see on the graph below, there is no significant absorption in the green range (510–550 nm), only in the red range. This means that both green and blue photons are transmitted. The green and blue photons make a color called cyan blue and indeed, the solution of  $\text{Cu(NO}_3)_2$  is light green-blue.

Table 2: Wavelengths of white and the primary colors

Color Range	Wavelength (nm)
White	380–760
Blue	430–490
Green	510–550
Red	>660

The  $\text{Co(NO}_3)_2$  solution is a dark pink or light purple. Matching the color of the solution to the color chart, the color matches the 15–0–15 block (rightmost column in the middle of the color chart) for the components transmitted. This indicates significant absorbance in the green and little or no absorbance in the red and blue areas. Indeed, the absorption spectrum below shows this behavior.

Absorption spectra



**1. If an object appears to be bright red, what wavelength range do you think it reflects? Explain!**

Bright red color suggests that only red photons are reflected. Red photons are above 600 nm in wavelength.

**2. What wavelength range do you think it absorbs? Explain!**

It must absorb the green and blue photons which have wavelengths in the range of 430–600 nm.

**3. Record in Table 3 the color of the solutions you will be working with, as well as the ions and molecules present in each solution.**

Table 3: Solution composition and color

Solution	Color	Ions, Molecules
CoCl <sub>2</sub>	Pink/purple	Co <sup>2+</sup> , Cl <sup>-</sup>
NiCl <sub>2</sub>	Green	Ni <sup>2+</sup> , Cl <sup>-</sup>
FeCl <sub>3</sub>	Faint yellow	Fe <sup>3+</sup> , Cl <sup>-</sup>
CuCl <sub>2</sub>	Cyan	Cu <sup>2+</sup> , Cl <sup>-</sup>
NaCl	Colorless	Na <sup>+</sup> , Cl <sup>-</sup>

**4. Based on the colors of the solutions, complete the table, predicting the transmitted colors and the colors and corresponding wavelength ranges of the light absorbed!**

Table 4: Solution color predictions

Solution	Visible Color	Transmitted Primary Colors	Absorbed Primary Colors	Wavelength Ranges Absorbed
$\text{CoCl}_2$	Purple/pink	Blue and Red	Some blue, all green and some red	380–500 nm (blue), some 500–550 nm (green), all 650–750 nm (red) a some
$\text{NiCl}_2$	Green	Much green and a little blue	Some of blue, all of red	380–500 nm (some blue) 650–750 nm (all red)
$\text{FeCl}_3$	Yellow	Some green, some red	All blue, some red	380–500 nm (most blue) 650–750 nm (some red)
$\text{CuCl}_2$	Blue	Much blue, some green	All red, some green	650–750 nm (all red) 500–550 nm (some green)
$\text{NaCl}$	Clear	All	None	None

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

- 0.10 M  $\text{FeCl}_3$ :** Dissolve 13.51g of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  in some water in a 500-mL volumetric flask and fill the flask to the mark.
- 0.10 M  $\text{CuCl}_2$ :** Dissolve 8.52g of  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  in some water in a 500-mL volumetric flask and fill the flask to the mark.
- 0.10 M  $\text{CoCl}_2$ :** Dissolve 11.89g of  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$  in some water in a 500-mL volumetric flask and fill the flask to the mark.
- 0.10 M  $\text{NiCl}_2$ :** Dissolve 11.88g of  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  in some water in a 500-mL volumetric flask and fill the flask to the mark.
- 0.10 M  $\text{NaCl}$ :** Dissolve 2.92g of  $\text{NaCl}$  in some water in a 500-mL volumetric flask and fill the flask to the mark.
- Print or project the color images of this activity from the last page of the source file contained in the accompanying CD.

## Safety

Follow all standard laboratory procedures.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

5	4	1	3	2
Record and analyze the data.	Fill the cuvette with the first solution and measure the absorbance. Do this for the remaining solutions.	Set up the data collection system and connect the spectrometer.	Configure the display to graph absorbance versus wavelength. Start recording the data.	Then fill the test tubes with the different solutions.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "♦" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

- Start a new experiment on the data collection system. ♦<sup>(1,2)</sup>
- Set up the spectrometer. ♦<sup>(4,4)</sup>
- Add about 10 mL of each of the 5 solutions into separate, labeled test tubes.
- Are the solutions strong electrolytes, weak electrolytes, or non-electrolytes? List the ions and molecules present in the solutions.

The solutions are made from ionic compounds which are strong electrolytes; upon solution they dissociate to form ions. In this solution the ions formed are  $\text{Fe}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Na}^+$ ,  $\text{Co}^+$ , and  $\text{Cl}^-$ . Of course, water molecules are present as well.

5.  Observe the color of the solutions. Based on your observation and on your answer to the previous question, what ions do you think are responsible for the colors of the solutions?

Since every solution is a different color and every solution has  $\text{H}_2\text{O}$  and  $\text{Cl}^-$  ions, those species cannot be responsible for the color difference. Therefore, the colors have to be determined by the cations.

### Collect Data

6.  Display Absorbance on the y-axis of a graph with Wavelength on the x-axis.  $\diamond^{(7.1.1)}$

7.  Start data recording.  $\diamond^{(6.2)}$

8.  Measure the absorbance of the five solutions following the steps below.

a. Rinse the cuvette with a small portion of the first solution and fill the cuvette two-thirds full. Wipe the cuvette clean and dry and place it into the spectrometer.

b. Why do you have to rinse the cell with some of the solution?

If there is any residual water in the cuvette, it will dilute the concentration of the solution and falsify the data.

c. Dispose of the solution and rinse the cell thoroughly with water.

d. Why do you think it is important to rinse the cell thoroughly between measurements?

You need to rinse the cell to avoid contamination of the solutions.

9.  Stop data recording.  $\diamond^{(6.2)}$

10.  Display all data runs.  $\diamond^{(7.1.3)}$

11.  Print the graph.  $\diamond^{(11.2)}$

12.  Save your experiment  $\diamond^{(11.1)}$  and clean up according to your teacher's instructions.

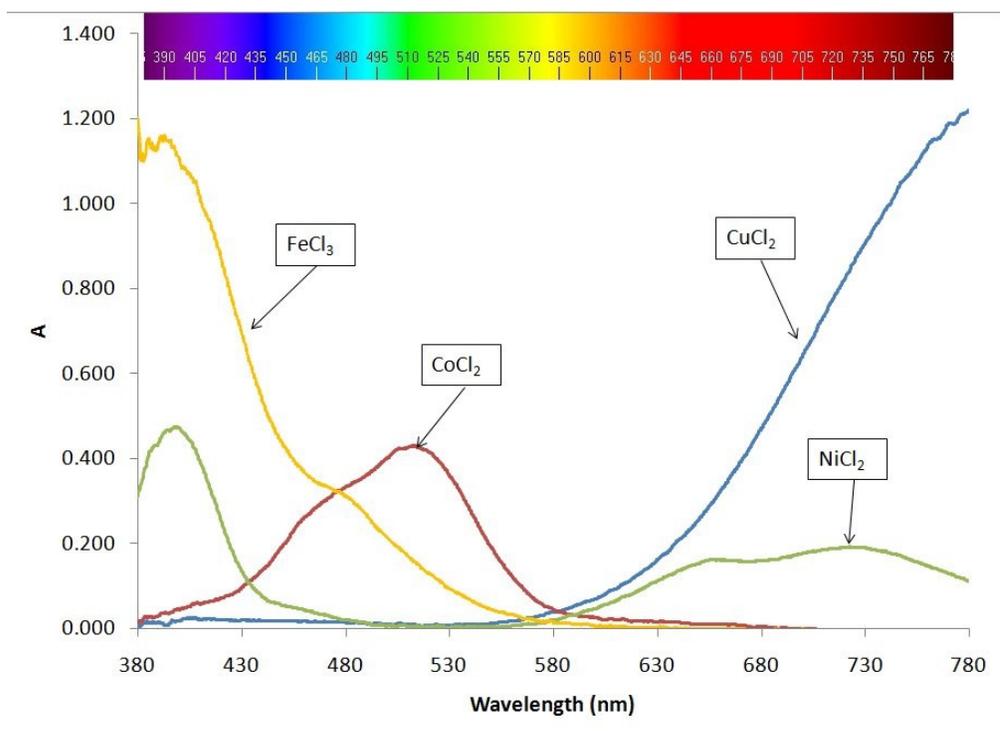
## Data Analysis

1.  Summarize your data in the table below.

Table 5: Absorption data

Solution	Visible Color	Predicted Absorption Wavelength Ranges	Wavelength Ranges Absorbed
CoCl <sub>2</sub>	Red	380–500 nm (some blue) 500–550 nm (all green) 650–750 nm (some red)	380–600 nm (blue, green)
NiCl <sub>2</sub>	Green	380–500 nm (some blue) 650–750 nm (all red)	380–475 nm (some blue) 575–750 nm (red all)
FeCl <sub>3</sub>	Yellow	380–500 nm (most blue) 650–750 nm (some red)	380–575 nm (all blue and green)
CuCl <sub>2</sub>	Blue	650–750 nm (all red) 500–550 nm (some green)	575–750 nm (all red)
NaCl	Clear	none	none

2.  Sketch or paste the spectrum curves for the five solutions.



### Analysis Questions

1. Find the color of the FeCl<sub>3</sub> solution on the color chart. What portions of the red, green, and blue photons are absorbed? Does it match with the absorption spectrum of your solution?

The best matching color shows the presence of blue 0, green 12, and red 15. The absorbed blue is 15, green is 3 and red is 0. This absorption pattern represents high absorption in the blue, some absorption in the green and no absorption in the red range. The absorption spectrum for the solution is in agreement with this.

2. Find the color of the NiCl<sub>2</sub> solution on the color chart. What portions of the red, green, and blue photons are absorbed? Does it match with the obtained absorption spectrum?

The absorbed blue is 12, green is 3 and red is 12. This absorption pattern represents high absorption in the blue, no absorption in the green and significant absorption in the red range. The absorption spectrum obtained is in agreement with this.

### Synthesis Questions

Use available resources to help you answer the following questions.

1. The solution of KMnO<sub>4</sub> is purple. Describe the absorption spectrum of MnO<sub>4</sub>!

The purple color is most likely a blend of red and blue light suggesting the absorption of green. Therefore it is most likely that there will be little or no absorption in the blue and red range and significant absorption in the green range.

**2. An absorption spectrum shows significant absorption in the blue and little or no absorption in the green and red range. What color do you think the solution is?**

The remaining green and red make some shade of yellow.

### Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

**1. A colorless solution:**

- A. Absorbs no photons from the white light.**
- B. Emits photons to make up white light.**
- C. Absorbs all photons; therefore we cannot observe any colors.**
- D. Cannot transmit light.**

**2.  $\text{MnSO}_4$  has a very faint yellow color. Which color of photons do you think are *not* absorbed?**

- A. Blue and green**
- B. Red and blue**
- C. Red and green.**
- D. Blue and red.**

**3. The Liquid Chromatography experiment analyzes grape Kool-Aid. The analysis shows both a red and a light blue component. Which statement cannot be correct regarding the two colored components?**

- A. The red and light blue, once mixed, gives a colorless solution.**
- B. The red component has significant absorbance in the green and blue range**
- C. The mix of the red and blue components will not contain green photons.**
- D. The blue component has significant absorbance in the red range.**

### Extended Inquiry Suggestions

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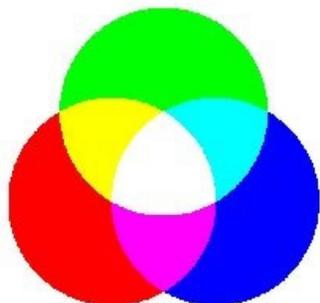
The eluent solution from the Liquid Chromatography experiment can be analyzed with the spectrometer. Since the red component absorbs the green and blue ranges, and the blue component absorbs in the red and green ranges, the process can be followed by monitoring the absorption spectrum of the eluent leaving the column.

Eluent aliquots of ~0.5mL can be collected and analyzed. The reconstructed graph of absorbance versus time (or volume) represents the chromatogram. This process demonstrates how High Performance Liquid Chromatography (HPLC) works with an absorbance detector. HPLC is one of the most commonly used techniques today. For details, please refer to the activity "Lab 18: Separation by Liquid Chromatography".

## Absorption Spectra Color Images



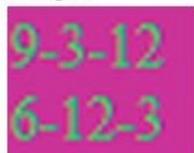
### The three primary colors and the various color combinations



### The color chart

0-0-0	3-0-0	6-0-0	9-0-0	12-0-0	15-0-0	0-0-6	3-0-6	6-0-6	9-0-6	12-0-6	15-0-6	0-0-12	3-0-12	6-0-12	9-0-12	12-0-12	15-0-12
15-15-15	12-15-15	9-15-15	6-15-15	3-15-15	0-15-15	15-15-0	12-15-0	9-15-0	6-15-0	3-15-0	0-15-0	15-15-3	12-15-3	9-15-3	6-15-3	3-15-3	0-15-3
0-3-0	3-3-0	6-3-0	9-3-0	12-3-0	15-3-0	0-3-6	3-3-6	6-3-6	9-3-6	12-3-6	15-3-6	0-3-12	3-3-12	6-3-12	9-3-12	12-3-12	15-3-12
15-12-15	12-12-15	9-12-15	6-12-15	3-12-15	0-12-15	15-12-9	12-12-9	9-12-9	6-12-9	3-12-9	0-12-9	15-12-3	12-12-3	9-12-3	6-12-3	3-12-3	0-12-3
0-6-0	3-6-0	6-6-0	9-6-0	12-6-0	15-6-0	0-6-6	3-6-6	6-6-6	9-6-6	12-6-6	15-6-6	0-6-12	3-6-12	6-6-12	9-6-12	12-6-12	15-6-12
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0-9-0	3-9-0	6-9-0	9-9-0	12-9-0	15-9-0	0-9-6	3-9-6	6-9-6	9-9-6	12-9-6	15-9-6	0-9-12	3-9-12	6-9-12	9-9-12	12-9-12	15-9-12
15-6-15	12-6-15	9-6-15	6-6-15	3-6-15	0-6-15	15-6-9	12-6-9	9-6-9	6-6-9	3-6-9	0-6-9	15-6-3	12-6-3	9-6-3	6-6-3	3-6-3	0-6-3
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15-3-15	12-3-15	9-3-15	6-3-15	3-3-15	0-3-15	15-3-9	12-3-9	9-3-9	6-3-9	3-3-9	0-3-9	15-3-3	12-3-3	9-3-3	6-3-3	3-3-3	0-3-3
0-15-0	3-15-0	6-15-0	9-15-0	12-15-0	15-15-0	0-15-6	3-15-6	6-15-6	9-15-6	12-15-6	15-15-6	0-15-12	3-15-12	6-15-12	9-15-12	12-15-12	15-15-12
15-9-15	12-9-15	9-9-15	6-9-15	3-9-15	0-9-15	15-9-9	12-9-9	9-9-9	6-9-9	3-9-9	0-9-9	15-9-3	12-9-3	9-9-3	6-9-3	3-9-3	0-9-3
0-0-3	3-0-3	6-0-3	9-0-3	12-0-3	15-0-3	0-0-9	3-0-9	6-0-9	9-0-9	12-0-9	15-0-9	0-0-15	3-0-15	6-0-15	9-0-15	12-0-15	15-0-15
15-15-12	12-15-12	9-15-12	6-15-12	3-15-12	0-15-12	15-15-6	12-15-6	9-15-6	6-15-6	3-15-6	0-15-6	15-15-3	12-15-3	9-15-3	6-15-3	3-15-3	0-15-3
0-3-3	3-3-3	6-3-3	9-3-3	12-3-3	15-3-3	0-3-9	3-3-9	6-3-9	9-3-9	12-3-9	15-3-9	0-3-15	3-3-15	6-3-15	9-3-15	12-3-15	15-3-15
15-12-12	12-12-12	9-12-12	6-12-12	3-12-12	0-12-12	15-12-6	12-12-6	9-12-6	6-12-6	3-12-6	0-12-6	15-12-3	12-12-3	9-12-3	6-12-3	3-12-3	0-12-3
0-6-3	3-6-3	6-6-3	9-6-3	12-6-3	15-6-3	0-6-9	3-6-9	6-6-9	9-6-9	12-6-9	15-6-9	0-6-15	3-6-15	6-6-15	9-6-15	12-6-15	15-6-15
15-9-12	12-9-12	9-9-12	6-9-12	3-9-12	0-9-12	15-9-6	12-9-6	9-9-6	6-9-6	3-9-6	0-9-6	15-9-3	12-9-3	9-9-3	6-9-3	3-9-3	0-9-3
0-9-3	3-9-3	6-9-3	9-9-3	12-9-3	15-9-3	0-9-9	3-9-9	6-9-9	9-9-9	12-9-9	15-9-9	0-9-15	3-9-15	6-9-15	9-9-15	12-9-15	15-9-15
15-6-12	12-6-12	9-6-12	6-6-12	3-6-12	0-6-12	15-6-6	12-6-6	9-6-6	6-6-6	3-6-6	0-6-6	15-6-3	12-6-3	9-6-3	6-6-3	3-6-3	0-6-3
0-12-3	3-12-3	6-12-3	9-12-3	12-12-3	15-12-3	0-12-9	3-12-9	6-12-9	9-12-9	12-12-9	15-12-9	0-12-15	3-12-15	6-12-15	9-12-15	12-12-15	15-12-15
15-3-12	12-3-12	9-3-12	6-3-12	3-3-12	0-3-12	15-3-6	12-3-6	9-3-6	6-3-6	3-3-6	0-3-6	15-3-3	12-3-3	9-3-3	6-3-3	3-3-3	0-3-3
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15-9-12	12-9-12	9-9-12	6-9-12	3-9-12	0-9-12	15-9-6	12-9-6	9-9-6	6-9-6	3-9-6	0-9-6	15-9-3	12-9-3	9-9-3	6-9-3	3-9-3	0-9-3

Example:



9 of blue, 3 of green and 12 of red is left  
6 of blue, 12 of green and 3 of red is absorbed



## Lab 17b: Colorimetric Analysis

### Objectives

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Students learn how the factors of concentration and path length affect the absorbance of a colored solution.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Diluting a copper (II) sulfate solution of known concentration in order to create five calibration standards
- ◆ Creating a calibration curve of the absorbance of 660 nanometers of light versus the concentration of the five  $\text{CuSO}_4$  standards
- ◆ Determining the concentration of a sixth copper(II) sulfate solution using the calibration curve and the equation of the slope of the best-fit line of the calibration curve

### Time Requirement

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◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	30 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

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#### *For each student or group:*

- |                          |   |
|--------------------------|---|
| ◆ Data collection system | ◆ Graduated cylinder, 50-mL   |
| ◆ Colorimeter            | ◆ Pipet with pump or bulb, 10-mL                                    |
| ◆ Cuvette                | ◆ Glass stirring rod  |
| ◆ Sensor extension cable | ◆ 0.40 M copper(II) sulfate ( $\text{CuSO}_4$ ), 30 mL <sup>1</sup> |
| ◆ Beakers (2), 100-mL    | ◆ Distilled water, 30 mL  |
| ◆ Test tubes (6), large  | ◆ Marking pen   |
| ◆ Test tube rack         | ◆ Wash bottle with distilled water                                  |

<sup>1</sup>For the preparation of 0.40 M copper (II) sulfate solution, refer to the Lab Preparation section.

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Moles
- ◆ Using linear graphs and equations
- ◆ Molarity
- ◆ Ionic nomenclature

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 10: Determine the Equilibrium Constant for a Chemical Reaction
- ◆ Lab 17a: Absorption Spectra
- ◆ Lab 25: Order of Reaction

## Using Your Data Collection System

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting sensors to the data collection system ◆<sup>(2.1)</sup>
- ◆ Calibrating the colorimeter ◆<sup>(3.2)</sup>
- ◆ Using the colorimeter to collect data with red light ◆<sup>(4.1)</sup>
- ◆ Putting the data collection system into manual sampling mode with manually entered data. ◆<sup>(5.2.1)</sup>
- ◆ Starting a manually sampled new data set ◆<sup>(6.3.1)</sup>
- ◆ Recording a manually sampled data point ◆<sup>(6.3.2)</sup>
- ◆ Stopping a manually sampled data set ◆<sup>(6.3.3)</sup>
- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>
- ◆ Adjusting the scale of the graph ◆<sup>(7.1.2)</sup>

- ◆ Finding the slope and intercept of a best-fit line ◆<sup>(9.6)</sup>
- ◆ Creating calculated data ◆<sup>(10.3)</sup>
- ◆ Saving your experiment ◆<sup>(11.1)</sup>
- ◆ Printing the graph ◆<sup>(11.2)</sup>

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## Background

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To characterize the ability of solutions to absorb light, we use two physical quantities: transmittance  $T$  and absorbance  $A$ . Transmittance is defined as

$$T \equiv \frac{I}{I_0}$$

where

$I$  = the number of transmitted photons (the intensity) per unit time with the absorbing species present

$I_0$  = the number of transmitted photons per unit time with the absorbing species absent

Transmittance multiplied by 100 gives the percentage of the photons that can pass through a solution. It is rather difficult to work with the number of photons, so “absorbance” is used instead. Absorbance is defined as follows:

$$A \equiv -\log T = -\log \frac{I}{I_0}$$

where

$I$  = the number of transmitted photons (the intensity) per unit time with the absorbing species present

$I_0$  = the number of transmitted photons per unit time with the absorbing species absent

It is important to keep in mind that absorption depends on wavelength, and therefore is color specific. That is, the ability of a substance to absorb light is different for photons with different wavelengths.

## Lab 17b: Colorimetric Analysis

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The wavelength dependence of absorbance is expressed by Beer's Law:

$$A = \epsilon l c \quad (1)$$

where

$A$  = absorbance

$\epsilon$  = absorptivity coefficient ( $M^{-1}cm^{-1}$ )

$l$  = path length that light travels through the solution (cm)

$c$  = molar concentration of the absorbing species (M).

**Note:** The AP exam version of Equation 1 is written as “ $A = a b c$ ” where  $a$  and  $b$  correspond to  $\epsilon$  and  $l$ , respectively.

In this activity, you will prepare five solutions of  $CuSO_4$  of different concentrations. Since  $CuSO_4$  is blue, it absorbs light in the visible wavelength region. You will measure the absorbance of the five solutions and construct a calibration curve. Based on the calibration curve and the absorbance obtained of a  $CuSO_4$  solution with unknown concentration, you will determine the concentration of that solution.

### Pre-Lab Activity

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#### Setting the stage for the activity

When light interacts with light-absorbing particles, some of the light is removed. Consequently, objects with many light-absorbing particles appear darker than objects with fewer light-absorbing particles. The ability to absorb color, as well as what color is to be absorbed, depends on the type of particle present.

One can vary the number of particles with which light interacts in two ways. If you compare two solutions of the same colored substance with different concentrations, you see that the one with the higher concentration appears darker because it has more absorbing particles and it absorbs more of the incident light. Also, if you pour the same solution into a test tube and a 100-mL beaker, the solution in the beaker appears darker. Even though the concentration in the two solutions is the same, the light has to travel a longer path in the beaker. Therefore, photons have a higher probability of being absorbed so less light will leave the beaker and the solution appears darker.

Next, if you pour a little bit of the 1 M copper(II) sulfate solution into a 1-L beaker nearly full with water, why is the original beaker so much darker? It is darker because the “light catching” particles in the more diluted solution are much farther apart. From this we see that concentration is another variable to consider when looking at absorbed light.

To measure the amount of light absorbed, we use a colorimeter. Like all electronic measuring devices, the colorimeter produces a voltage based on the amount of light that hits it. The voltage is converted to an absorbance level in optical density units (o.d.).

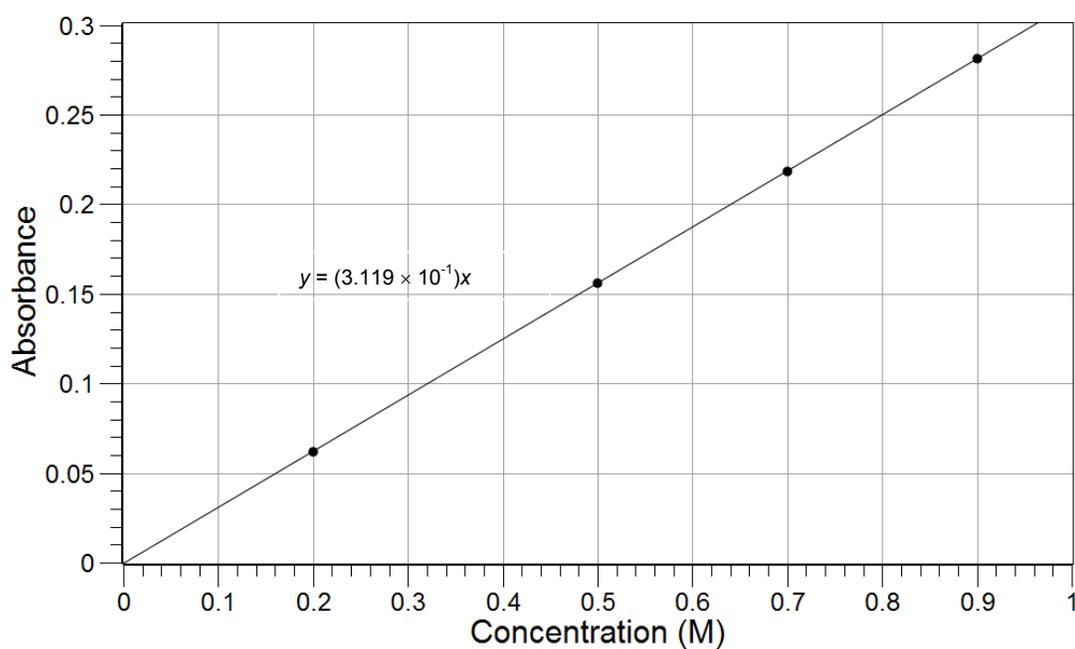
#### Example calculation to try

In an experiment, four calibrating solutions are used to determine the concentration of a  $CuSO_4$  solution with unknown concentration. Given the following data, create a calibration graph, find the equation, and use the equation to solve for the concentration of the unknown solution.

Table 1: Concentration and absorbance calibration data

Sample	Concentration (M)	Absorbance
1	0.20	0.062
2	0.50	0.156
3	0.70	0.218
4	0.90	0.281

The graph of these calibrating solutions:



The absorbance  $A$  of the solution of unknown concentration is 0.200.

The concentration  $c$  of the unknown can be found on the graph, which looks to be between 0.63 M and 0.64 M or calculated from the obtained equation:

The equation of the line is

$$y = 0.3119x$$

Solving for the concentration:

$$A = (0.3119\text{M}^{-1})c$$

$$0.200 = (0.3119\text{M}^{-1})c$$

$$c = \frac{0.200}{0.3119\text{M}^{-1}} = 0.641\text{M}$$

## Lab 17b: Colorimetric Analysis

---

It is worth mentioning that the equation provides a more accurate estimate of the unknown concentration, since the graph-based estimate can be subjective.

### 1. The $\text{CuSO}_4$ solution is blue. Which photons are absorbed and which ones are transmitted through the solution?

The blue photons are transmitted (they reach our eyes, which is why we can see them) and the red and green photons are absorbed.

### 2. Explain why the transmitted light intensity depends on the path length of the solution.

As light is transmitted through the solution, photons are absorbed. The longer the path is, the more light-absorbing particles the photons encounter, and the greater the probability that a particular photon is absorbed. In general, fewer photons exit a solution in a wider container than those exiting a solution in a narrow container, as indicated by their respective light intensities.

## Lab Preparation

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These are the materials and equipment to set up prior to the lab:

1. **0.40 M  $\text{CuSO}_4$ :** Dissolve 49.93 g of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  in some water in a 500-mL volumetric flask. Fill the flask to the mark and mix the solution well.
2. An unknown can be produced by taking a sample of the stock solution and diluting it.

## Safety

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Follow all standard laboratory procedures.

## Sequencing Challenge

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The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

2	1	5	3	4
Then create sample solutions of known concentration.	Set up the data collection system. Establish the measurement for zero absorbance by calibrating the colorimeter with a blank.	Use the graph and the equation to determine the concentration of the unknown.	Measure the absorbance of the sample solutions and the solution of unknown concentration.	Graph absorbance versus concentration and write an equation for the slope of the calibration graph.

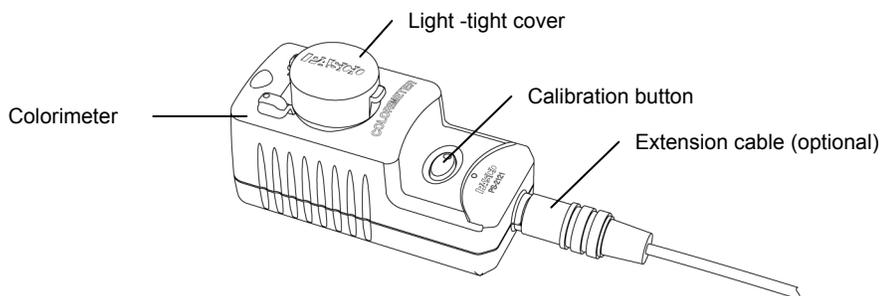
## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box (☐) next to that step.

**Note:** When students see the symbol "◆" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

1. ☐ Start a new experiment on the data collection system. ◆<sup>(1,2)</sup>
2. ☐ Connect the colorimeter to the data collection system using the extension cable. ◆<sup>(2,1)</sup>
3. ☐ Put the data collection system into manual sampling mode with manually entered data. Name the manually entered data "molarity" and give it a unit of "M". ◆<sup>(5,2,1)</sup>
4. ☐ Throughout this activity, collect data using red light (610 nm) in the colorimeter. ◆<sup>(4,1)</sup>
5. ☐ Calibrate the colorimeter with the blank solution (use distilled water for the blank). ◆<sup>(3,2)</sup>



### Collect Data

6. ☐ Water absorbs a small amount of light. Explain why not calibrating with distilled water causes a systematic error in your data.

Since water absorbs light, each absorbance value for the  $\text{CuSO}_4$  solutions would be a little greater, but they would all be greater by about the same amount because of the additional absorbance due to the water. The calibration graph would be shifted upwards.

7. ☐ Measure 30 mL of 0.40 M copper(II) sulfate stock solution into a 100-mL beaker.
8. ☐ Measure 30 mL of distilled water into another 100-mL beaker.
9. ☐ Label four clean, dry test tubes "1" through "4" and place them into a test tube rack. Label a fifth test tube "Stock" and the sixth test tube "Unknown".

## Lab 17b: Colorimetric Analysis

10. □ Why do the test tubes need to be dry? What error would be caused by wet test tubes?

Wet test tubes would change the molarity of the standards, making our calibration graph incorrect.

11. □ Pipet 2.0, 4.0, 6.0, and 8.0 mL of the 0.40 M copper(II) sulfate solution into test tubes 1 through 4, respectively.



12. □ Wash the pipet and use it to deliver 8.0, 6.0, 4.0, and 2.0 mL of distilled water into test tubes 1 through 4 so that each test tube has 10.0 mL of solution.

13. □ Thoroughly mix each solution with a stirring rod.

**Note:** Clean and dry the stirring rod before stirring a different solution.

14. □ Pour the remaining 0.40 M copper(II) sulfate solution into the "Stock" test tube to use as the fifth data point for your calibration graph.

15. □ Put 10 mL of a solution of unknown concentration, obtained from your instructor, in your test tube labeled "Unknown".

Table 2: Volumes and concentrations for the calibration solutions

Trial #	0.40 M CuSO <sub>4</sub> (mL)	H <sub>2</sub> O (mL)	Concentration (M)
1	2.0	8.0	0.08
2	4.0	6.0	0.16
3	6.0	4.0	0.24
4	8.0	2.0	0.32
5	~10	0	0.40
6	10	0	Unknown

**Collect Data**

- 16.**  Start a new, manually sampled data set.  $\diamond^{(6.3.1)}$
- 17.**  Measure the absorbance of the six solutions following the steps below.
- Rinse the cuvette twice with a small portion of the first solution and then fill the cuvette two-thirds full. Wipe the cuvette clean and dry and place it into the colorimeter.
  - Why do you have to rinse the cell with some of the solution?

If there is any residual water in the cuvette, it will dilute the concentration of the solution and falsify the data.

- After the reading stabilizes, record a data point.  $\diamond^{(6.3.2)}$
- Dispose of the solution appropriately and rinse the cell thoroughly with water.
- Why do you think it is important to rinse the cell thoroughly between measurements?

You need to rinse the cell to avoid contamination of the solutions.

- When you have recorded all of your data, stop the data set.  $\diamond^{(6.3.3)}$

- 18.**  Save your experiment  $\diamond^{(11.1)}$  and clean up according to your teacher's instructions.

**Data Analysis**

- 1.**  Record your data in the table below.

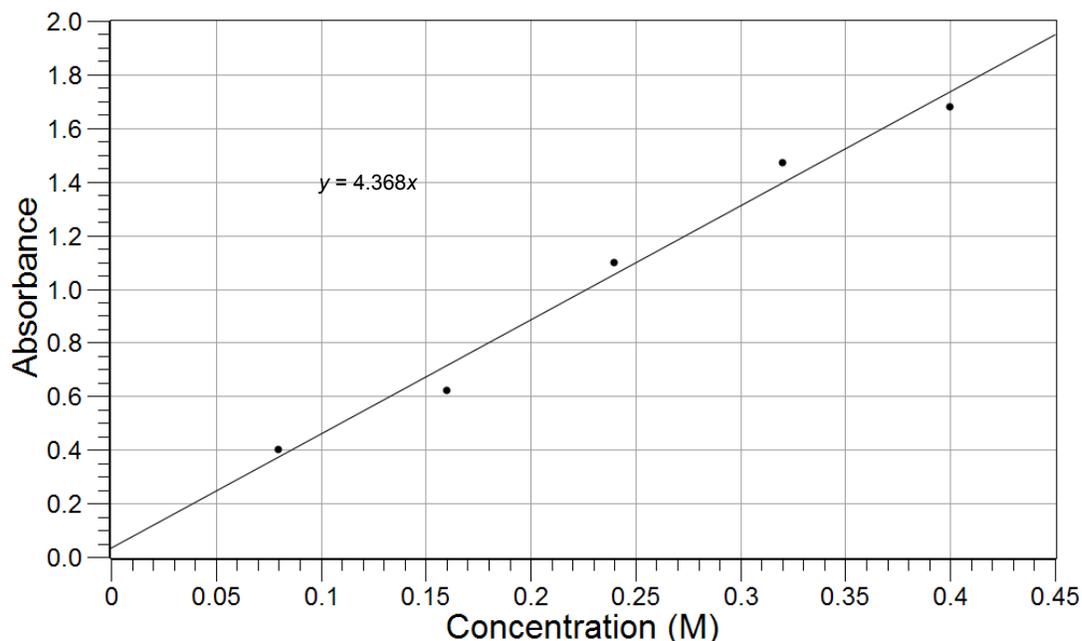
Table 3: Concentration and absorbance data

Test Tube	Concentration (M)	Absorbance
1	0.08	0.402
2	0.16	0.620
3	0.24	1.099
4	0.32	1.470
5	0.40	1.680
6	Unknown	0.956

- 2.**  Display Concentration on the x-axis and Absorbance on the y-axis.  $\diamond^{(7.1.1)}$
- 3.**  Adjust the scale of the graph to show all data.  $\diamond^{(7.1.2)}$

## Lab 17b: Colorimetric Analysis

- Find the slope of the best-fit line. ♦<sup>(9.6)</sup>
- Print the graph. ♦<sup>(11.2)</sup>
- Sketch or attach your graph of Concentration versus Absorbance.



- Using your understanding of dependent and independent variables, explain why concentration should be on the horizontal (x) axis for this graph.

The concentration is the controlled variable and the absorbance was a response to the concentration. That makes absorbance the dependent variable and concentration the independent variable, which should be on the x-axis.

- Use the equation for the line to calculate the concentration of the unknown. Show all of your work.

The equation of the line is:

$$y = 4.368x$$

$$x = \frac{y}{4.368}$$

Solving for the concentration:

$$c = \frac{A}{\epsilon l} = \frac{0.956}{4.368 \text{ M}^{-1}} = 0.219 \text{ M}$$

- Use the calibration curve to calculate the concentration of the unknown.

The concentration that corresponds to  $A = 0.956$  is about 0.213 M.

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## Analysis Questions

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**1. Does using the graph or using the equation give a more accurate estimate of the concentration of the unknown?**

The equation relies on the best-fit line of all five points so is more reliable than estimating the value from the graph.

**2. Beer's law states that absorbance is proportional to concentration. How does your data support this statement?**

For two variables to be proportional, a graph must be linear and may go through the origin. The graph from the data was linear and did go through the origin.

**3. Why would one or more dirty cuvettes cause error in the data?**

The absorbance readings would be incorrect for individual samples because some light would be absorbed by the dirt.

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## Synthesis Questions

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Use available resources to help you answer the following questions.

**1. In a copper(II) sulfate solution, copper(II) ions cause the blue color. Sulfate ions have no color. Sodium sulfate solution has no color because neither ion has color. Could you use Beer's law to find the concentration of a sodium sulfate solution?**

No. The absorbance would not change with the concentration of sodium sulfate.

**2. Hypothetically, how would you modify the experiment if the measured absorbance of the unknown was too low to measure?**

Increasing the path length would increase the absorbance. Therefore, using a cell with a longer path length would work.

**3. How would you modify your experiment if the measured absorbance of the unknown was too high to measure?**

Making a proportional dilution of the solution would decrease the absorbance.

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## Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

**1. Which of the following variables affects the absorbance of light in a solution?**

- A.** The distance the light must travel through the solution (path length)
- B.** The amount of solute in each volume (concentration)
- C.** The wavelength of the light that is interacting with the solution
- D.** All of the above

### *Lab 17b: Colorimetric Analysis*

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**2.** A sample of 0.10 M copper(II) chloride is placed into a cuvette with a 1.00-cm path length. The solution has a measured absorbance of 2.000. What would you expect the absorbance of a 0.05 M copper(II) chloride solution to be?

- A.** 2.000
- B.** 1.000
- C.** 4.000
- D.** Not enough information.

### **Extended Inquiry Suggestions**

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Many water quality studies are done using colorimetric techniques. PASCO offers water quality test kits for use with the water quality colorimeter. The kits allow rapid testing of water for specific ions as the calibration curves are stored in the device. Have students analyze a nearby stream, taking advantage of the learning from this lab.

## Lab 18: Separation by Liquid Chromatography

### Objectives

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Students use liquid chromatography to separate the ingredients of a mixture.

### Procedural Overview

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Students will gain experience conducting the following procedures:

- ◆ Separation processes
- ◆ Practicing basic principles of liquid chromatography
- ◆ Calculating the effectiveness of the column and its ability to separate the compounds of the mixture

### Time Requirement

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◆ Preparation time	20 minutes
◆ Pre-lab discussion and activity	20 minutes
◆ Lab activity	120 minutes

### Materials and Equipment

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***For each student or group:***

- ◆ C18 Sep-Pak<sup>®</sup> cartridge
- ◆ Syringe, 1-mL
- ◆ Syringe, 10-mL, or dropper bottle or wash bottle
- ◆ Graduated cylinder, 10-mL
- ◆ 18% Isopropanol, 100 mL<sup>1</sup>
- ◆ Unsweetened Kool-Aid<sup>®</sup> drink, 10 mL<sup>2</sup>
- ◆ Distilled water

<sup>1-2</sup>To prepare the solutions, refer to the Lab Preparation section.

## **Concepts Students Should Already Know**

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Students should be familiar with the following concepts:

- ◆ Interaction between molecules
- ◆ Polarity of molecules
- ◆ Basic separation principles

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 28: Molecular Interaction in Ethanol and Acetone

## **Using Your Data Collection System**

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

As this lab activity does not use a data collection system, no Tech Tips (indicated by the symbol "◆" and a superscripted number following a step) are needed.

## **Background**

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Chromatography is a separation method that exploits the differences in the behavior of substances between a mobile phase (solvent or eluent) and a stationary phase (substrate), to separate the components of a mixture. The stationary phase may interact with the substances in the mixture based on charge, relative solubility, or adsorption. The mobile phase carries each substance along at a rate that depends on the attraction of the substance to the stationary phase. The components of the mixture will separate if their interactions with the substrate and solvent are significantly different.

## **Pre-Lab Activity**

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### ***Setting the stage for the activity***

In this activity, you will separate the dyes: FD&C Blue and FD&C Red, from the other ingredients in grape-flavored Kool-Aid<sup>®</sup>. You will use a C18 Sep-Pack<sup>®</sup> chromatography column. The column contains a silica matrix with a non-polar substrate consisting of a C18 hydrocarbon bonded to the silica. The C18-coated surface can bind non-polar molecules, particularly organic molecules with a carbon chain with London-type dispersion forces. The more polar a molecule is, the less it is bound to the C18-coated matrix.

If a mixture of molecules with different polarity is put through the column, the ones with greatest polarity will bind the least to the C18-coated surface. Therefore, they will move to the end of the column first. The molecules with the least polarity will be trapped at the beginning of

the column, on the C18-coated surface. Therefore, they will be "retained" and show up last at the end of the column.

The separation of the molecules can be improved by using a polar solvent (or solvent mixture, the eluent) to promote the removal (elution) of the more polar molecules first and less polar molecules last. In a more sophisticated setting, the polarity of the eluting solvent can be changed continuously during the course of the experiment.

### Example calculation to try

A sample of unsweetened, grape-flavored Kool-Aid was analyzed for two major coloring ingredients. The Sep-Pak column used for the analysis was flushed with 10 mL of 26% isopropanol solution and then 10 ml of water. A 1.00 mL sample of Kool-Aid was loaded into the column and eluted with 26% isopropanol from a 10-mL syringe at the rate of 10 mL/min into a 10-mL graduated cylinder. The red coloring agent started to elute after 0.8 mL of the mobile phase was completely eluted after 1.7 mL of eluent was collected in the graduated cylinder.

At this point the blue coloring agent started to come out. The blue component was completely eluted after a total of 4.1 mL of eluent was collected. The data obtained was collected in the table below. The band width,  $W_{\text{red}}$ , can be calculated for the red compound as follows:

$$W_{\text{red}} = V_{R(\text{end,red})} - V_{R(\text{start,red})} = 1.7 \text{ mL} - 0.8 \text{ mL} = 0.9 \text{ mL}$$

where

$V_{R(\text{end, red})}$  = volume of eluent collected by the time all red dye left the column

$V_{R(\text{start, red})}$  = volume of eluent collected by the time the red dye starts to show up in the eluent

Note: The "R" subscript means "retention," common terminology in chromatography.

For the blue compound the same calculation applies:

$$W_{\text{blue}} = V_{R(\text{end,blue})} - V_{R(\text{start,blue})} = 4.1 \text{ mL} - 1.7 \text{ mL} = 2.4 \text{ mL}$$

The center of both bands can be obtained:

$$V_{R(\text{avg,red})} = V_{R(\text{start,red})} + 0.5 W_{\text{red}} = 0.8 \text{ mL} + (0.5)(0.9 \text{ mL}) = 1.2 \text{ mL}$$

$$V_{R(\text{avg,blue})} = V_{R(\text{start,blue})} + 0.5 W_{\text{blue}} = 1.7 \text{ mL} + (0.5)(2.4 \text{ mL}) = 2.9 \text{ mL}$$

where

$V_{R(\text{avg,red})}$  = volume of eluent collected by the time half of the red dye left the column

$V_{R(\text{avg,blue})}$  = volume of eluent collected by the time half of the blue dye left the column

The length  $L$  of the column was 1.25 cm and the radius  $r$  of the column was 0.50 cm. The mobile phase volume,  $V_M$ , was

$$V_M = 0.5 \pi r^2 L = (0.5)(3.14)(0.50 \text{ cm})^2(1.25 \text{ cm}) = 0.49 \text{ cm}^3 = 0.49 \text{ mL}$$

## Lab 18: Separation by Liquid Chromatography

---

The retention factors,  $k_{\text{blue}}'$  and  $k_{\text{red}}'$ , which are the measure of how much the stationary phase can "retain" the two components, were:

$$k_{\text{red}}' = \frac{V_{R(\text{avg,red})} - V_M}{V_M} = \frac{(1.2 \text{ mL}) - (0.49 \text{ mL})}{(0.49 \text{ mL})} = 1.4$$
$$k_{\text{blue}}' = \frac{V_{R(\text{avg,blue})} - V_M}{V_M} = \frac{(2.9 \text{ mL}) - (0.49 \text{ mL})}{(0.49 \text{ mL})} = 4.9$$

The separation,  $\alpha$ , can be obtained:

$$\alpha = \frac{k_{\text{blue}}'}{k_{\text{red}}'} = \frac{4.9}{1.4} = 3.5$$

The theoretical plate number,  $N$ , which characterizes the number of interactions between the stationary phase and mobile phase (calculated from the last eluted component, is:

$$N = 16 \left( \frac{V_{R(\text{end,blue})}}{W_{\text{blue}}} \right)^2 = 16 \left( \frac{4.1 \text{ mL}}{2.4 \text{ mL}} \right)^2 = 46.7$$

The most important factor, the resolution ( $R$ ) for the two ingredients is:

$$R = \frac{2(V_{R(\text{avg,blue})} - V_{R(\text{avg,red})})}{W_{\text{red}} + W_{\text{blue}}} = \frac{(2)(2.9 \text{ mL} - 1.2 \text{ mL})}{2.4 \text{ mL} + 0.9 \text{ mL}} = 1.0$$

The numerator is the volume between the center of the bands made by the two dyes in the column, which is related to the selectivity factor  $\alpha$ . The denominator is the sum of the band widths.  $R$  is proportional to the efficiency of the column. As the value of  $R$  increases above 1 there is greater separation of the dyes.

Table 1: Effectiveness of the separation of the red and blue dyes in Kool-Aid

Parameters	Red Dye	Blue Dye
$V_{R(\text{start})}$ (mL)	0.8	1.7
$V_{R(\text{end})}$ (mL)	1.7	4.1
$W$ (mL)	0.9	2.4
$V_{R(\text{avg})}$ (mL)	1.25	2.9
$L$ (cm)	1.25	1.25
$r$ (cm)	0.50	0.50
$V_M$ (mL)	0.49	0.49
$k'$	1.4	4.9
$\alpha$	3.5	
$N$	46.7	
$R$	1.0	

**1. The greater the resolution  $R$ , the better the column separates the two different components. What can you tell about the column for this particular separation process based on the obtained resolution factor?**

The conditions are not ideal as  $R = 1.0$ , indicating a small degree of separation.

**2. Based on the process described above, can you propose a modification that might improve the resolution?**

Changing the polarity of the eluent or increasing the column length (that is, using two columns connected) might improve the separation process.

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

- 18% Isopropanol:** Combine 260 mL of 70% isopropanol with some water in a 1-L volumetric flask and fill it to the mark.
- Unsweetened Grape Kool-Aid:** Prepare as instructed on the package without the sugar.

## Safety

Follow all standard laboratory procedures.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

2	3	1	5	4
Use the syringe to load the column with the Kool-Aid sample.	Elute the column with 18% isopropanol at a steady rate of 5 to 10 mL per minute.	Rinse the column first with 18% isopropanol and then with water.	Calculate the retention parameters.	Record the volumes at which both dyes start to appear in the eluent and where they stop appearing.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** This activity does not use a data collection system, so there are no Tech Tips (indicated by the symbol "♦" and a superscripted number following a step).

### Set Up

- Cut off the exit tube of the C18 Sep-Pack chromatography cartridge (see Figure 1).

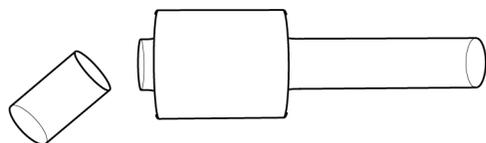


Figure 1: Cutting the Sep Pak to size

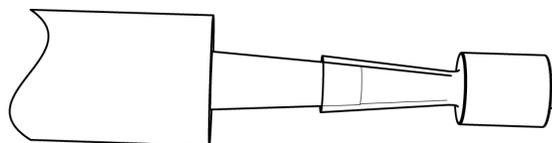


Figure 2: Mounting the Sep Pak on a syringe

### Collect Data

Perform the following steps three times and record your data for each trial in Table 2.

- Use a 10-mL syringe (see Figure 2), a dropper bottle, or a wash bottle as a solvent pump to flush the C18 Sep-Pack cartridge with 10 mL of undiluted 18% isopropanol at a rate of 5 to 10 mL per minute.
- Collect the eluate in a 10-mL graduated cylinder to monitor the flow rate of the isopropanol.

4.  Using the 10-mL syringe, flush the Sep-Pack cartridge with 10 mL of distilled water.
5.  Draw up 1 mL of the grape Kool-Aid sample with the 1-mL syringe.
6.  Slowly inject the Kool-Aid sample into the Sep-Pack cartridge.
7.  Collect and discard the effluent that washes out of the column as you inject the sample.
8.  Fill the solvent pump (10-mL syringe, dropper, or wash bottle) with 18% isopropanol solution.
9.  Which is the most and which is the least polar solvent of the following: pure isopropanol, 18% isopropanol solution, and water?

Water is the most polar and pure isopropanol is the least polar.

10.  Pump the solvent through the Sep-Pack cartridge at a steady rate of 5 to 10 mL per minute.
11.  Collect the eluent in the 10-mL graduated cylinder.
12.  What do you think will happen if you pump the eluent at too high a rate?

The separation of the two components will be reduced.

13.  Record (in Table 2) the volume of liquid in the graduated cylinder when the red or blue dye is first observed in the eluent from the Sep-Pak column ( $V_{R(\text{start})}$ ) and when that color is no longer observed ( $V_{R(\text{end})}$ ).

**Note:** If the red and blue bands are not completely separated, the overlapping region will be purple. Use the center of the purple band as the end of the first band and the beginning of the second band.

14.  Clean up according to your teacher's instructions.

## Data Analysis

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1.  For each trial, calculate  $V_M$ , the mobile phase volume. This factor represents about half of the empty column volume. The unit for  $V_M$  is  $\text{cm}^3$  (mL) if the values of  $r$  and  $L$  are used in the calculations in centimeters. Record these values in Table 2.

$$V_M = 0.5 \pi r^2 L = 0.5(3.14)(0.5 \text{ cm})^2(1.25 \text{ cm}) = 0.49 \text{ cm}^3 = 0.49 \text{ mL}$$

## Lab 18: Separation by Liquid Chromatography

2.  For each trial, calculate the band width for the two components. Record these values in Table 2.

Performing the calculations for Trial #1:

$$W_{\text{red}} = V_{R(\text{end,red})} - V_{R(\text{start,red})} = 2.00 \text{ mL} - 1.50 \text{ mL} = 0.50 \text{ mL}$$

$$W_{\text{blue}} = V_{R(\text{end,blue})} - V_{R(\text{start,blue})} = 3.40 \text{ mL} - 2.10 \text{ mL} = 1.30 \text{ mL}$$

3.  For each trial, calculate the center of the bands for the two components. Record these values in Table 2.

Performing the calculations for Trial #1:

$$V_{R(\text{avg,red})} = V_{R(\text{start,red})} + 0.5 W_{\text{red}} = 1.50 \text{ mL} + (0.5)(0.50 \text{ mL}) = 1.75 \text{ mL}$$

$$V_{R(\text{avg,blue})} = V_{R(\text{start,blue})} + 0.5 W_{\text{blue}} = 2.1 \text{ mL} + (0.5)(1.30 \text{ mL}) = 2.75 \text{ mL}$$

4.  For each trial, calculate the retention factor  $k'$  for both components, which is the measure of how much the stationary phase can "retain" the two components. Record these values in Table 2. (Optimum values of  $k'$  are commonly between 1 and 10, where 10 indicates the greatest retention.)

Performing the calculation for Trial #1:

$$k'_{\text{red}} = \frac{V_{R(\text{avg,red})} - V_M}{V_M} = \frac{(1.75 \text{ mL}) - (0.49 \text{ mL})}{(0.49 \text{ mL})} = 2.57$$

$$k'_{\text{blue}} = \frac{V_{R(\text{avg,blue})} - V_M}{V_M} = \frac{(2.75 \text{ mL}) - (0.49 \text{ mL})}{(0.49 \text{ mL})} = 4.61$$

5.  For each trial, calculate the selectivity, or separation factor,  $\alpha$ . Record these values in Table 2. (The value of  $\alpha$  is always larger than 1, and the larger it is, the greater the separation of the eluates.)

Performing the calculation for Trial #1:

$$\alpha = \frac{4.61}{2.57} = 1.79$$

6.  For each trial, calculate the number of theoretical plates,  $N$ , in the column. Think of  $N$  as the number of times a dye molecule is exchanged between the stationary phase and the mobile phase. The value of  $N$  is generally based on the dye which is eluted last. A large value of  $N$  means that the column is more efficient. (The range of  $N$  is normally between 20 and 200).

Record these values in Table 2.

Performing the calculations for Trial #1:

$$N = 16 \left( \frac{V_{R(\text{end,blue})}}{W_{\text{blue}}} \right)^2 = 16 \left( \frac{3.40}{1.30} \right)^2 = 109$$

7.  For each trial, calculate the resolution  $R$ , the major objective of a chromatographic separation.  $R$  measures how well the two dyes were separated by the Sep-Pack cartridge. Record these values in Table 2.

Performing the calculations for Trial #1:

$$R = \frac{2(V_{R(\text{avg,blue})} - V_{R(\text{avg,red})})}{W_{\text{red}} + W_{\text{blue}}} = \frac{2(2.75 \text{ mL} - 1.75 \text{ mL})}{0.50 \text{ mL} + 1.30 \text{ mL}} = 1.11$$

8.  Calculate the average values for  $\alpha$ ,  $N$ , and  $R$ . Record these values in Table 2.

$$\alpha: (1.79 + 1.76 + 1.75)/3 = 1.64$$

$$N: (109.4 + 90.9 + 111.2)/3 = 103.8$$

$$R: (1.11 + 1.00 + 1.00)/3 = 1.04$$

Table 2: Effectiveness of the separation of the red and blue dyes in Kool-Aid

Parameters	Trial #1		Trial #2		Trial #3		Average	
	Red Dye	Blue Dye	Red Dye	Blue Dye	Red Dye	Blue Dye		
$V_{R(\text{start})}$ (mL)	1.50	2.10	1.40	1.80	1.30	1.80		
$V_{R(\text{end})}$ (mL)	2.00	3.40	1.80	3.10	1.80	2.90		
$W$ (mL)	0.50	1.30	0.40	1.30	0.50	1.10		
$V_{R(\text{avg})}$ (mL)	1.75	2.75	1.60	2.45	1.55	2.35		
$L$ (cm)	1.25							
$r$ (cm)	0.50							
$V_M$ (mL)	0.49							
$k'$	2.57	4.61	2.27	4.00	2.16	3.80		
$\alpha$	1.79		1.76		1.75			1.76
$N$	109		91		111			103.8
$R$	1.11		1.00		1.00			1.04

## Analysis Questions

1. What does your value of  $R$  indicate?

The resolution is close to 1.0 which means the two dyes will come immediately after each other; a larger number would indicate better separation.

2. Propose an approach to improve the resolution.

Increasing the polarity of the solvents or changing the stationary phase improves resolution.

## Lab 18: Separation by Liquid Chromatography

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**3. What conclusion can you draw about the polarity of the dye molecules relative to each other, based on the fact that the red dye came off first and the blue second with the 18% isopropanol solution?**

The red dye is more polar than the blue as the blue was "retained" more on the non-polar C18 surface.

**4. Would the solution process change if you used water (a more polar solvent than the 18% isopropanol solution) and if so, how?**

Yes, it would change. The more polar molecules of the red dye probably would come off sooner and the less polar molecules of the blue dye would be retained longer on the surface of the stationary phase.

**5. Would the solution process change if we used benzene (a non-polar solvent) and if so, how?**

The separation of the two types of molecules would be poorer since both the stationary and mobile phases would be non-polar.

## Synthesis Questions

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Use available resources to help you answer the following questions.

**1. What physical parameter of the column would you change to optimize separation?**

Increasing the length should improve separation.

**2. How would the elution process have been different if you used a column with a polar matrix?**

A polar matrix would have retained the blue dye longer as it is more polar. Also, a non-polar solvent would have been necessary.

## Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

**1. The resolution is a measure of:**

- A. The polarity of the molecules.
- B. The polarity of the eluent .
- C. The effectiveness of the separation of the two types of molecules.
- D. The polarity of the column.

**2. The parameter of  $k'$  is a measure of:**

- A. The resolution.
- B. The retention for each of the dyes.
- C. The selectivity, which is the ratio of the  $k$  values for the two dyes.
- D. The plate number.

**3. You can tell the red dye is more polar than the blue dye because:**

- A.** It eluted last with 18% isopropanol, which means the red dye bound to the matrix more.
- B.** It eluted last with 18% isopropanol, which means the blue dye bound to the matrix less.
- C. It eluted first with 18% isopropanol, which means the blue dye bound to the matrix more.**
- D.** It eluted first with 18% isopropanol which means the blue dye bound to the matrix less.

**4. Decreasing the polarity of the eluent will:**

- A. Decrease the selectivity since the dye molecules will be equally attracted to the non-polar matrix and the non-polar eluent.**
- B.** Increase the selectivity since the dye molecules will be attracted to the non-polar matrix more than the non-polar eluent.
- C.** Decrease the selectivity since the dye molecules will be less attracted to the non-polar matrix than to the non-polar eluent.
- D.** Increase the selectivity since the dye molecules will more attracted to the non-polar matrix and the non-polar eluent.

**Extended Inquiry Suggestions**

Since the resolution determined for this activity may not be better than one, it would be a great challenge for students to devise a method to improve selectivity. Tackle the possible parameters to be changed with your students. The ideas should include attaching multiple columns together (effectively increasing the column length) and optimizing the solvent composition for separation.

Also, if an Amadeus spectrometer system is available, 0.5 mL aliquots of the eluent can be collected and analyzed using the spectrometer. The absorbance of both components would have to be recorded at the wavelength of maximum absorbance. The plot of absorbance as a function of aliquot number would be a simple representation of a "chromatogram". This technique would demonstrate how High Performance Liquid chromatography (HPLC) works with an absorbance detector.



## Lab 19: Properties of Buffer Solutions

### Objectives

Students learn the properties of buffer solutions and buffer capacity.

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Preparing three buffer solutions using different concentrations of acetic acid
- ◆ Testing the buffering capacity of the three buffers by adding HCl
- ◆ Calculating the predicted and actual buffer capacity of the three buffer solutions
- ◆ Comparing the buffer capacity and buffering effect of the three buffer solutions and water.

### Time Requirement

◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

#### *For each student or group:*

- |                                     |  |
|-------------------------------------|--|
| ◆ Data collection system            | ◆ Clamp, buret   |
| ◆ pH sensor                         | ◆ Clamp, utility                                       |
| ◆ Beaker, 400-mL                    | ◆ Funnel   |
| ◆ Buret, 50-mL                      | ◆ 2.000 M Sodium hydroxide (NaOH), 250 mL <sup>1</sup> |
| ◆ Pipet, 5-mL and rubber bulb       | ◆ 0.1 M Acetic acid (HOAc), 250 mL <sup>2</sup>        |
| ◆ Beaker, 100-mL                    | ◆ 0.3 M HOAc, 250 mL <sup>3</sup>                      |
| ◆ Beaker (2), 25-mL                 | ◆ 0.5 M HOAc, 250 mL <sup>4</sup>                      |
| ◆ Graduated cylinder, 250-mL        | ◆ 6.00 M Hydrochloric acid (HCl), 5 mL <sup>5</sup>    |
| ◆ Magnetic stirrer and stirring bar | ◆ Buffers, pH 4 and pH 10, 10 mL                       |
| ◆ Ring stand                        | ◆ Wash bottle with deionized water                     |

<sup>1-5</sup>To prepare the solutions, refer to the Lab Preparation section.

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Acids and bases
- ◆ Electrolytes
- ◆ Le Chatelier's Principle
- ◆ Molarity

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 24: Determining  $K_a$  by Half-Titration of a Weak Acid
- ◆ Lab 30: Determination of the  $K_a$  Values of Two Isomeric Multi-Protic Acids

## Using Your Data Collection System

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system <sup>(1.2)</sup>
- ◆ Connecting a sensor to the data collection system <sup>(2.1)</sup>
- ◆ Calibrating the pH sensor ◆<sup>(3.6)</sup>
- ◆ Monitoring live data without recording ◆<sup>(6.1)</sup>

## Background

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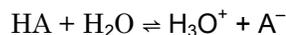
Adding small amounts of acid or base to water dramatically changes the hydrogen ion concentration, which changes the pH. If species are present in the water that can neutralize the added acid or base, then the pH change will be much smaller. Solutions that resist pH change in this fashion are called buffers.

A buffer is made by adding a weak acid and a conjugate base of the same weak acid to water. If acid is then added to the buffer system, the conjugate base “consumes” the added acid. The opposite is true for the addition of a base. In either case, the resulting hydrogen ion concentration changes little.

## Pre-Lab Activity

### Setting the stage for the activity

Weak acids (HA) partially dissociate, as shown below:



The corresponding acidity constant is

$$K_a = \frac{[\text{H}_3\text{O}^+][\text{A}^-]}{[\text{HA}]}$$

If the solution contains the salt of the acid (that is, the conjugate base of the acid, A<sup>-</sup>) the equilibrium shifts to the left in accordance with the Le Chatelier Principle. This also means that the auto-dissociation of the acid is suppressed, so that:

$$[\text{HA}] \approx c_{\text{HA}}$$

$$[\text{A}^-] \approx c_{\text{A}^-}$$

$$K_a = \frac{[\text{H}_3\text{O}^+]c_{\text{A}^-}}{c_{\text{HA}}}$$

where  $c_{\text{HA}}$  and  $c_{\text{A}^-}$  are the concentrations of the weak acid and the salt, respectively. Solving for  $[\text{H}_3\text{O}^+]$  yields

$$[\text{H}_3\text{O}^+] = K_a \frac{c_{\text{HA}}}{c_{\text{A}^-}}$$

$$\log[\text{H}_3\text{O}^+] = \log K_a + \log \frac{c_{\text{HA}}}{c_{\text{A}^-}}$$

$$-\log[\text{H}_3\text{O}^+] = -\log K_a + \log \frac{c_{\text{A}^-}}{c_{\text{HA}}}$$

$$\text{pH} = \text{p}K_a + \log \frac{c_{\text{A}^-}}{c_{\text{HA}}}$$

The final equation is known as the Henderson-Hasselbach equation.

It is worth mentioning that the pH of buffers does not depend on the actual concentration of the acid or salt; it only depends on the ratio of the two.

How well buffers maintain their pH when acids and bases are added is measured by their buffer capacity. Buffer capacity is the amount of acid or base, in mol/L, that changes the pH by one unit. Adding  $x$  mol/L of acid increases the amount of acid by  $x$  amount and removes the same amount of salt. Notice that the larger the value of  $x$  is, the more acid or base it takes to change the pH one unit, hence the larger the buffer capacity. The Henderson-Hasselbach equation can be modified to incorporate the pH change of one unit:

$$\text{pH} - 1 = \text{p}K_a + \log \frac{c_{\text{A}^-} - x}{c_{\text{HA}} + x}$$

## Lab 19: Properties of Buffer Solutions

Solving for  $x$ , the buffer capacity, from this last equation yields:

$$x = \frac{c_{A^-} - c_{HA} 10^{\text{pH} - 1 - \text{pKa}}}{1 + 10^{\text{pH} - 1 - \text{pKa}}}$$

A buffer can be made by adding, for example, some NaOH solution to the solution of a weak acid, HA, producing the solution of the conjugate base,  $A^-$ . You must ensure that the acid is in excess. The necessary concentrations can be calculated using the ICE box. (Keep in mind that concentrations change not only because of the reaction but also because the volume of the solution changes by adding the NaOH solution.)

Table 1: ICE box for calculating concentrations of the weak acid and conjugate base

Conditions	[HA]	[A <sup>-</sup> ]
Initial	$c_{HA}^0 \frac{V_{HA}}{V_{NaOH} + V_{HA}}$	0
Change	$-c_{NaOH}^0 \frac{V_{NaOH}}{V_{NaOH} + V_{HA}}$	$c_{NaOH}^0 \frac{V_{NaOH}}{V_{NaOH} + V_{HA}}$
Equilibrium	$\frac{c_{HA}^0 V_{HA} - c_{NaOH}^0 V_{NaOH}}{V_{NaOH} + V_{HA}}$	$c_{NaOH}^0 \frac{V_{NaOH}}{V_{NaOH} + V_{HA}}$

Therefore from the table the equilibrium concentrations of the conjugate base and the acid are:

$$c_{A^-} = \frac{c_{NaOH}^0 V_{NaOH}}{V_{NaOH} + V_{HA}}$$

$$c_{HA} = \frac{c_{HA}^0 V_{HA} - c_{NaOH}^0 V_{NaOH}}{V_{NaOH} + V_{HA}}$$

where

$c_{A^-}$  = concentration of conjugate base (salt) in the buffer (M)

$c_{NaOH}^0$  = initial concentration of NaOH solution (M)

$V_{NaOH}$  = added volume of NaOH solution (mL)

$c_{HA}$  = concentration of HA in buffer (M)

$c_{HA}^0$  = initial concentration of HA (M)

$V_{HA}$  = volume of acid (mL)

**Example calculation to try**

A sample of 100 mL of 0.5 M acetic acid solution was titrated with 2.000 M NaOH solution to make a buffer with a pH of 5.5. To reach the desired pH, 21.20 mL of NaOH solution had to be added. The concentration of the conjugate base (salt) and acid can be calculated by using the ICE box:

Table 2: Example calculations using the ICE box

Conditions	[HA]	[A <sup>-</sup> ]
Initial	$\frac{(0.500 \text{ M})(100.00 \text{ mL})}{(21.20 \text{ mL} + 100.00 \text{ mL})} = 4.12 \times 10^{-1} \text{ M}$	0
Change	$-\frac{(2.000 \text{ M})(21.20 \text{ mL})}{(21.20 \text{ mL} + 100.00 \text{ mL})} = -0.350 \text{ M}$	$\frac{(2.000 \text{ M})(21.20 \text{ mL})}{(21.20 \text{ mL} + 100.00 \text{ mL})} = 0.350 \text{ M}$
Equilibrium	$0.412 \text{ M} - 0.350 \text{ M} = 6.2 \times 10^{-2} \text{ M}$	$\frac{(2.000 \text{ M})(21.20 \text{ mL})}{(21.20 \text{ mL} + 100.00 \text{ mL})} = 0.350 \text{ M}$

Therefore from the table the equilibrium concentrations of the conjugate base and the acid are:

$$c_{\text{A}^-} = \frac{(2.000 \text{ M})(21.20 \text{ mL})}{(21.20 \text{ mL} + 100.00 \text{ mL})} = 0.350 \text{ M}$$

$$c_{\text{HA}} = \frac{(0.500 \text{ M})(100.00 \text{ mL}) - (2.000 \text{ M})(21.20 \text{ mL})}{(21.20 \text{ mL} + 100.00 \text{ mL})} = 6.2 \times 10^{-2} \text{ M}$$

The buffer capacity of the prepared buffer is predicted to be

$$x = \frac{(0.350 \text{ M}) - (6.2 \times 10^{-2} \text{ M})10^{5.5-1-4.75}}{1 + 10^{5.5-1-4.75}} = 0.20 \text{ M}$$

To test the buffer capacity of the buffer, 6.00 M HCl solution was added by drops until the pH of the buffer reached 4.5. This required 4.00 mL of the HCl solution. The concentration of HCl in the final solution is

$$c_{\text{HCl}} = \frac{(6.00 \text{ M})(4.00 \text{ mL})}{(21.20 \text{ mL} + 100.00 \text{ mL} + 4.00 \text{ mL})} = 0.192 \text{ M}$$

This is close to the predicted value of 0.20 M.

**1. Would adding NaOH solution to acetic acid solution increase or decrease the pH of the solution?**

The pH would increase.

## Lab 19: Properties of Buffer Solutions

---

### 2. When calculating the added acid concentration ( $c_{\text{HCl}}$ ) why didn't we convert the volume to liters to be consistent with M?

The volume units cancel out. Therefore, the answer does not depend on the volume units as long as the same unit is used throughout the calculation.

## Lab Preparation

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These are the materials and equipment to set up prior to the lab:

1. **2.000 M NaOH:** Dissolve 80 g of NaOH in some water in a 1-L volumetric flask, and fill it to the mark.

**Note:** Standardize the solution and report the actual concentration to the students.

2. **0.1 M HOAc:** Combine 11.20 mL of glacial HOAc with some water in a 2-L volumetric flask and fill it to the mark.
3. **0.3 M HOAc:** Combine 33.60 mL of glacial HOAc with some water in a 2-L volumetric flask and fill it to the mark.
4. **0.5 M HOAc:** Combine 56 mL of glacial HOAc with some water in a 2-L volumetric flask and fill it to the mark.
5. **6.00 M HCl:** Combine 50 mL of a 36% HCl solution with some water in a 100-mL volumetric flask and fill it to the mark.

**Note:** Standardize the solution and report the actual concentration to the students.

## Safety

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Add these important safety precautions to your normal laboratory procedures:

- ◆ Wash off any HCl solution that comes in contact with your skin with large amounts of water.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

1	5	2	4	3
Set up the data collection system and pH sensor. Set up the titration apparatus. Fill the buret with the conjugate base (NaOH).	Complete the process for each HOAc solution. Calculate and compare the buffer capacity of the 0.1 M, 0.3, M and 0.5 M HOAc solutions.	Put an accurately measured quantity of the first HOAc solution into a beaker. Place it onto the magnetic stirrer.	Test the buffer capacity: Add 6.0 M HCl drop wise until the pH changes one unit. Record the volume of HCl solution.	Prepare the buffer: Add the NaOH solution drop wise into the acetic acid solution until the pH of the buffer reaches 5.0.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "♦" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

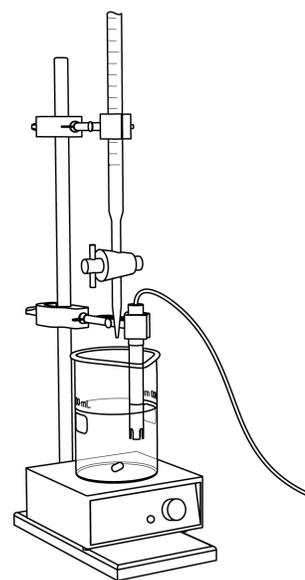
### Set Up

- Start a new experiment on the data collection system. ♦<sup>(1.2)</sup>
- Connect a pH sensor to the data collection system. ♦<sup>(2.1)</sup>
- Use pH 4 and pH 10 buffer solutions to calibrate the pH sensor. ♦<sup>(3.6)</sup>
- Monitor live data without recording (you will not need to collect data). ♦<sup>(6.1)</sup>

### Collect Data

- Which solution do you expect to take the greatest amount of NaOH solution to set the pH to 5?

The solution with the highest concentration since that solution has the most HOAc.



## Lab 19: Properties of Buffer Solutions

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6.  Set up a buret over a waste beaker (100-mL beaker).
7.  Rinse the buret with several milliliters of the 2.000 M NaOH solution:
- Ensure that the stopcock is closed and rinse the inside of the buret with several milliliters of the standardized NaOH solution.
  - Open the stopcock on the buret and drain the rinse NaOH into the waste container.
  - Repeat this process two more times.
8.  Why is it necessary to rinse the buret with the NaOH solution?

If there is any residual water or contaminant in the buret, it will dilute the NaOH and change its concentration. Rinsing eliminates any such contamination.

9.  Make sure the stopcock on the buret is in the “off” position and then use a funnel to fill the buret with about 50 mL of the 2.000 M NaOH solution (titrant).
10.  Drain a small amount of the titrant through the drop counter into the waste beaker (100-mL beaker) to remove any air in the tip of the buret.
11.  Why is it important to remove air from the tip of the buret?

Any air trapped in the buret tip is counted as volume of NaOH. If this happens, the recorded amount of NaOH added will be inaccurate.

12.  Add additional 2.000 M NaOH to the buret so the solution is above the zero mark. Allow some of the NaOH solution to drip into the waste container until the bottom of the meniscus is lined up with or just below the zero mark and record the initial reading in Table 3.

**Perform the following steps with each solution:**

**250 mL of 0.1 M HOAc**

**250 mL of 0.3 M HOAc**

**250 mL of 0.5 M HOAc**

13.  Transfer 250 mL of the acetic acid solution into a 400-mL beaker and set it on the magnetic stirrer (remove the waste beaker).
14.  Place a stirring bar into the solution and set the stirring to a gentle rate.
15.  Slowly add the NaOH solution by drops (1 to 2 per second) until the pH reaches 5.0.
16.  Record the final reading of the NaOH solution in Table 3.
17.  Calculate the volume of NaOH added (final reading minus initial reading) and record this value in Table 3 and Table 4.

Table 3: Amount of NaOH added to HOAc solutions to reach pH 5.0

Addition of NaOH	0.1 M HOAc	0.3 M HOAc	0.5 M HOAc
Initial reading of NaOH on the buret (mL)	0.0	0.0	0.0
Final reading of NaOH on the buret (mL)	9.20	25.20	42.00
Volume of NaOH added (mL)	9.20	25.20	42.00

18.  To measure buffer capacity, use the pipet to add the 6 M HCl solution, by drops, until the pH reaches 4.0. Record the volume added in Table 4.

19.  Which solution do you expect to need the greatest volume of the HCl solution to set the pH to 4; that is, which solution do you think will have the highest buffer capacity?

The solution with the highest concentration of acids and conjugate base will have the highest buffer capacity.

20.  To complete the process with all three HOAc solutions:

- Refill the buret with 2.000 M NaOH so the solution is above the zero mark. Allow some of the NaOH solution to drip into the beaker until the bottom of the meniscus is lined up with or just below the zero mark and record the initial reading in Table 3.
- Rinse the pH probe tip with deionized water.
- Remove the beaker and dispose of its contents according to the teacher's instructions.
- Repeat this process with the next solution.

21.  Clean up according to your teacher's instructions.

## Data Analysis

1.  Obtain the exact concentration of the NaOH solution from your teacher and record it in Table 2.

2.  Calculate the concentration of the conjugate base after adding the necessary amount of NaOH solution. Record the results for all three HOAc solutions in Table 4.

For the 0.1 M HOAc solution:

$$c_{A^-} = \frac{(2.000 \text{ M})(9.20 \text{ mL})}{(9.20 \text{ mL} + 250.00 \text{ mL})} = 7.10 \times 10^{-2} \text{ M}$$

3.  Calculate the concentration of the acid after adding the necessary amount of NaOH solution. Record the results for all three HOAc solutions in Table 4.

For the 0.1 M HOAc solution:

$$c_{HA} = \frac{(0.100 \text{ M})(250.00 \text{ mL}) - (2.000 \text{ M})(9.20 \text{ mL})}{(9.20 \text{ mL} + 250.00 \text{ mL})} = 2.55 \times 10^{-2} \text{ M}$$

## Lab 19: Properties of Buffer Solutions

4.  Calculate the predicted buffer capacity of the solutions. Record the results for all three HOAc solutions in Table 4.

For the 0.1 M HOAc solution:

$$x = \frac{(7.10 \times 10^{-2} \text{ M}) - (2.55 \times 10^{-2} \text{ M})10^{5.00-1-4.75}}{1 + 10^{5.00-1-4.75}} = 5.64 \times 10^{-2} \text{ M}$$

5.  Calculate the actual buffer capacity, which is the final concentration of HCl in the solution, for all 3 HOAc solutions. Record the results for all three solutions in Table 4.

$$c_{\text{HCl}} = \frac{(6.00 \text{ M})(1.80 \text{ mL})}{(9.20 \text{ mL} + 250.00 \text{ mL} + 1.80 \text{ mL})} = 4.14 \times 10^{-2} \text{ M}$$

Table 4: Buffer capacity measurements and calculation results

Parameters	0.1 M HOAc	0.3 M HOAc	0.5 M HOAc
Concentration of standardized NaOH solution (M)	2.000		
Concentration of standardized HCl solution (M)	6.00		
Volume of acetic acid solution (mL)	250.00	250.00	250.00
Volume of 2.0 M NaOH added to reach pH 5.00 (mL)	9.20	25.20	42.00
Volume of 6.0 M HCl solution added to reach pH 4.0	1.80	5.50	8.90
Concentration of conjugate base (M)	$7.10 \times 10^{-2}$	$1.83 \times 10^{-1}$	$2.88 \times 10^{-1}$
Concentration of remaining acid (M)	$2.55 \times 10^{-2}$	$8.94 \times 10^{-2}$	$1.40 \times 10^{-1}$
Predicted buffer capacity (M)	$5.64 \times 10^{-2}$	$1.41 \times 10^{-1}$	$2.23 \times 10^{-1}$
Actual buffer capacity (M)	$4.14 \times 10^{-2}$	$1.18 \times 10^{-1}$	$1.77 \times 10^{-1}$

### Analysis Questions

1. Does the pH of the buffer depend on the concentration of the conjugate base (salt) and the acid concentration? Explain your answer

The pH of a buffer does not depend on the actual value of either concentration. It depends only on the ratio of the concentration of the conjugate base (salt) and the acid.

**2. How does the buffer capacity change as the weak acid concentration increases?**

Increasing acid concentration results in increasing buffer capacity.

**3. In the example, the volume of the buffer solution was 121.20 mL. By adding 4.0 mL of 6 M HCl solution the pH changed one unit. How much will the pH change if the same amount of 6 M HCl is added to 121.20 mL of water?**

$$[\text{H}_3\text{O}^+] = \frac{(6.000 \text{ M})(4.00 \text{ mL})}{(121.20 \text{ mL} + 4.00 \text{ mL})} = 0.192 \text{ M}$$

$$\text{pH} = -\log[\text{H}_3\text{O}^+] = -\log(0.192) = 0.717$$

The pH would change from 7.00 to 0.717

**4. Consider the buffer capacity of the solution that was made with the 0.5 M HOAc. How would the pH change if the same amount of acid was added to pure water instead?**

The buffer capacity was 0.177 M. Adding 0.177 M acid to pure water will change the pH to

$$\text{pH} = -\log(0.177) = 0.75$$

**Synthesis Questions**

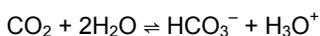
Use available resources to help you answer the following questions.

**1. If you had to design a buffer from phosphoric acid ( $\text{H}_3\text{PO}_4$ ), what conjugate bases could you use?**

You can use any salt made from  $\text{H}_3\text{PO}_4$ , for example,  $\text{Na}_3\text{PO}_4$ ,  $\text{NaH}_2\text{PO}_4$ , and  $\text{Na}_2\text{HPO}_4$ , and a strong base, such as KOH or NaOH.

**2. One of the many buffer systems in the human body is one that maintains the pH of blood. How do you think the pH of blood would be affected if too much  $\text{CO}_2$  is inhaled (in an environment where the air is stale)?**

Excess  $\text{CO}_2$  will cause the pH to shift towards more acidic values:

**Multiple Choice Questions**

Select the best answer or completion to each of the questions or incomplete statements below.

**1. The pH of a buffer:**

- A.** Depends on the concentration of the acid, the concentration of the conjugate base (salt), and the  $\text{p}K_a$  of the acid.
- B.** Depends on the ratio between the concentration of the acid, the concentration of the conjugate base (salt), and the  $\text{p}K_a$  of the acid.
- C.** Depends on the concentration of the acid, and the concentration of the conjugate base (salt) only.
- D.** Depends only on the  $\text{p}K_a$  of the acid.

## Lab 19: Properties of Buffer Solutions

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### 2. Buffer capacity for acids is:

- A. The amount of acid, in M, that increases the pH of a buffer to a measurable extent.
- B. The amount of acid, in M, that decreases the pH of a buffer to a measurable extent.
- C. The amount of acid, in M, that increases the pH of a buffer by one pH unit.
- D. The amount of acid, in M, that decreases the pH of a buffer by one pH unit.

### 3. A buffer can be made by mixing:

- A. A weak acid and a strong acid.
- B. A weak acid and a strong base, allowing the weak acid to be in excess.
- C. A weak base and a salt of the weak base.
- D. Water and a salt (conjugate base).

### 4. The pH of a buffer will:

- A. Not change at all when adding any amount of an acid or base.
- B. Not change substantially if the amount of acid or base added is less than the buffer capacity.
- C. Slightly increase as acid is added and slightly decrease as base is added.
- D. Change 0.1 pH units if the amount of acid added is equal to the buffer capacity.

## Extended Inquiry Suggestions

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Buffers can be discussed in context by analyzing the so called *buffer zone* in a titration of a weak acid with strong base. Please refer to the activity "Lab 24: Determining  $K_a$  by Half-Titration of a Weak Acid."

## Lab 20: Determination of Electrochemical Series

### Objectives

Students determine the half-reactions that relate to the anode and cathode of a galvanic cell. They also calculate the electromotive force for a battery, knowing the electrode materials used, by comparing the reduction potentials of metals.

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Constructing electrochemical cells
- ◆ Measuring the voltage produced in electrochemical cells with different metals and salt solutions as the anode and cathode

### Time Requirement

◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

#### *For each student or group:*

- |                                 |   |
|---------------------------------|---|
| ◆ Data collection system        | ◆ Circular filter paper, 11- cm diameter                                |
| ◆ Voltage sensor                | ◆ 1.0 M Zinc sulfate ( $\text{ZnSO}_4$ ), 10 mL <sup>1</sup>            |
| ◆ Beaker (6), 50-mL             | ◆ 1.0 M Iron sulfate ( $\text{FeSO}_4$ ), 10 mL <sup>2</sup>            |
| ◆ Glass plate (5 × 5 in)        | ◆ 1.0 M Copper sulfate ( $\text{CuSO}_4$ ), 10 mL <sup>3</sup>          |
| ◆ Disposable droppers (6), 1 mL | ◆ 1.0 M Silver nitrate ( $\text{AgNO}_3$ ), 10 mL <sup>4</sup>          |
| ◆ Iron strip, 1-cm × 1-cm       | ◆ 1.0 M Lead nitrate ( $\text{Pb}(\text{NO}_3)_2$ ), 10 mL <sup>5</sup> |
| ◆ Lead strip, 1-cm × 1-cm       | ◆ 1.0 M Sodium nitrate ( $\text{NaNO}_3$ ), 20.0 mL <sup>6</sup>        |
| ◆ Copper strip, 1-cm × 1-cm     | ◆ Sand paper  |
| ◆ Silver wire, 1-cm             | ◆ Scissors  |
| ◆ Zinc strip, 1-cm × 1-cm       |   |

<sup>1-6</sup>To prepare the solutions, refer to the Lab Preparation section.

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Redox (oxidation-reduction) reactions
- ◆ Redox potential
- ◆ Electrolytes
- ◆ Conductivity
- ◆ Molarity

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 8: Oxidation–Reduction Titration
- ◆ Lab 21: Electroplating

## Using Your Data Collection System

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a sensor to your data collection system ◆<sup>(2.1)</sup>
- ◆ Monitoring live data without recording it. ◆<sup>(6.1)</sup>

## Background

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The basis for an electrochemical cell is an oxidation-reduction (or "redox") reaction. This reaction can be divided into two half-reactions:

Oxidation half-reaction (loss of electrons) takes place at the anode, which is the positive electrode that the anions migrate to (hence the name anode).

Reduction half-reaction (gain of electrons) takes place at the cathode, which is the negative electrode that the cations migrate to (hence the name cathode).

Due to the difference in the electric potential between the two electrodes, an electrical current can be generated. The difference in potential is a result of the differences between the individual potentials of the metal electrodes with respect to the electrolyte, which is an electrically

conductive ionic solution. The electric potential also varies with temperature, concentration of electrolyte, and pressure.

The standard electrode potential,  $E^\circ$ , is the measure of potential of any electrode at standard ambient conditions (temperature at 298 K, solutes at 1 M, and gases at  $10^5$  Pa). Since the oxidation potential of a half-reaction is the negative of the reduction potential in a redox reaction, it is adequate to calculate either one of the potentials. The standard electrode potential is commonly written as the standard reduction potential.

In this experiment, students will compare the potentials of five different metals: copper, zinc, lead, silver, and iron, by measuring combinations of their half reactions.

## Pre-Lab Activity

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### *Setting the stage for the activity*

Standard potential is defined as the potential of an electrode that consists of a metal and the 1 M solution of the salt of that metal. The representation of an electrode is always "oxidation/reduction." For example,  $\text{Zn}^{2+}/\text{Zn}$  represents a zinc electrode.

If the solution portion of two electrodes is connected electrically, we have a galvanic cell. The potential measured between the two metals is the electromotive force. In one electrode, spontaneous reduction occurs; in the other, spontaneous oxidation occurs. For example, if a  $\text{Zn}^{2+}/\text{Zn}$  electrode and a  $\text{Cu}^{2+}/\text{Cu}$  electrode are connected, copper will oxidize zinc since the zinc is located above the copper (that is, zinc has lower reduction potential than copper, according to the table below).

In the table below, metals are arranged in increasing reduction (or decreasing oxidation) potential from top to bottom. That is, for a metal further down in the table, the greater its reduction potential is since it is easier to reduce it. The opposite is true as well: the higher up a metal is in the table, the greater its "oxidation potential," since it is easier to oxidize it.

In this activity, you will compare the oxidation and reduction potential of five metals. The point of reference will be copper and the other four metals are iron, lead, zinc, and silver. Metals located above the copper are expected to reduce copper (since for those, copper has the highest reduction potential). Metals located below the copper have higher reduction potential. Therefore, those will be reduced and the copper will be oxidized.

Table 1: Standard reduction potentials ( $E^\circ$ ) for selected metals

Metal	Reduction Half-Reaction	$E^\circ$ (V)
Lithium	$\text{Li}^+ + e^- \rightarrow \text{Li(s)}$	-3.04
Cesium	$\text{Cs}^+ + e^- \rightarrow \text{Cs(s)}$	-3.03
Rubidium	$\text{Rb}^+ + e^- \rightarrow \text{Rb(s)}$	-2.98
Potassium	$\text{K}^+ + e^- \rightarrow \text{K(s)}$	-2.93
Barium	$\text{Ba}^{2+} + 2e^- \rightarrow \text{Ba(s)}$	-2.91
Strontium	$\text{Sr}^{2+} + 2e^- \rightarrow \text{Sr(s)}$	-2.90
Calcium	$\text{Ca}^{2+} + 2e^- \rightarrow \text{Ca(s)}$	-2.87
Sodium	$\text{Na}^+ + e^- \rightarrow \text{Na(s)}$	-2.71
Magnesium	$\text{Mg}^{2+} + 2e^- \rightarrow \text{Mg(s)}$	-2.37
Berium	$\text{Be}^{2+} + 2e^- \rightarrow \text{Be(s)}$	-1.85
Aluminum	$\text{Al}^{3+} + 3e^- \rightarrow \text{Al(s)}$	-1.66
Manganese	$\text{Mn}^{2+} + 2e^- \rightarrow \text{Mn(s)}$	-1.18
Zinc	$\text{Zn}^{2+} + 2e^- \rightarrow \text{Zn(s)}$	-0.76
Chromium	$\text{Cr}^{3+} + 3e^- \rightarrow \text{Cr(s)}$	-0.74
Iron	$\text{Fe}^{2+} + 2e^- \rightarrow \text{Fe(s)}$	-0.44
Cadmium	$\text{Cd}^{2+} + 2e^- \rightarrow \text{Cd(s)}$	-0.40
Nickel	$\text{Ni}^{2+} + 2e^- \rightarrow \text{Ni(s)}$	-0.25
Tin	$\text{Sn}^{2+} + 2e^- \rightarrow \text{Sn(s)}$	-0.13
Lead	$\text{Pb}^{2+} + 2e^- \rightarrow \text{Pb(s)}$	-0.13
Hydrogen	$2\text{H}^+ + 2e^- \rightarrow \text{H}_2(\text{s})$	0.00
Bismuth	$\text{Bi}^{3+} + 3e^- \rightarrow \text{Bi(s)}$	+0.32
Copper	$\text{Cu}^{2+} + 2e^- \rightarrow \text{Cu(s)}$	+0.34
Silver	$\text{Ag}^+ + e^- \rightarrow \text{Ag(s)}$	+0.80
Mercury	$\text{Hg}_2^{2+} + 2e^- \rightarrow 2\text{Hg(l)}$	+0.80
Mercury	$\text{Hg}^{2+} + 2e^- \rightarrow \text{Hg(l)}$	+0.85
Palladium	$\text{Pd}^{2+} + 2e^- \rightarrow \text{Pd(s)}$	+0.91
Platinum	$\text{Pt}^{2+} + 2e^- \rightarrow \text{Pt(s)}$	+1.19
Cerium	$\text{Ce}^{4+} + e^- \rightarrow \text{Ce}^{3+}(\text{s})$	+1.44
Gold	$\text{Au}^+ + e^- \rightarrow \text{Au(s)}$	+1.83

### Example calculation to try

A galvanic cell is assembled from a  $\text{Zn}^{2+}/\text{Zn}$  and a  $\text{Cu}^{2+}/\text{Cu}$  electrode:



where the single vertical bars represent the junction between solid and liquid, and the double vertical bars represent the junction between the two solutions.

By definition, the cathode is on the right and the anode is on the left. Therefore, in the right electrode there is reduction and in the left electrode there is oxidation. In this example, the zinc is oxidized and the copper is reduced (copper has the greater reduction potential). The standard reduction potentials of these electrodes are as follows:



The electromotive force can be calculated as:

$$E = (\text{Potential of Cathode}) - (\text{Potential of Anode})$$

$$E = E_1 - E_2 = (+0.34 \text{ V}) - (-0.76 \text{ V}) = +1.10 \text{ V}$$

This would be the potential shown by a voltmeter. A galvanic cell assembled in this fashion is called a Daniell cell and can be represented by the following diagram:



**1. Would silver form a galvanic cell with copper? Explain your answer.**

Yes, it would, because the standard potential of the two metals are different.

**2. If your answer is "yes" to the question above, which electrode would be the cathode and which one would be the anode? Explain.**

Since copper has a lower reduction potential than silver, silver will oxidize copper; therefore, silver will be reduced. The cathode is the electrode where the reduction takes place, therefore the cathode will be the  $\text{Ag}^+/\text{Ag}$  electrode and the anode will be the  $\text{Cu}^{2+}/\text{Cu}$  electrode.

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

- 1. 1.0 M  $\text{ZnSO}_4$ :** Dissolve 71.89 g of  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  in some distilled water in a 250-mL volumetric flask and fill it to the mark.
- 2. 1.0 M  $\text{FeSO}_4$ :** Dissolve 69.50 g of  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  in some distilled water in a 250-mL volumetric flask and fill it to the mark.
- 3. 1.0 M  $\text{CuSO}_4$ :** Dissolve 62.42 g of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  in some distilled water in a 250-mL volumetric flask and fill it to the mark.
- 4. 1.0 M  $\text{AgNO}_3$ :** Dissolve 42.46 g of  $\text{AgNO}_3$  in some distilled water in a 250-mL volumetric flask and fill it to the mark.
- 5. 1.0 M  $\text{Pb}(\text{NO}_3)_2$ :** Dissolve 82.80 g of  $\text{Pb}(\text{NO}_3)_2$  in some distilled water in a 250-mL volumetric flask and fill it to the mark.
- 6. 1.0 M  $\text{NaNO}_3$ :** Dissolve 85.00 g of  $\text{NaNO}_3$  in some distilled water in a 250-mL volumetric flask and fill it to the mark.

## Safety

Add these important safety precautions to your normal laboratory procedures:

- ◆ Dispose of solutions properly.
- ◆ Wear safety goggles throughout this activity.
- ◆ In case of contact with skin, chemicals should be washed off with large amounts of water.

### Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

4	5	3	1	2
Measure and record the potential between any two of the metal strips, making sure you get a positive value.	Rank the metals in the order of their reduction potential, from greatest to least.	Complete the electrodes and connect them using $\text{NaNO}_3$ solution.	Draw five circles on a circular filter paper around its edge and cut out the wedges between the circles.	Begin creating the electrodes by putting the metal ion solutions on the filter paper.

### Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

#### Set Up

- Draw five small circles with connecting lines on a piece of circular filter paper (11-cm diameter), as shown in Figure 1. Label the circles  $M_1$ ,  $M_2$ ,  $M_3$ ,  $M_4$ , and  $M_5$ .

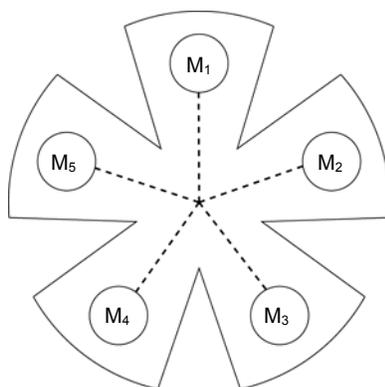


Figure 1: Filter paper diagram

- Using a pair of scissors, cut wedges between the circles as shown.

3.  Place the filter paper on top of the glass plate.
4.  Obtain pieces of each of the five test metals. Sand each piece of metal on both sides so that a good electrical connection can be made.
5.  Using a separate dropper for each solution, place three drops of each metal ion solution on the appropriate circle ( $M_1$ ,  $M_2$  etc.). Then, according to Table 2, place the corresponding piece of metal on the spot with its respective cation. The top side of the metal should be kept dry. These are the electrodes.

Table 2: Metals and salt solutions for setting up each half-cell

Material	$M_1$	$M_2$	$M_3$	$M_4$	$M_5$
Metal	Copper	Zinc	Lead	Silver	Iron
Salt Solution	Copper sulfate	Zinc sulfate	Lead nitrate	Silver nitrate	Iron sulfate

6.  Based on the table above, complete Table 3a and 3b with your predictions of which metals will be oxidized and which ones reduced when coupled with copper. Provide the oxidation/reduction half-reaction for each metal and for the copper!

Table 3a: Predicted oxidation and reduction processes

<b>Copper reduced</b>	$\text{Cu}^{2+}(\text{aq}) + 2\text{e}^- \rightarrow \text{Cu}(\text{s})$
<b>Other metals oxidized</b>	$\text{Zn}(\text{s}) \rightarrow \text{Zn}^{2+}(\text{aq}) + 2\text{e}^-$ $\text{Fe}(\text{s}) \rightarrow \text{Fe}^{2+}(\text{aq}) + 2\text{e}^-$

Table 3b: Predicted oxidation and reduction processes

<b>Copper oxidized</b>	$\text{Cu}(\text{s}) \rightarrow \text{Cu}^{2+}(\text{aq}) + 2\text{e}^-$
<b>Other metals reduced</b>	$\text{Pb}^{2+}(\text{aq}) + 2\text{e}^- \rightarrow \text{Pb}(\text{s})$ $\text{Ag}^+(\text{aq}) + 2\text{e}^- \rightarrow \text{Cu}(\text{s})$

7.  Add enough 1.0 M sodium nitrate ( $\text{NaNO}_3$ ) solution to make a continuous trail along a line drawn between each circle and the center of the filter paper. You may have to dampen the filter paper with more  $\text{NaNO}_3$  during the experiment.

**Note:** The  $\text{NaNO}_3$  trace is the liquid-to-liquid junction between the electrodes. Any two of the electrodes coupled represent a galvanic cell.

8.  Why do you think the  $\text{NaNO}_3$  solution is being used?

The  $\text{NaNO}_3$  trace is the liquid-to-liquid junction between the electrodes. Any two of the electrodes coupled represent a galvanic cell.

## Lab 20: Determination of Electrochemical Series

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### Collect Data

Use  $M_1$  (copper) as the reference metal. You will measure the potential of four cells by connecting  $M_1$  to  $M_2$  (copper to zinc),  $M_1$  to  $M_3$  (copper to lead),  $M_1$  to  $M_4$  (copper to silver), and  $M_1$  to  $M_5$  (copper to iron).

9.  Start a new experiment on the data collection system.  $\diamond^{(1.2)}$
10.  Connect a voltage sensor to the data collection system.  $\diamond^{(2.1)}$
11.  Monitor live data without recording.  $\diamond^{(6.1)}$
12.  Touch the tip of the red (+) wire of the voltage sensor to one metal sample (for example,  $M_1$ ) and the tip of the black (-) wire to the other metal sample (for example,  $M_2$ ). If the voltage reading is below 0.00 V, reverse the ends of the voltage sensor, that is, switch the red (+) end of the sensor to  $M_2$  and the black (-) end of the sensor to  $M_1$ .
13.  When the voltage reading stabilizes, record the voltage for the half-cell (half-reaction) combination and the color of the lead, or clip that is touching each of the metals, in Table 4 in the Data Analysis section.
14.  Use the same procedure to measure the potential of the other three "half cells" with copper,  $M_1$ , as the reference electrode.  
  
**Note:** If you get a voltage reading of 0.00 V or a fluctuating reading, add more  $\text{NaNO}_3$  solution along the lines connecting the metal spots.
15.  Analyze your data for copper and make predictions about the other possible half-cell combinations using the same metals and solutions you used in this experiment.

### Data Analysis

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1.  Complete the table below with your data. Use the Standard Reduction Potential table to calculate the expected potential difference between the two electrodes.

**Note:**  $E = (\text{Potential of Cathode}) - (\text{Potential of Anode})$

Table 4: Comparison of measured and calculated cell electromotive force

Half-cell Combination	Half-cell Standard Reduction Potential		Half-cell Standard Reduction Potential		Potential Using Standard Reduction Table	Measured Voltage
	Cathode (Red Wire) (+)	Voltage	Anode (Black Wire) (-)	Voltage		
M <sub>1</sub> , M <sub>2</sub>	M <sub>1</sub>	0.34 V	M <sub>2</sub>	-0.76 V	1.10 V	1.10 V
M <sub>1</sub> , M <sub>3</sub>	M <sub>1</sub>	0.34 V	M <sub>3</sub>	-0.13 V	0.47 V	0.47 V
M <sub>1</sub> , M <sub>4</sub>	M <sub>4</sub>	0.80 V	M <sub>1</sub>	0.34 V	0.46 V	0.45 V
M <sub>1</sub> , M <sub>5</sub>	M <sub>1</sub>	0.34 V	M <sub>5</sub>	-0.44 V	0.78 V	0.71 V

2.  The red lead represents the cathode, where reduction takes place. The black lead represents the anode, where oxidation takes place. By arranging the clips so a positive voltage is obtained, you have determined that the metal at the red clip is more easily reduced than the one at the black clip.

Which metals were reduced by copper? Which metals oxidized copper?

Silver was reduced by copper and all other metals were oxidized by copper.

3.  Based on your results, arrange the five metals (including copper, M<sub>1</sub>) in order of reduction potential from the highest reduction potential at the top to the lowest reduction potential at the bottom.

**Hint:** In relation to copper, a metal that has a higher reduction potential will result in a higher potential of the cell. The metals that reduced copper are more easily oxidized than copper, so their reduction potentials will be lower. Also, if a metal has a higher oxidation potential, it will have a lower reduction potential.

Zinc, iron, lead, copper, silver

## Analysis Questions

1. Which metal turned out to be the easiest to oxidize?

The easiest to oxidize was Zn.

2. Which metal was the most difficult to oxidize?

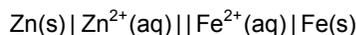
The most difficult to oxidize was silver.

3. Which electrode combination gave the most electromotive force?

The potential between the metal with the largest reduction potential and the metal with the smallest reduction potential gives the largest electromotive force. Therefore, the two metals are zinc and silver.

**4. What is the cell diagram representation of a galvanic cell assembled from Zn<sup>2+</sup>/Zn and Fe<sup>2+</sup>/Fe?**

Since zinc has more negative reduction potential than iron in the reduction potential series, iron will oxidize zinc which means iron will be the cathode (listed on the right) and zinc the anode (listed on the left):



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## Synthesis Questions

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Use available resources to help you answer the following questions.

**1. Can you think of any metal that is more difficult to oxidize than silver? What are the consequences of a metal being difficult to oxidize in terms of the properties of the metal?**

Gold, platinum, and mercury have an even lower reduction potential than silver. The fact that a metal is hard to oxidize means that the metal form is more stable than the ion form of that specific metal. Metals that are hard to oxidize do not easily react, which is a common property of precious metals.

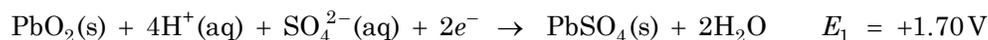
**2. Do you think you would have obtained different potential readings if the nitrate salt of the metals would have been used instead of the sulfate salts?**

No, the potential only depends on the redox properties of the metals.

**3. If you were to assemble a Daniell cell from bigger pieces of copper and zinc, and light a light bulb by placing it between the two metals until the galvanic cell was depleted, what would happen to the pieces of copper and zinc?**

Since the Cu<sup>2+</sup> ions are reduced, some copper would be deposited on the surface of the copper electrode, and since the zinc is oxidized, some zinc would go into the solution and the piece of zinc would be smaller.

**4. In lead acid batteries, which are used in cars to start the engine, the half-reactions for the two connected electrodes are:**



**4a. Which electrode is the cathode and which is the anode? Explain your answer!**

The redox potential is less for the second reaction. Therefore it will be the oxidation half-reaction which means the first reaction will be the reduction half-reaction. Since the cathode is where the reduction occurs, the first electrode will be the cathode and the second one will be the anode.

**4b. What is the electromotive force of this cell?**

The electromotive force of the galvanic cell will be, since the second reaction will be occurring in reverse:

$$E_{\text{cell}} = E_1 - E_2 = (+1.70 \text{ V}) - (-0.13 \text{ V}) = +1.83 \text{ V}$$

It is worth mentioning that six of these cells are connected in series to provide the approximate 12 V that car batteries provide. Also, the process is reversible and goes backwards when the battery is being charged.

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**Multiple Choice Questions**

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Select the best answer or completion to each of the questions or incomplete statements below.

- 1. In the  $\text{Fe}^{2+}/\text{Fe}$  and  $\text{Cu}^{2+}/\text{Cu}$  electrode pair:**
  - A. The Cu will be the anode
  - B. The  $\text{CuSO}_4$  solution will be the anode
  - C. The Cu will be the cathode**
  - D. The  $\text{CuSO}_4$  solution will be the cathode
- 2. The chemical reaction on the cathode is:**
  - A. The oxidation of the metal plate
  - B. The reduction of the metal plate
  - C. Reduction of the metal ion**
  - D. Oxidation of the metal ion
- 3. Copper could oxidize:**
  - A. All of the other four metals
  - B. All of the other metals except silver**
  - C. All of the other metals except silver and lead
  - D. Only zinc
- 4. A metal can oxidize any other metal that has a:**
  - A. More negative oxidation potential
  - B. Less negative reduction potential
  - C. More positive reduction potential
  - D. Less positive reduction potential**

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**Extended Inquiry Suggestions**

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An excellent demonstration of an electrochemical series is the method of preventing corrosion of steel structures:

Take two small sections of a steel or iron pipe. Connect a piece of zinc to one of them so there is an electrical connection between the zinc and the pipe. Place both sections of pipe into diluted (~0.01 M) HCl solution for one week.

What students should see is that the pipe without the zinc corrodes while the zinc pipe doesn't. Students should, however, also notice that the piece of zinc gets smaller. Steel structures do not corrode until there is no zinc present, therefore, the zinc plate has to be replaced periodically.



## Lab 21: Electroplating

### Objectives

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Students construct an electrochemical cell that deposits copper onto another metal surface. They also apply Faraday's law to relate the total electric charge to the mass of metal deposited.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Determining the change of mass of each electrode due to electroplating
- ◆ Comparing the experimental changes in mass at each electrode to the expected change in mass due to the total electrical charge, calculated using Faraday's law

### Time Requirement

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◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	120 minutes

### Materials and Equipment

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#### *For each student or group:*

- |  |  |
|--|--|
| ◆ Data collection system                   | ◆ Magnetic stir plate and stir bar                             |
| ◆ Voltage/current sensor                   | ◆ Balance  |
| ◆ DC power supply                          | ◆ Metal object (key or spoon)                                  |
| ◆ Banana plug cords, red (2) and black (1) | ◆ Copper strip or heavy gauge copper wire (3 in)               |
| ◆ Alligator clips, red (1) and black (1)   | ◆ 1.0 M Copper sulfate ( $\text{CuSO}_4$ ), 50 mL <sup>1</sup> |
| ◆ Ring stand                               | ◆ Sand paper   |
| ◆ Clamps (2)                               | ◆ Electrical tape  |
| ◆ Beaker, 100-mL                           | ◆ Paper towel, 1 sheet   |

<sup>1</sup>To prepare the solution, refer to the Lab Preparation section.

### Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Redox reactions
- ◆ Redox potential

## Electroplating

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- ◆ Electrolytes
- ◆ Conductivity
- ◆ Molarity

### Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 8: Oxidation–Reduction Titration
- ◆ Lab 20: Determination of Electrochemical Series

### Using Your Data Collection System

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a sensor to your data collection system ◆<sup>(2.1)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>
- ◆ Finding the area under a curve. ◆<sup>(9.7)</sup>

### Background

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Electroplating is the process of coating an electrically conductive object with a layer of metal using an electric current. The process, also known as electrodeposition, is used to improve the appearance and increase hardness and corrosion resistance of the plated objects.

The cathode (article to be plated) in the electroplating cell is connected to the negative terminal of a direct current power supply. The anode is connected to the positive terminal of the power supply. The cathode and anode are immersed in an electrolyte, which is an electrically conductive ionic solution. When the power supply is turned on, the metal at the anode is oxidized from the 0 valence electron state to form positively charged cations. These cations migrate through the solution toward the negatively charged cathode. At the cathode, the cations are reduced and are deposited in their metallic, 0 valence electron state onto the surface of the cathode.

The electricity generated by the moving electrons can be measured as a rate of electric current flow in amperes, A. An ampere is equal to 1 coulomb, C, of charge per second. You can calculate the total amount of charge  $Q$  in coulombs by multiplying the current flow  $I$  by the amount of time  $t$  the current is flowing:

$$Q = It$$

1 mol of a metal with +1 charge requires 96,485 C to be reduced to neutral state:

$$F = (6.2 \times 10^{-19} \text{ C}) \left( 6.02 \times 10^{23} \frac{1}{\text{mol}} \right) = 96,485 \frac{\text{C}}{\text{mol}}$$

where:

$F$  = Faraday's constant (96,485 C/mol)

$6.2 \times 10^{-19} \text{ C}$  = the charge of one electron

$6.02 \times 10^{23} \text{ mol}^{-1}$  = Avogadro's number

The relationship between the charge that passes through the solution and the amount of metal ion (in our case  $\text{Cu}^{2+}$  ion) neutralized is

$$It = zF \frac{m}{AW}$$

$$m = \frac{It AW}{zF}$$

where:

$m$  = mass of neutralized ion or deposited metal (g)

$I$  = current (A)

$t$  = time of electroplating (s)

$AW$  = molar mass of copper (g/mol)

$z$  = charge of copper (+2)

$F$  = Faraday constant (96,485 C)

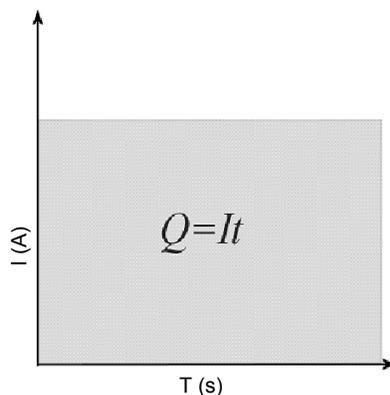
## Pre-Lab Activity

### Setting the stage for the activity

Students will assemble an electroplating cell from  $\text{CuSO}_4$  solution, a key or spoon, and a piece of copper strip. The key or spoon will be connected to the negative terminal of the power supply (the cathode). The positive terminal of the power supply will be connected to the positive terminal of the current sensor. The negative terminal of the current sensor will be connected to the copper plate.

The two metal objects will then be immersed in the  $\text{CuSO}_4$  solution. The flow of electrons moves from the negative to the positive terminal of the power supply. From the negative terminal, the electrons flow to the surface of the key or spoon where they get picked up by the  $\text{Cu}^{2+}$  ions and the neutralized copper is deposited onto the surface of the object. On the other electrode, copper atoms lose two electrons and go into solution as  $\text{Cu}^{2+}$  ions. The electrons left by the copper ions flow back into the power supply.

The current and the time of the electroplating will be monitored with a current sensor.



Total charge is shown as the area of current  $\times$  time

The total charge used for electroplating can be calculated as the product of the current and time. This is the area shown on the graph of current versus time. If the current is kept constant, the total charge is simply the area of a rectangle. If the current is inconsistent, determine the area under the line using the area tool from the data collection system.

### Example calculation to try

The mass of a silver spoon was measured and found to be 15.456 g. It was mounted in an electroplating cell as the cathode. A piece of copper strip (4.556 g) was mounted as the anode. The cell was filled with 1 M  $\text{CuSO}_4$  solution. Wiring was completed from the negative terminal of the power supply to the spoon, from the copper plate to the negative terminal of a current sensor, and from the positive terminal of the current sensor to the positive terminal of the power supply.

The power supply was turned on and the current was maintained at 1.1 A for 30 min. The spoon was then removed, washed and dried. The mass of the spoon was found to be 16.066 g. The copper strip was washed and dried, and was found to be 4.010 g. This indicates that 0.546 g of copper dissolved:

$$4.556 \text{ g} - 4.010 \text{ g} = 0.546 \text{ g}$$

The charge that passed through the cell was

$$Q = It = (1.1 \text{ A})(1,800 \text{ s}) = 1,980 \text{ A s} = 1,980 \text{ C}$$

The mass of copper equivalent to the total charge that passed through the cell is

$$m = \frac{ItAW}{zF} = \frac{(1.1 \text{ A})(1,800 \text{ s})(63.55 \frac{\text{g}}{\text{mol}} \text{ Cu})}{(2)(96,485 \frac{\text{A s}}{\text{mol}})} = 0.652 \text{ g Cu}$$

The actual amount of deposited copper was

$$16.066 \text{ g} - 15.456 \text{ g} = 0.610 \text{ g}$$

Table 1: Compare the theoretical and actual amounts of copper deposited

	Electroplated Object	Copper Strip
Original mass (g)	15.456	4.556
Mass after electroplating (g)	16.066	4.010
Change of mass (g)	+0.610	-0.546
Theoretical change of mass (g)	0.652	-0.652
Yield (%)*	93.5	83.7

\*The percent yield compares the experimental to the theoretical amount of copper deposited or dissolved.

**1.** Do you think it matters how much of the surface of the metal objects is in contact with the solution and if so, why?

More ions interact with the surface of the electrode when a greater surface area contacts the solution within a certain amount time, producing a larger current.

**2.** Why do you think the experimental quantity is less than the theoretical?

Due to the electrical resistance of the solution and secondary chemical processes, the utilization of the electricity is less than 100%.

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

- 1. 1.0 M CuSO<sub>4</sub>:** Dissolve 62.42 g of CuSO<sub>4</sub>·5H<sub>2</sub>O in some distilled water in a 250-mL volumetric flask and fill it to the mark.

## Safety

Add this important safety precaution to your normal laboratory procedures:

- ◆ In case of contact with skin, CuSO<sub>4</sub> solution should be washed off with large amounts water.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

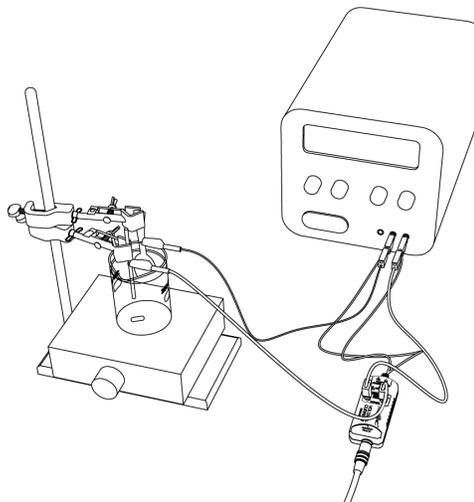
1	2	4	5	3
Obtain the mass of the metal spoon or key to be electroplated and the copper strip that will be used as the anode.	Create the electrochemical cell: Mount the copper strip as the anode and the object to be electroplated as the cathode.	Remove the electrodes and determine the mass of the copper strip and spoon or key.	Calculate the efficiency of the actual versus theoretical amount of copper deposited.	Run the electrolysis for 30 minutes.

## Procedure with Inquiry

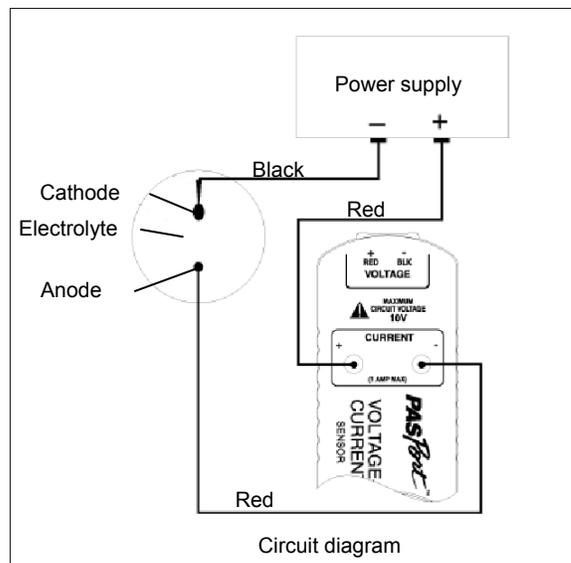
After you complete a step (or answer a question), place a check mark in the box () next to that step.

### Set Up

- Place the beaker containing the copper sulfate solution on a magnetic stir plate and add a magnetic stirring bar.
- Start a new experiment on the data collection system. ♦<sup>(1,2)</sup>
- Connect a voltage/current sensor to the data collection system. ♦<sup>(2,1)</sup>
- Carefully clean and dry the copper wire and spoon or key with steel wool.
- Obtain the mass of the wire and spoon or key to the nearest 0.001 g and record the mass in Table 3.
- Use a red alligator clip to attach one end of the red banana plug patch cord to one end of the heavy gauge copper wire.
- Use a black alligator clip to attach one end of the black banana plug patch cord to the end of the handle of the metal spoon or key.



8.  Wrap electrical tape around the spoon or key and copper wire near one end to provide insulation. Mount clamps on a support rod and clamp the wire and spoon or key in place by the insulated portion.
9.  Position the end of the heavy gauge copper wire and the spoon or key so that they are immersed in the solution in the beaker.
10.  Plug the black patch cord from the spoon or key into the negative (black) terminal of the power supply.
11.  Connect the red patch cord from the positive (red) terminal of the power supply to the positive (+) jack on the current input of the voltage/current sensor.
12.  Connect the red patch cord connected to the heavy gauge copper wire to the negative (-) jack on the voltage/current sensor.



**Note:** The Voltage Sensor leads are not used during this experiment.

13.  What would the consequences be if you switched the leads on the power supply?

Switching the leads on the power supply would reverse the sign of the current and therefore the electroplating process would be reversed: the spoon or key would dissolve while copper would be deposited on the surface of the copper plate.

### Collect Data

14.  Display current on the y-axis of a graph with time on the x-axis.  $\diamond^{(7.1.1)}$
15.  Start data recording.  $\diamond^{(6.2)}$
16.  Turn on the power supply and adjust the current and voltage until the current reads 0.2 A.
17.  Adjust the magnetic stirrer to achieve a reasonable amount of mixing; be sure to avoid splashing. Make sure the stir bar does not hit either of the electrodes.
18.  Run the electroplating process for at least an hour, but no longer than two.
19.  Stop the data recording  $\diamond^{(6.2)}$  and turn off the power supply.

## Electroplating

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20.  Find the area under the Current versus Time curve. ♦<sup>(9.7)</sup> Record the value, in coulombs, in Table 2.
21.  Carefully remove the metal spoon or key and the copper wire, very carefully rinse it with distilled water, and place them on a paper towel. Gently blot and air dry the spoon or key and the wire.
- Note:** Do not rub the surface of the spoon or key as the deposited copper is loosely attached to the surface.
22.  Measure again the mass of the copper wire and metal spoon or key to the nearest 0.001 g and record these values in Table 3.

## Data Analysis

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1.  Calculate the mass of copper equivalent to the total charge (area under  $I$  versus  $t$  curve) that passed through the cell and enter it in Table 2.

$$Q = It = (0.210\text{ A})(4637\text{ s}) = 974\text{ A s} = 9.74 \times 10^2\text{ C}$$

$$m = \frac{ItAW}{zF} = \frac{(0.210\text{ A})(4637\text{ s})(63.55\frac{\text{g}}{\text{mol}}\text{ Cu})}{(2)(96,485\frac{\text{A s}}{\text{mol}})} = 0.321\text{ g Cu}$$

Table 2: Theoretical amount of copper deposited due to the total charge

Parameters	Calculated Values
Total charge (area under $I$ versus $t$ curve)(C):	974
Theoretical amount of Cu deposited (g):	0.321

2.  Complete Table 3. Calculate the percent yield based on the theoretical and experimental amount of copper deposited.

The actual amount of deposited copper was:  $13.996\text{ g} - 13.685\text{ g} = 0.311\text{ g}$

The amount of dissolved copper was:  $13.736\text{ g} - 13.422\text{ g} = 0.314\text{ g}$

Table 3: Compare the theoretical and actual amounts of copper deposited and dissolved

	Electroplated Object	Copper Strip
Original mass (g)	13.685	13.736
Mass after electroplating (g)	13.996	13.422
Change of mass (g)	+ 0.311	- 0.314
Theoretical change of mass (g)	0.321	- 0.321
Yield (%)*	96.9	97.8

\*The percent yield compares the experimental to the theoretical amount of copper.

**3. □** Calculate the percent yield.

The percent yield of copper deposited is:

$$\frac{0.311 \text{ g of copper (experimental)}}{0.321 \text{ g of copper (theoretical)}} \times 100 = 96.9\%$$

The percent yield of copper dissolved is:

$$\frac{0.314 \text{ g of copper (experimental)}}{0.321 \text{ g of copper (theoretical)}} \times 100 = 97.8\%$$

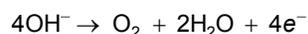
## Analysis Questions

**1. Propose an explanation for the less than 100 % yield.**

The solution has some electrical resistance. Therefore, some of the charge was lost to the solution.

**2. Was the amount of copper lost from the copper wire the same as the amount of copper gained on the metal object? If not, propose an explanation. (Hint: what other ions can migrate to the anode and what reaction can they become part of?)**

The loss of copper from the wire was slightly greater than the gain of copper on the metal object. Some of the charge could have been used for other reactions, like the oxidation of the  $\text{OH}^-$  ions which are present inherently from the dissociation of the water molecules:



**3. The color of the deposited copper is slightly different from the original color of the object. (Hint: Consider the difference between the particle size in the original object and the deposited copper)**

In the copper strip, the copper is "molded," while the deposited copper is formed from atomic-sized copper atoms. The different particle size results in different color.

## Electroplating

**4.** Earlier we identified one parameter that could change the current. Can you think of changing another parameter of the cell (that is, the two metal electrodes, the solution, or the beaker) that can potentially increase the current? (Hint: reducing the electrical resistance of the solution would increase the current)

Moving the electrodes closer reduces the electrical resistance of the solution.

## Synthesis Questions

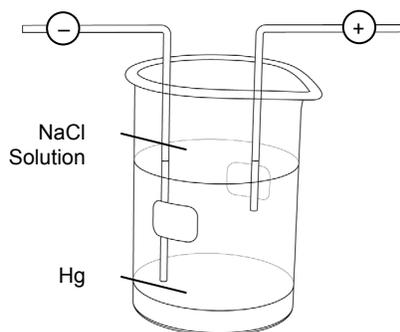
Use available resources to help you answer the following questions.

**1.** Can you propose a setup to silver plate a spoon?

The same cell configuration with a silver strip instead of a copper strip and the object to be silver plated mounted in the place of the copper object with  $\text{AgNO}_3$  solution in the cell.

**2.** Electrolysis is a commonly used industrial technique. The diagram of the cell below show how  $\text{NaOH}$  is produced by the electrolysis of  $\text{NaCl}$  solution. Mercury is used to promote the reduction of  $\text{Na}^+$  ions on the cathode (-) in the form of  $\text{Na}^0$ . The neutral sodium will then dissolve in mercury as  $\text{NaHg}$  (called sodium amalgam).

Hydrolysis of  $\text{NaHg}$  yields the desired  $\text{NaOH}$  solution. What do you think is happening on the anode during the electrolysis? (Hint: what anions could get oxidized in the  $\text{NaCl}$  solution?)



Of the two anions,  $\text{OH}^-$  and  $\text{Cl}^-$ , the  $\text{Cl}^-$  ion gets oxidized to form chlorine gas:  $2\text{Cl}^- \rightarrow \text{Cl}_2 + 2e^-$

**3.** All components of hot rods and Harley Davidson bikes that have a shiny chrome "finish" are made using electroplating. Are those components mounted as anodes or cathodes in the electroplating cells?

They are located in place of the copper object which was the cathode.

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**Multiple Choice Questions**

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Select the best answer or completion to each of the questions or incomplete statements below.

**1. Copper on the anode will:**

- A. Get reduced.
- B. Get oxidized.
- C. Not react. It will only get reduced on the cathode to form the copper deposit.
- D. Not react. It will only get oxidized on the cathode to form the copper deposit.

**2. If you mount an iron object instead of a copper one as the cathode, with the copper strip as the anode:**

- A. The copper won't be deposited on the surface of the iron object.
- B. The iron object will dissolve.
- C. **The copper will be deposited on the iron object the same way as on the surface of a copper object.**
- D. There will be no redox reaction as the metal for a  $\text{Cu}/\text{Cu}^{2+}$  electrode is missing.

**3. Using  $\text{Cu}(\text{NO}_3)_2$  instead of  $\text{CuSO}_4$ :**

- A. Will result in a different anode reaction and prevent the copper from being deposited.
- B. Will result in a different cathode reaction and prevent the copper from being deposited.
- C. Will result in forming  $(\text{NO}_3)_2$  gas on the anode.
- D. **Will not make a difference in the process of copper deposition on the cathode.**

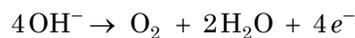
**4. Changing the concentration of the solution will:**

- A. Prevent the deposition of copper on the cathode.
- B. Prevent the reduction of copper on the electrode.
- C. **Influence the electrical resistance of the solution and, therefore, the efficiency of the electrolysis.**
- D. Not make any difference.

### **Extended Inquiry Suggestions**

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Have students apply electro-oxidation (also called “anodizing”) to an aluminum object. Electro-oxidation is commonly used as a method to colorize aluminum objects. The process is based on the fact that aluminum objects are covered and protected by a thin oxide layer. Mounting the aluminum object as an anode exposes the aluminum object to the process:



In this process, atomic oxygen is formed first. This is a very strong oxidant (the same as ozone). Atomic oxygen can oxidize aluminum in a special way. If the oxidation is taking place in a solution that is colored, due to the oxidation by atomic oxygen, the color of the solution becomes embedded in the porous surface of the aluminum metal. For example, for orange color,  $\text{K}_2\text{Cr}_2\text{O}_7$  solution is commonly used. It is quite intriguing to electrolyze an aluminum object in a  $\text{K}_2\text{Cr}_2\text{O}_7$  solution to turn the object orange. The object would have to be mounted as the anode (+).

## Lab 22a: Organic Synthesis I—Preparation

### Objectives

Organic Synthesis I: Students synthesize an organic compound (aspirin).

Organic Synthesis II: Students analyze the purity of the aspirin they synthesized.

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Organizing and conducting the steps of a chemical reaction (Organic Synthesis I)
- ◆ Performing separation processes to isolate the product (Organic Synthesis I)
- ◆ Determining the percent yield of the reaction (Organic Synthesis I)
- ◆ Performing qualitative and quantitative analytical methods (Organic Synthesis II)

### Time Requirement

◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	120 minutes

### Materials and Equipment

#### *For each student or group:*

- |                                      |  |
|--------------------------------------|--|
| ◆ Data collection system             | ◆ Filter paper                                     |
| ◆ Stainless steel temperature sensor | ◆ Salicylic acid ( $C_7H_6O_3$ ), 2 g              |
| ◆ Ring stand                         | ◆ Acetic anhydride ( $C_4H_6O_3$ ), 4 mL           |
| ◆ Clamp (2)                          | ◆ Concentrated phosphoric acid ( $H_3PO_4$ ), 1 mL |
| ◆ Erlenmeyer flask, 125-mL           | ◆ Wash bottle with distilled water                 |
| ◆ Graduated cylinder, 10-mL          | ◆ Eye dropper                                      |
| ◆ Beaker, 100-mL                     | ◆ Rubber policeman                                 |
| ◆ Beaker, 400-mL                     | ◆ Ice cold distilled water, 50 mL <sup>1</sup>     |
| ◆ Hot plate                          | ◆ Ice for ice bath, 300 mL <sup>2</sup>            |
| ◆ Filter flask                       | ◆ Forceps  |
| ◆ Büchner funnel                     |  |

<sup>1,2</sup> To prepare these items, refer to the Lab Preparation section.

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Stoichiometry
- ◆ Titration
- ◆ Acid-base reactions

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 6: Standardizing a Solution of Sodium Hydroxide
- ◆ Lab 7: Acid–Base Titration
- ◆ Lab 15a: Synthesis of a Coordination Compound
- ◆ Lab 22b: Organic Synthesis II—Analysis

## Using Your Data Collection System

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

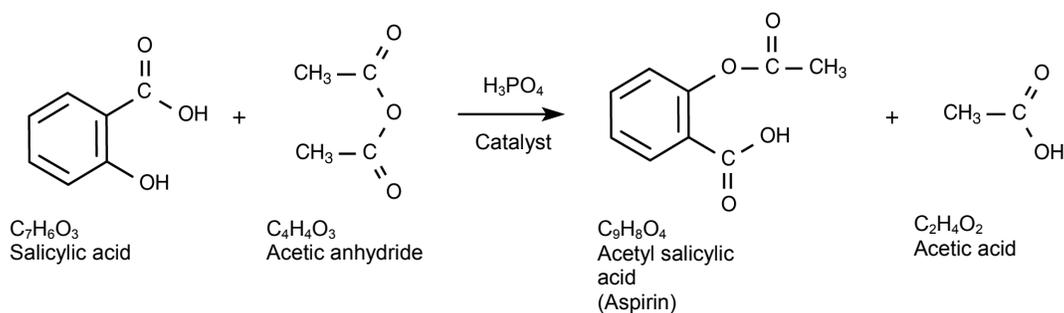
- ◆ Starting a new experiment on the data collection system ◆<sup>(1,2)</sup>
- ◆ Connecting a sensor to the data collection system ◆<sup>(2,1)</sup>
- ◆ Monitoring live data ◆<sup>(6,1)</sup>

## Background

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Aspirin is a widely used analgesic (pain killer), antipyretic (fever reducer), anti-inflammatory (inflammation fighter) and blood thinner. It was the first synthetic drug to be manufactured. Aspirin is the patented trade name for acetylsalicylic acid. It was synthesized from salicylic acid by Felix Hoffman in 1897 and patented by Friedrich Bayer & Co. in Germany in 1899. Bayer named its new product "aspirin"; the name originated from "a" for acetyl, and the root "-spir", from the Latin name *Spiraea Ulmaria*, the meadow sweet flower, from which salicylic acid had been isolated.

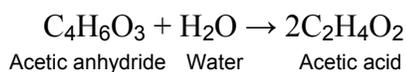
The synthesis of aspirin from salicylic acid can be accomplished by reacting it with acetic anhydride (also called "acetic acid anhydride") in the presence of a catalyst, phosphoric acid:



## Pre-Lab Activity

### Setting the stage for the activity

The synthesis of aspirin involves the reaction of salicylic acid and acetic anhydride in the presence of a catalyst, phosphoric acid,  $H_3PO_4$ . Once the aspirin is synthesized it has to be separated from the unreacted salicylic acid and purified. Aspirin is insoluble in cold water, and can be isolated by filtering the chilled reaction solution. Purification is necessary to remove any unreacted salicylic acid and acetic anhydride, as well as the acetic acid product and the phosphoric acid which is used as the catalyst. Acetic anhydride will decompose with the addition of water once the formation of aspirin is complete:



### Example calculation to try

In a test experiment, 2.5 g of salicylic acid was measured and transferred into a 125-mL Erlenmeyer flask. 5 mL of acetic anhydride and 5 drops of 85 %  $H_3PO_4$  was added drop wise under a hood. The flask was mounted in a water bath and kept at 60 °C for 20 min. 10 mL of ice cold water was added to the reaction mixture drop wise and the flask was set in an ice bath.

Once the mixture cooled, 25 mL of ice cold distilled water was added; after which the crystals started to precipitate. The precipitate was filtered out using a Büchner funnel and washed with 15 mL of ice cold distilled water. The filter paper was set aside, allowing the crystals to dry. The mass of the product was measured and found to be 2.8 g. Considering that the formula weight of salicylic acid (SA) is 138.12 g/mol, and 180.15 g/mol for aspirin (A) the theoretical yield was:

$$(2.5 \text{ g SA}) \left( \frac{180.157 \text{ g A}}{138.120 \text{ g SA}} \right) = 3.3 \text{ g A}$$

The experimental yield was:

$$\left( \frac{2.8 \text{ g}}{3.3 \text{ g}} \right) \times 100 = 85 \%$$

### 1. Can you provide some explanation for the yield being less than 100%?

The reaction may not have been 100% complete. Also, aspirin is somewhat soluble in cold water and some of the product could have remained in solution.

**2. What do you think the purpose of adding the ice cold 10 mL water drop wise was?**

The purpose of adding the 10 mL ice cold water drop wise was to remove the unreacted acetic anhydride and decrease the solubility of the product.

## Lab Preparation

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These are the materials and equipment to set up prior to the lab:

1. Place 2000 mL of deionized water on ice in a bucket or pail.
2. Place a pound of shaved ice for the class in a bucket or pail.

## Safety

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Add this important safety precaution to your normal laboratory procedures:

- ◆ Acetic anhydride and phosphoric acid are extremely caustic and must be handled under a hood. Handle them with care.

## Sequencing Challenge

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The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

1	4	2	5	3
Add a measured amount of salicylic acid to an Erlenmeyer flask and combine it with a catalyst.	Form a crystal precipitate: Add ice cold water to the mixture. After crystals are formed, filter the reaction mixture.	Start the reaction: Add acetic anhydride very carefully. Mount the flask in a hot water bath for 20 minutes.	Dry and measure the mass of the product. Calculate your yield.	Remove the unreacted acetic anhydride to decrease the solubility of the product.

## Procedure with Inquiry

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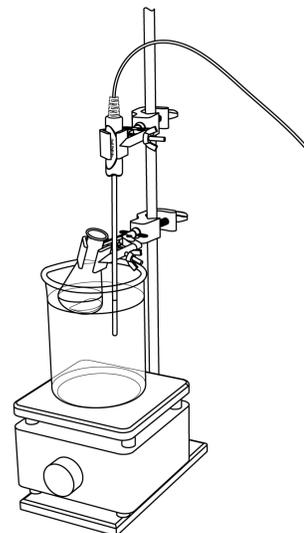
After you complete a step (or answer a question), place a check mark in the box (☐) next to that step.

**Note:** When students see the symbol "◆" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

**Set Up**

1.  Measure about 2.0 g of salicylic acid and place it into a 125-mL Erlenmeyer flask. Record in Table 1 the actual mass of salicylic acid used, to the precision of the balance.
2.  In the hood:
  - a. Add 4 mL of acetic anhydride, by drops, to the Erlenmeyer flask containing the salicylic acid.
  - b. Add 5 drops of 85% phosphoric acid to the Erlenmeyer flask.

**CAUTION:** Both acetic anhydride and phosphoric acid can cause burns. Handle them carefully.



3.  Clamp the Erlenmeyer flask in the water bath so that the reaction mixture is below the level of the water. Tilt the flask to minimize the chance of the mixture boiling over.
4.  Start a new experiment on the data collection system. ♦<sup>(1.2)</sup>
5.  Connect the stainless steel temperature sensor to the data collection system. ♦<sup>(2.1)</sup>
6.  Clamp a stainless steel temperature sensor to the ring stand and immerse the tip of the sensor in the water.

**Note:** Do not allow the temperature sensor to touch the sides or bottom of the flask.
7.  Monitor live data without recording. ♦<sup>(6.1)</sup>
8.  Use the temperature sensor to maintain the temperature of the water bath around 90 °C.

**Collect Data**

9.  Heat the mixture in the water bath for 20 minutes. Check that all solids have dissolved.
10.  Remove the reaction flask from the water bath.
11.  Obtain 50 mL of ice cold distilled water. Measure 10 mL of cold distilled water into a 10 mL graduated cylinder. Allow the flask to cool down to room temperature. Add the 10 mL of cold distilled water to the reaction flask 3 to 5 drops at a time.

**CAUTION:** The decomposition of excess acetic anhydride is exothermic. Beware of vapors and spattering.

## Lab 22a: Organic Synthesis I

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- 12.**  Why do you think you have to add water to the reaction mixture?

Water is added to remove unreacted acetic anhydride.

- 13.**  Prepare the ice bath in the 400-mL beaker. Chill the reaction mixture in the ice bath for about 30 minutes.

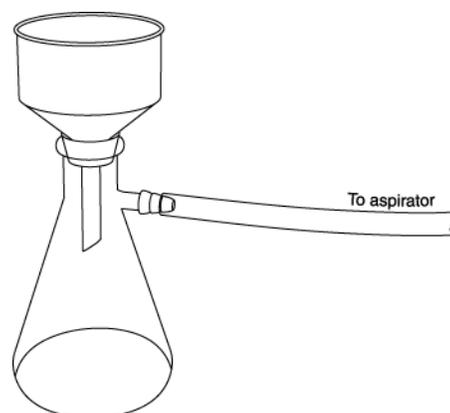
- 14.**  Add 25 mL of cold, distilled water to the reaction mixture. Aspirin crystals should begin to precipitate.

- 15.**  Why do the crystals precipitate?

The crystals precipitate because the product has little solubility in cold water.

- 16.**  While the flask is cooling in the ice bath, prepare a filter setup as illustrated.

- 17.**  Obtain the mass of a piece of filter paper and place it in the Büchner filter. Using a wash bottle, moisten the filter paper with a small amount of distilled water in order to seal it to the surface of the funnel.



- 18.**  Turn on the aspirator. Swirl the contents of the flask and pour them into the center of the filter paper. Use a rubber policeman to make sure all of the contents of the flask have been transferred to the filter paper.

- 19.**  When all of the liquid has been drawn through the filter, wash the precipitate with a measured amount of ice cold distilled water while the suction is still being applied. Do not exceed 15 mL.

- 20.**  Break the vacuum and shut off the aspirator. Carefully remove the filter paper from the Büchner filter with the forceps and set it aside to dry.

- 21.**  Obtain the mass of the filter and product after they have dried and enter this measurement in Table 1.

- 22.**  Save your product (aspirin crystals) for analysis (refer to the Organic Synthesis II activity).

- 23.**  Clean up according to the teacher's instructions.

## Data Analysis

1.  Calculate the theoretical amount of aspirin to be produced in this reaction.

$$2.000 \text{ g Salicylic Acid} \times \frac{180.157 \text{ g Aspirin}}{138.120 \text{ g Salicylic Acid}} = 2.608 \text{ g Aspirin}$$

2.  Calculate the percent yield of the synthesis and enter it in Table 1.

$$\frac{2.149 \text{ g Aspirin (experimental)}}{2.608 \text{ g Aspirin (theoretical)}} \times 100 = 82.40\% \text{ yield}$$

Table 1: Determine the efficiency of the reaction

Parameter	Data and Calculated Results
Mass of salicylic acid (g):	2.000
Mass of theoretical amount of aspirin (g):	2.608
Mass of experimental aspirin (g):	2.149
Yield of the synthesis (%):	82.40

## Analysis Questions

1. **What compound or compounds do you think your product contains?**

The product could contain unreacted salicylic acid and aspirin.

2. **What compounds from the reaction mixture would you not find in your product?**

The product cannot have acetic anhydride. The excess acetic anhydride was removed by adding water. Also, being volatile compounds, both acetic anhydride and acetic acid (the product of the hydrolysis of acetic anhydride) are liquid at room temperature but drying removes them.

3. **Propose ways of improving the yield.**

Longer reaction time would probably improve the yield. Also, lowering the temperature of the ice bath may help remove more products.

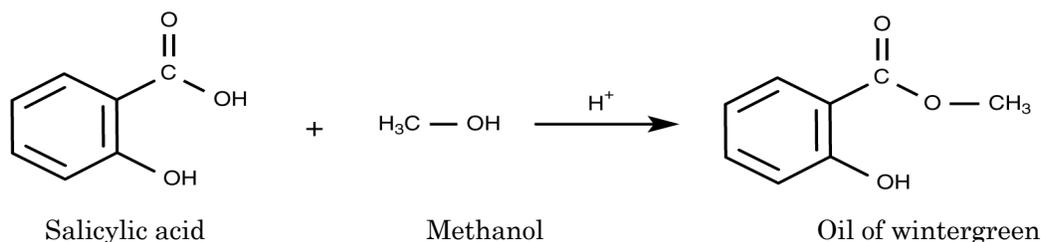
4. **Explain in your own words what is happening during the chemical reaction that produces aspirin.**

Acetic anhydride breaks up into  $\text{CH}_3\text{CO}^+$  and acetate ions ( $\text{CH}_3\text{COO}^-$ ). The  $\text{CH}_3\text{CO}^+$  ion replaces the hydrogen on the OH group in the salicylic acid. The displaced hydrogen forms an acetic acid molecule with the acetate ion.

## Synthesis Questions

Use available resources to help you answer the following questions.

**1.** A similar reaction can produce “oil of wintergreen.” Based on the procedure used to synthesize aspirin, propose a method to synthesize oil of wintergreen.



Instead of acetic anhydride one would use methanol as a solvent and as the second reactant. The rest of the procedure should be the same.

**2.** Propose a method to purify your product.

Recrystallization from ice cold water would improve the purity of the product.

## Multiple Choice Questions

Select the best answer or completion to each of the questions or incomplete statements below.

- What happens to the excess acetic anhydride in the reaction mixture?
  - There is no excess acetic anhydride.
  - The excess acetic anhydride is removed by cooling the reaction mixture in an ice cold water bath.
  - The excess acetic anhydride is removed by reacting the reaction mixture with ice cold water.
  - The excess acetic anhydride is removed by filtering the reaction mixture.
- The dry product will have:
  - Salicylic acid only
  - Aspirin only
  - Aspirin and salicylic acid
  - Acetic anhydride only
- The yield can be improved by:
  - Using more salicylic acid
  - Using more acetic anhydride
  - Allowing a longer reaction time
  - Using a different kind of acid catalyst

- 4. The filtration process will introduce an error if:**
- A. Not all of the product is collected from the filter paper**
  - B. Not all acetic anhydride is removed**
  - C. Not all salicylic acid is reacted**
  - D. Too much acid catalyst is used**

### **Extended Inquiry Suggestions**

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If time allows, have students synthesize oil of wintergreen in the same way as aspirin. Isolation of the product from the reaction mixture however, is not necessary. The product can be identified by its pungent aroma.



## Lab 22b: Organic Synthesis II—Analysis

### Objectives

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Organic Synthesis I: Students synthesize an organic compound (aspirin).

Organic Synthesis II: Students analyze the purity of the aspirin they synthesized.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Performing qualitative and quantitative analytical methods to determine the composition of their synthesized product, including melting point determination and titration.

### Time Requirement

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◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	120 minutes

### Materials and Equipment

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#### *For each student or group:*

- ◆ Data collection system
- ◆ Stainless steel temperature sensor
- ◆ pH sensor
- ◆ Drop counter with micro stir bar
- ◆ Ring stand
- ◆ Clamp, utility
- ◆ Clamp, right-angle
- ◆ Clamp, buret
- ◆ Beaker (2), 150-mL
- ◆ Beaker, 100-mL
- ◆ Beaker (2), 25-mL
- ◆ Test tubes (3), 15-mL
- ◆ Melting point capillary tube
- ◆ Buret, 50-mL
- ◆ Graduated cylinder, 100-mL
- ◆ Magnetic stirrer and stir bar
- ◆ Hot plate with magnetic stirrer and stir bar
- ◆ Mortar and pestle
- ◆ Product from Organic Synthesis I activity
- ◆ Aspirin tablet
- ◆ Ethanol, 15 mL
- ◆ 0.1 M Sodium hydroxide (NaOH), 75 mL<sup>1</sup>
- ◆ 1% Iron chloride (FeCl<sub>3</sub>), 2 mL<sup>1</sup>
- ◆ Mineral oil, 150 mL
- ◆ Buffers, pH 4 and pH 10, 10 mL
- ◆ Water, distilled, 100 mL
- ◆ Rubber band, small
- ◆ Wash bottle with deionized water

<sup>1</sup>To prepare the solutions, refer to the Lab Preparation section.

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Stoichiometry
- ◆ Titration
- ◆ Acid-base reactions

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 6: Standardizing a Solution of Sodium Hydroxide
- ◆ Lab 7: Acid–Base Titration
- ◆ Lab 22a: Organic Synthesis I—Preparation

## Using Your Data Collection System

---

Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a sensor to the data collection system ◆<sup>(2.1)</sup>
- ◆ Connecting multiple sensors to your data collection system ◆<sup>(2.2)</sup>
- ◆ Calibrating a drop counter ◆<sup>(3.4)</sup>
- ◆ Calibrating a pH sensor ◆<sup>(3.6)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>
- ◆ Changing the variable on the x-axis and y-axis of a graph ◆<sup>(7.1.9)</sup>
- ◆ Finding the coordinates of a point in a graph ◆<sup>(9.1)</sup>

## Background

The synthesis reaction may not have been 100% complete. Therefore, it is likely that there will be some unreacted salicylic acid (SA) left in the product. In this activity, students perform a qualitative and a quantitative test to determine how much of the SA has remained unreacted.

## Pre-Lab Activity

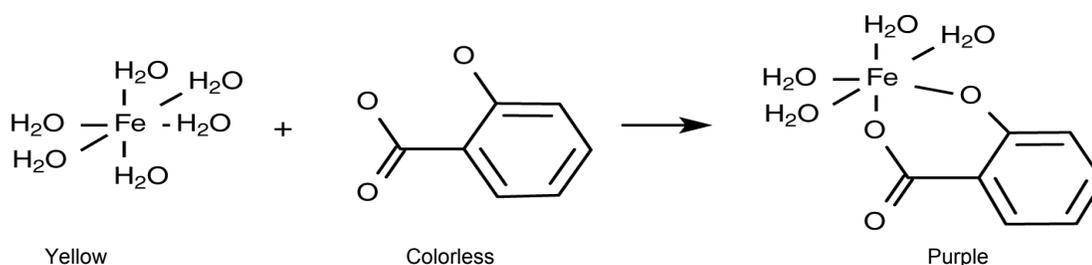
### Setting the stage for the activity

Three types of tests for unreacted salicylic acid impurities will be performed: a qualitative color test, a melting point test, and a titration test.

Students will test both their product and factory-made aspirin for the presence of salicylic acid.

#### *Iron(III) chloride test*

Salicylic acid forms a purple complex with the  $\text{Fe}(\text{H}_2\text{O})_6^{3+}$  ion (which is the common form of  $\text{Fe}^{3+}$  in water):



#### *Melting point test*

Students will measure the melting point of their product. Salicylic acid and aspirin have distinctly different melting points (see table below). If there is salicylic acid in the product, the melting point will be between the melting points of the two chemicals.

#### *Titration test*

Both salicylic acid and aspirin are acids. They do, however, have distinctly different acidity constants ( $K_a$ ) and therefore can be identified in a mixture by performing a titration with a base such as sodium hydroxide.

Properties of salicylic acid and acetylsalicylic acid

Parameter	Salicylic Acid (SA)	Acetylsalicylic Acid (ASA)
Formula	C <sub>7</sub> H <sub>6</sub> O <sub>3</sub>	C <sub>9</sub> H <sub>8</sub> O <sub>4</sub>
Molar mass (g/mol)	138.12	180.15
K <sub>a</sub>	1.10 × 10 <sup>-2</sup>	3.27 × 10 <sup>-4</sup>
pK <sub>a</sub>	1.96	3.48
Solubility (g/100 mL)	0.18	0.25
Melting point (°C)	159	138–140

**Example calculation to try**

The product of an aspirin synthesis was analyzed for salicylic acid impurities. The iron(III) chloride test showed a purple coloration, indicating a significant amount of salicylic acid impurity. The melting point was found to be 145 °C, which is slightly higher than the melting point of aspirin. This result also confirms the presence of salicylic acid.

The titration of 0.200 g of the product with 0.1000 M NaOH showed a jump in the pH at 5.80 mL and a second jump at 12.45 mL (the jumps in pH represent equivalence points). The first jump corresponds to salicylic acid, since it has the lower pK<sub>a</sub> value. The amount of titrant used to reach the pK<sub>a</sub> of SA (which is the same as the number of moles of the salicylic acid due to the 1:1 ratio between NaOH and salicylic acid) is

$$(5.80 \text{ mL}) \left( \frac{0.1000 \text{ mol}}{1000 \text{ mL}} \right) = 5.80 \times 10^{-4} \text{ mol NaOH}$$

The mass of salicylic acid in the sample then is

$$(5.80 \times 10^{-4} \text{ mol NaOH}) \left( \frac{1 \text{ mol SA}}{1 \text{ mol NaOH}} \right) \left( \frac{138.12 \text{ g SA}}{1 \text{ mol SA}} \right) = 0.0766 \text{ g SA}$$

The amount of titrant consumed to reach the pK<sub>a</sub> of aspirin was: 12.45 mL – 5.80 mL = 6.65 mL, which would account for

$$(6.65 \text{ mL NaOH}) \left( \frac{0.1000 \text{ mol NaOH}}{1000 \text{ mL}} \right) = 6.65 \times 10^{-4} \text{ mol NaOH}$$

The amount of titrant is equivalent to the amount of aspirin due to the 1:1 ratio between NaOH and aspirin. The amount of aspirin (A) in the sample is

$$(6.65 \times 10^{-4} \text{ mol NaOH}) \left( \frac{1 \text{ mol A}}{1 \text{ mol NaOH}} \right) \left( \frac{180.15 \text{ g A}}{1 \text{ mol A}} \right) = 0.120 \text{ g A}$$

The percent aspirin the product contains is, therefore

$$\left(\frac{0.120 \text{ g}}{0.200 \text{ g}}\right) \times 100 = 60.0\%$$

**1. Can you provide some explanation for the less than 100% yield?**

The reaction may not have been 100 % completed. Also, aspirin is somewhat soluble in cold water and some of the product remained in solution.

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

- 0.1 M NaOH:** Dissolve 8.00 g of NaOH in distilled water in a 2-L volumetric flask and then fill it to the mark.
- 1% FeCl<sub>3</sub>:** Dissolve 1.67 g of FeCl<sub>3</sub>·6H<sub>2</sub>O in distilled water in a 100-mL volumetric flask and then fill it to the mark.

## Safety

Follow all standard laboratory procedures.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

1	3	4	5	2
Prepare to perform three qualitative and quantitative analytical procedures to determine product purity.	Then determine the melting point of the product: is it closer to the melting point of aspirin or salicylic acid?	Set up the pH sensor and other titration apparatus. Determine exact volumes of NaOH to reach the equivalence points.	Calculate exactly how much salicylic acid and aspirin are present in the product.	Begin with the FeCl <sub>3</sub> test, which results in a purple color in the presence of salicylic acid.

## Procedure with Inquiry

---

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "◆" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Part 1 – FeCl<sub>3</sub> test

#### Set Up

1.  Put 1 mL ethanol in three clean test tubes.
2.  Put 1 to 2 drops of 1% FeCl<sub>3</sub> in each test tube.

#### Collect Data

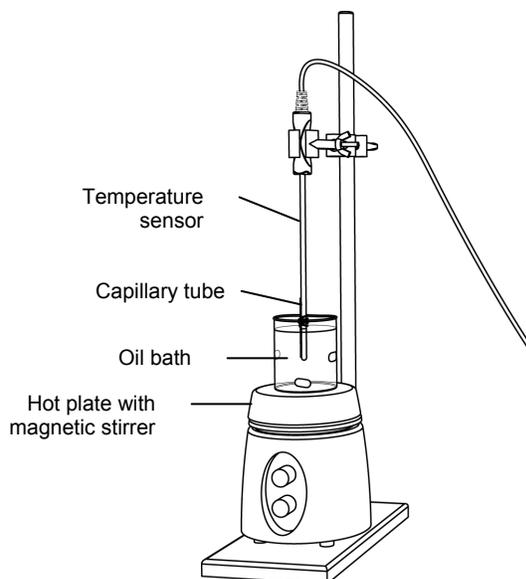
3.  Add a few crystals of factory-made aspirin to the first test tube and a few crystals of your product into the second test tube. The third test tube is your control. Label the test tubes appropriately.
4.  Record your observations in Table 2.

### Part 2 – Melting point test

#### Set Up

5.  Start a new experiment on the data collection system. ◆<sup>(1,2)</sup>
6.  Connect the stainless steel temperature sensor to the data collection system. ◆<sup>(2,1)</sup>
7.  Begin setting up the melting point detection apparatus: put the stir bar in the 100-mL beaker and fill it with mineral oil almost to the top. Put the beaker on the hot plate on the support stand.
8.  Use a mortar and pestle to crush about 20 mg of the product into a fine powder. Collect the powdered material in a pile in the center of the mortar.

9.  Push the open end of a capillary tube into the pile of crushed product. Pack it into the capillary tube by inverting the tube and tapping it lightly on the bench top. Pack about 1 cm of powder into the capillary tube.
10.  Use a small rubber band to fasten the capillary tube to the end of the stainless steel temperature sensor.
11.  Clamp the assembly to the support stand so that the end of the temperature sensor and capillary tube are immersed in the mineral oil bath without letting any oil get into the capillary tube.



### Collect Data

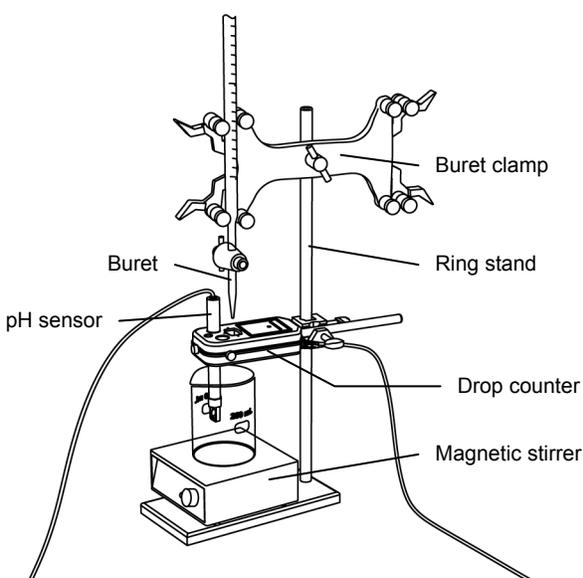
12.  Start data recording.  $\diamond^{(6.2)}$
13.  Turn on the hot plate to slowly heat the oil bath. Turn on the magnetic stirrer.
14.  Observe the material in the capillary tube until it has melted. Record the melting point in Part 2 of the Data Analysis section.

**CAUTION:** The oil bath will be extremely hot; exercise caution. Let it cool before cleaning up.

### Part 3 – Titration test

#### Set Up

15.  Start a new experiment on the data collection system.  $\diamond^{(1.2)}$
16.  Connect a pH sensor to the data collection system.  $\diamond^{(2.1)}$
17.  Calibrate the pH sensor.  $\diamond^{(3.6)}$
18.  Assemble the titration apparatus, using the steps below and the illustration as a guide.



- a. Position the magnetic stirrer on the base of the ring stand.
- b. Place a waste container on the magnetic stirrer.
- c. Use the buret clamp to attach the buret to the ring stand.
- d. Position the drop counter over the waste container and attach it to the ring stand using the right-angle clamp.
- e. Place the pH sensor through one of the slots in the drop counter.

**Note:** Do not connect the drop counter to the data collection system yet.

19.  Rinse the buret with several milliliters of the 0.1 M NaOH solution:
- a. Ensure that the stopcock is closed and rinse the inside of the buret with several milliliters of the standardized NaOH solution.
  - b. Open the stopcock on the buret and drain the rinse NaOH into the waste container.
  - c. Repeat this process two more times.

20.  Why is it necessary to rinse the buret with the NaOH solution?

If there is any residual water or contaminant in the buret, it will dilute the NaOH and change its concentration. Rinsing eliminates any such contamination.

21.  Make sure the stopcock on the buret is in the “off” position and then use a funnel to fill the buret with about 50 mL of the 0.1 M NaOH solution (titrant).
22.  Drain a small amount of the titrant through the drop counter into the waste beaker to remove any air in the tip of the buret.

23.  Why is it important to remove air from the tip of the buret?

Any air trapped in the buret tip is counted as volume of NaOH. If this happens, the amount of titrant used will be inaccurate.

24.  Practice adjusting the stopcock on the buret so that the titrant goes through the drop counter in distinguishable drops that fall at about 1 to 2 drops per second.

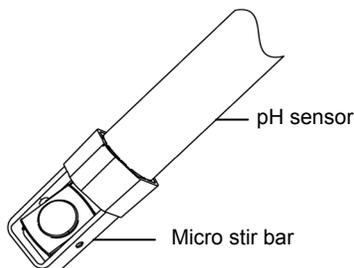
**Note:** Good control of the stopcock is important. If you accidentally open the stopcock too far and the NaOH streams out (as opposed to coming out in drops), you will have to start over.

25.  Why will it be necessary to start your titration over again if you accidentally allow the titrant to stream out of the stopcock instead of emerging by drops?

The drop counter counts distinct drops. If the drops are not sufficiently distinct from one another, the drop counter will not function properly and the fluid volume will not be accurate.

26.  Remove the waste container.

27.  Add the micro stir bar to the end of the pH sensor.



28.  Add additional 0.1 M NaOH to the buret so the solution is above the zero mark. Allow some of the NaOH solution to drip into the waste container until the bottom of the meniscus is lined up with or just below the zero mark and record the initial reading in Table 1.
29.  Measure about 0.2 g of your product to the nearest mg. Record the mass in Table 3.
30.  Transfer the sample into a 150-mL beaker.
31.  Dissolve the sample in 10 mL of ethanol.
32.  Add 100 mL of water to the solution.
33.  Place the solution on the magnetic stirrer.
34.  Turn on the magnetic stirrer at a gentle rate.
35.  Connect the drop counter to the data collection system.  $\diamond^{(2.2)}$
36.  Display pH versus Drop Count (drops) on a graph.  $\diamond^{(7.1.1)}$

### Collect Data

37.  Clean the lens of the drop counter inside the opening through which the drops are going with water and a cotton swab or tissue.
38.  Start recording data.  $\diamond^{(6.2)}$
39.  Turn the buret stopcock carefully, allowing the titrant to drip slowly (1 to 2 drops per second) into the solution.
40.  Add the titrant drop by drop until the second jump occurs and levels off. If you do not get a second jump after 20 mL of the titrant was added, stop the titration.

41.  Why is it important to go past the equivalence point?

It is necessary to go past the equivalence point in order to find the point where the slope is the steepest. The curve needs to continue past the steepest point to ensure you can tell when the slope begins to flatten.

42.  Stop recording data.  $\diamond^{(6.2)}$

43.  In Table 1, record the final drop count and the final reading of the titrant in the buret to a precision of 0.01 mL.

44.  Calculate the volume of titrant (final reading minus initial reading) and record this value in Table 1.

Table 1: Titration data

Titration Information	Measurement or Calculation
Initial reading of NaOH in the buret (to 0.01 mL)	0.00
Final reading of NaOH in the buret (to 0.01 mL)	17.00
Volume of titrant (to 0.01 mL)	17.00
Final drop count	333

45.  Calibrate the drop counter.  $\diamond^{(3.4)}$

46.  Set the horizontal axis to the calculated volume.  $\diamond^{(7.1.9)}$

47.  Find the volumes of titrant corresponding to the first and second jumps on the graph and record them in Table 3.  $\diamond^{(9.1)}$

## Data Analysis

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### Part 1 – FeCl<sub>3</sub> test

1.  Complete the tables below with your data.

Table 2: Iron(III) chloride test

Parameter	Observations
Aspirin	Clear with yellow tint
Product	Clear with purple tint
Control	Clear with yellow tint

**Part 2 – Melting point test**

2.  Enter the melting point of the product (°C): 137.7

**Part 3 – Titration test**

Table 3: Determination of product composition. Show calculations below.

Parameter	Salicylic Acid	Aspirin
Mass of product being titrated (g)	0.200	
Volume corresponding to the first equivalence point (mL)	1.58	
Volume corresponding to the second equivalence point (mL)		9.80
<b>Note:</b> Subtract the volume that was necessary for the first equivalence point.		
Amount of titrant added (mol)	$1.58 \times 10^{-4}$	$9.80 \times 10^{-4}$
Amount of titrated substance (mol)	$1.58 \times 10^{-4}$	$9.80 \times 10^{-4}$
Formula weight (g/mol)	138.12	180.15
Mass of titrated substance (g)	$2.18 \times 10^{-2}$	0.177
Composition of product (%)	10.9	88.3

3.  Calculate the number of moles of titrant added and enter these values in Table 3.

**a.** Salicylic acid

$$(1.58 \text{ mL NaOH}) \left( \frac{1 \text{ mol SA}}{1 \text{ mol NaOH}} \right) \left( \frac{0.1000 \text{ mol NaOH}}{1000 \text{ mL}} \right) = 1.58 \times 10^{-4} \text{ mol SA}$$

**b.** Aspirin

$$(9.8 \text{ mL NaOH}) \left( \frac{1 \text{ mol A}}{1 \text{ mol NaOH}} \right) \left( \frac{0.1000 \text{ mol NaOH}}{1000 \text{ mL}} \right) = 9.8 \times 10^{-4} \text{ mol A}$$

## Lab 22b: Organic Synthesis II

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4.  Calculate the number of moles of titrated substance added and enter these in Table 3.

a. Salicylic acid

$1.58 \times 10^{-4}$  moles

b. Aspirin

$9.80 \times 10^{-4}$  moles

The amount of titrated substance is equivalent to the amount of titrant due to the 1:1 ratio between NaOH and salicylic acid or aspirin.

5.  Calculate the mass of the titrated substances and enter these in Table 3.

a. Salicylic acid

$$\left(1.58 \times 10^{-4} \text{ mol SA}\right) \left(\frac{138.12 \text{ g SA}}{1 \text{ mol SA}}\right) = 0.0218 \text{ gSA}$$

b. Aspirin

$$\left(9.80 \times 10^{-4} \text{ mol A}\right) \left(\frac{180.15 \text{ g A}}{1 \text{ mol A}}\right) = 0.176 \text{ gA}$$

6.  Determine the percentage of the components in the product and enter these in Table 3.

a. Salicylic acid

$$\left(\frac{0.0218 \text{ g}}{0.200 \text{ g}}\right) \times 100 = 10.9\%$$

b. Aspirin

$$\left(\frac{0.177 \text{ g}}{0.200 \text{ g}}\right) \times 100 = 88.3\%$$

## Analysis Questions

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1. What conclusion have you drawn from the iron(III) chloride test?

Most likely students will find the test positive with their product while the blank and the industrial aspirin show negative results since the blank and the industrial aspirin will not have salicylic acid residue.

2. What conclusion can be drawn from the melting point test?

The melting point should be near the melting point of pure aspirin. The extent of deviation is related to the amount of unreacted salicylic acid.

3. What conclusion can you draw from the titration test?

The titration test may not show a reliable "jump" for salicylic acid on the titration curve, which indicates that the amount of salicylic acid in the product is small.

- 4. Combine the results of the tests and argue about the purity of your product. Consider the accuracy of the individual tests when you answer this question.**

Student should primarily rely on the fact that the titration is the most quantitative test, the melting point test is reliable, and the iron(III) chloride test is qualitative.

## Synthesis Questions

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Use available resources to help you answer the following questions.

- 1. How do you think the purity of the product can be improved?**

Recrystallization would improve the purity of the product.

- 2. What is the trade-off with increasing the purity of the product?**

Any purifying process will result in losing some of the product. Therefore, the overall yield of the synthesis will decrease.

## Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

- 1. The melting point of the product should be:**

- A. Higher than the melting point of salicylic acid
- B. Higher than the melting point of aspirin
- C. Between the melting points of aspirin and salicylic acid**
- D. Higher than salicylic acid but lower than aspirin

- 2. The lack of the first jump (around pH 4 to 5) and the presence of the second jump (around pH 6 to 7) on the titration curve indicate:**

- A. An error in the procedure
- B. The lack of salicylic acid in the product**
- C. The lack of aspirin in the product
- D. Poorly calibrated pH sensor

- 3. The purple color after adding iron(III) chloride to the product indicates:**

- A. The presence of salicylic acid**
- B. The presence of the product, aspirin
- C. The lack of salicylic acid in the product
- D. The lack of aspirin in the product

**4. The titration test will**

- A. Show the presence of both salicylic acid and aspirin**
- B. Show the presence of only salicylic acid**
- C. Show the presence of aspirin**
- D. The presence of any organic acids**

**Extended Inquiry Suggestions**

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Have students recrystallize the product from ice cold water to improve purity. Then have them repeat these tests to determine if there is any affect on the purity and yield due to the recrystallization.

## Lab 23: Determination of a Solubility Product

### Objectives

Students demonstrate a method of determining the solubility product of an ionic compound.

### Procedural Overview

Students will gain experience conducting the following procedures:

- ◆ Through titration and calculations, determine the solubility product of calcium hydroxide.

### Time Requirement

◆ Preparation time	15 minutes (plus 3 hours solution preparation)
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

#### *For each student or group:*

- ◆ Data collection system
- ◆ pH sensor
- ◆ Drop counter and micro stir bar
- ◆ Ring stand
- ◆ Clamp, buret
- ◆ Clamp, right-angle
- ◆ Beaker, 100-mL
- ◆ Beaker, 150-mL (2)
- ◆ Beaker (2), 25-mL
- ◆ Pipet, graduated or volumetric, 50-mL
- ◆ Rubber bulb
- ◆ Buret, 50-mL
- ◆ Filter flask, 250-mL
- ◆ Büchner funnel
- ◆ Pipet, transfer
- ◆ Filter paper
- ◆ Magnetic stirrer
- ◆ 0.1000 M Hydrochloric acid (HCl), 200 mL<sup>1</sup>
- ◆ Calcium hydroxide (Ca(OH)<sub>2</sub>), saturated, 200 mL<sup>2</sup>
- ◆ Buffers, pH 4 and pH 10, 10 mL
- ◆ Wash bottle with distilled water
- ◆ Parafilm<sup>®</sup> or aluminum foil
- ◆ Cotton swab or tissue

<sup>1,2</sup>To prepare the solutions, refer to the Lab Preparation section.

## **Concepts Students Should Already Know**

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Students should be familiar with the following concepts:

- ◆ Molarity
- ◆ Acid-Base reactions
- ◆ Titration
- ◆ pH
- ◆ Chemical equilibria
- ◆ Le Chatelier's principle

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 7: Acid–Base Titration
- ◆ Lab 6: Standardizing a Solution of Sodium Hydroxide
- ◆ Lab 24: Determining  $K_a$  by Half-Titration of a Weak Acid
- ◆ Lab 26: Conductometric Titration
- ◆ Lab 30: Determination of the  $K_a$  Values of Two Isomer Multi-Protic Acids

## **Using Your Data Collection System**

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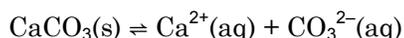
Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connect a sensor to the data collection system. ◆<sup>(2.1)</sup>
- ◆ Connecting multiple sensors to the data collection system ◆<sup>(2.2)</sup>
- ◆ Calibrating a drop counter ◆<sup>(3.4)</sup>
- ◆ Calibrating a pH sensor ◆<sup>(3.6)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>

- ◆ Changing the variable on the x-axis and y-axis of a graph ◆<sup>(7.1.9)</sup>
- ◆ Printing the graph. ◆<sup>(11.2)</sup>

## Background

The ability of a compound to dissolve in a solvent is called solubility. The solubility of an ionic compound refers to the maximum amount of substance (solute) that can dissolve in a given amount of solvent at standard temperature and pressure. The solution of an ionic compound containing the maximum amount of solute is known as a saturated solution and is in a state of equilibrium between dissolved and undissolved solute. The equation below describes the equilibrium of a saturated solution of calcium carbonate in water:



Calcium carbonate dissolves into the solution (forward reaction) and at the same time calcium carbonate precipitates from the solution (reverse reaction). When both reactions occur at the same rate, a state of equilibrium is established. The solubility product constant,  $K_{\text{sp}}$ , is a temperature-dependent constant that refers to this state. If a salt,  $\text{M}_x\text{A}_y$ , dissociates into cations  $[\text{M}^{m+}]$  and anions  $[\text{A}^{a-}]$  the expression for the solubility product will be

$$K_{\text{sp}} = [\text{M}^{m+}]^x [\text{A}^{a-}]^y$$

## Pre-Lab Activity

### Setting the stage for the activity

In this lab, you will determine the  $K_{\text{sp}}$  of calcium hydroxide. It will not be necessary to determine the concentration of both  $\text{Ca}^{2+}$  and  $\text{OH}^-$  because there is a fixed relationship between the two quantities. It will only be necessary to find the concentration of one, since the other can be easily calculated from it.

### Example calculation to try

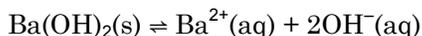
To determine the molar concentration of dissolved hydroxide ions, about 10 g of solid  $\text{Ba}(\text{OH})_2$  was placed into a 500-mL Erlenmeyer flask with about 300 mL of water. A stirring bar was placed into the solution and the flask was placed on a magnetic stirrer. The solution was stirred for 2 hours, during which time the solution became saturated with  $\text{Ba}(\text{OH})_2$ .

About 100 mL of the solution was filtered and 50.00 mL of the filtrate was pipetted into a 150-mL beaker. A stirring bar was placed into the solution and the beaker was set onto a magnetic stirrer. A pH electrode was calibrated and mounted in the solution. A 50-mL buret was filled with 0.2000 M HCl solution.

The saturated  $\text{Ba}(\text{OH})_2$  solution was titrated with the HCl solution. A sharp jump occurred after 20.50 mL (equivalence point) and the titration was continued until 25.00 mL HCl solution was added.

## Lab 23: Determination of a Solubility Product

The chemical reaction that occurred was:



The amount of HCl solution added until the equivalence point was:

$$(20.50 \text{ mL}) \left( \frac{0.2000 \text{ mol HCl}}{1000 \text{ mL}} \right) = 4.100 \times 10^{-3} \text{ mol HCl}$$

Based on the stoichiometry of the reaction between  $\text{H}_3\text{O}^{+}$  and  $\text{OH}^{-}$  ions, the same amount of  $\text{OH}^{-}$  ions were in the solution as  $\text{H}_3\text{O}^{+}$  ions were in HCl. The concentration of the  $\text{OH}^{-}$  ions is:

$$\left( \frac{4.100 \times 10^{-3} \text{ mol OH}^{-}}{5.000 \times 10^{-2} \text{ L}} \right) = 8.200 \times 10^{-2} \text{ M OH}^{-}$$

Based on the stoichiometry, the  $\text{Ba}^{2+}$  concentration is:

$$[\text{Ba}^{2+}] = \frac{[\text{OH}^{-}]}{2} = \frac{(8.200 \times 10^{-2} \text{ M})}{2} = 4.100 \times 10^{-2} \text{ M}$$

Substituting the concentrations into the expression for  $K_{\text{sp}}$  yields:

$$K_{\text{sp}} = [\text{Ba}^{2+}][\text{OH}^{-}]^2 = (4.100 \times 10^{-2})(8.200 \times 10^{-2})^2 = 2.760 \times 10^{-4}$$

Experimental data and calculated solubility product for  $\text{Ba(OH)}_2$

Parameter	Value
Volume of 0.2000 M HCl until equivalence point (mL)	20.50
Amount of HCl added until the equivalence point (mol)	$4.100 \times 10^{-3}$
Amount of $\text{OH}^{-}$ ions that were present in the solution (mol)	$4.100 \times 10^{-3}$
Concentration of $\text{OH}^{-}$ ions in the solution (M)	$8.200 \times 10^{-2}$
Concentration of $\text{Ba}^{2+}$ ions in the solution (M)	$4.100 \times 10^{-2}$
Calculated solubility product	$2.760 \times 10^{-4}$
Known value	$2.55 \times 10^{-4}$

**1. Do you think it matters how much solid  $\text{Ba(OH)}_2$  is used to make the saturated solution? Explain your answer!**

As long as there is a sufficient amount to leave some undissolved  $\text{Ba(OH)}_2$  after the solution is made, it doesn't matter.

**2. List three additional, chemically different solutions, and their concentrations, that would be appropriate to determine the  $\text{OH}^-$  concentration in the saturated  $\text{Ba}(\text{OH})_2$  solution.**

Any strong acid solution would be appropriate: acids with one hydrogen ion,  $\text{H}^+$ , such as  $\text{HNO}_3$  would have to have about the same concentration (0.2 M), acids with two hydrogen ions, such as  $\text{H}_2\text{SO}_4$ , would need half of the concentration (0.1 M), acids with three hydrogen ions, such as  $\text{H}_3\text{PO}_4$  would have to have a third of the concentration (0.07 M).

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

- 0.1000 M HCl:** Carefully add 8.4 mL of 36% (concentrated) HCl to some distilled water in a 1-L volumetric flask. Fill the flask to the mark.

**Note:** This method prepares a solution with an approximate concentration. Standardization is necessary to determine the exact concentration of the solution using a standardized NaOH solution or any appropriate primary standard.

- Saturated  $\text{Ca}(\text{OH})_2$ :** Place about 2 g of  $\text{Ca}(\text{OH})_2$  in a 1-L Erlenmeyer flask and fill it to about the 1-L mark with distilled water. Place a stirring bar in the flask and place the flask on a magnetic stirrer. Stir for about three hours to saturate the solution.

## Safety

Add these important safety precautions to your normal laboratory procedures:

- ◆ In case of contact with skin, HCl should be washed off with large amounts water.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

5	2	4	1	3
Calculate the solubility product.	Before preparing the analyte, fill the buret with the titrant. Practice using the drop counter, then set the meniscus to the zero mark.	Perform the titration until the pH level flattens after reaching the equivalence point. Record the volume and repeat 2 times.	Set up the titration apparatus with a pH sensor and drop counter. Calibrate the pH sensor.	Filter the saturated $\text{Ca}(\text{OH})_2$ solution and put a known volume of it (to 0.01 mL accuracy) into a beaker.

## Procedure with Inquiry

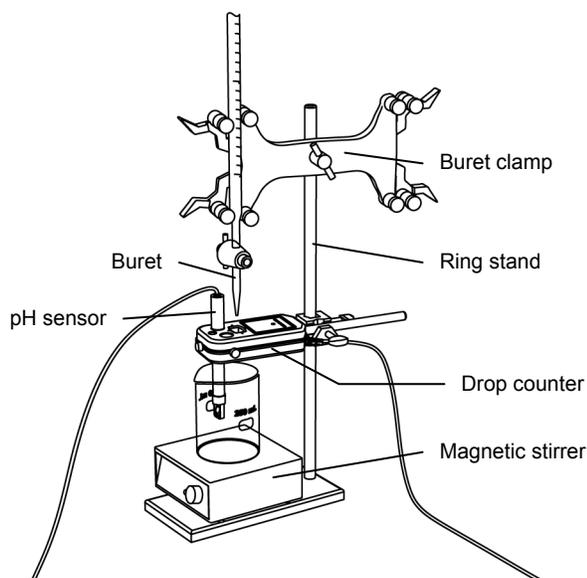
After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "◆" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

1.  Start a new experiment on the data collection system. ◆<sup>(1.2)</sup>
2.  Connect a pH sensor to the data collection system. ◆<sup>(2.1)</sup>
3.  Calibrate the pH sensor. ◆<sup>(3.6)</sup>
4.  Assemble the titration apparatus, using the steps below and the illustration as a guide.

- a. Position the magnetic stirrer on the base of the ring stand.
- b. Place a waste container on the magnetic stirrer.
- c. Use the buret clamp to attach the buret to the ring stand.
- d. Position the drop counter over the waste container and attach it to the ring stand using the right-angle clamp.
- e. Place the pH sensor through one of the slots in the drop counter.



**Note:** Do not connect the drop counter to the data collection system yet.

5.  Rinse the buret with several milliliters of the 0.1 M HCl solution:
  - a. Ensure that the stopcock is closed and rinse the inside of the buret with several milliliters of the standardized HCl solution.
  - b. Open the stopcock on the buret and drain the rinse HCl into the waste container.
  - c. Repeat this process two more times.

6.  Why is it necessary to rinse the buret with the HCl solution?

If there is any residual water or contaminant in the buret, it will dilute the HCl and change its concentration. Rinsing eliminates any such contamination.

7.  Make sure the stopcock on the buret is in the “off” position, and then use a 100-mL beaker to fill the buret with about 50 mL of the 0.1000 M HCl solution (titrant).
8.  Drain a small amount of the titrant through the drop counter into the waste beaker to remove any air in the tip of the buret.
9.  Why is it important to remove air from the tip of the buret?

Any air trapped in the buret tip is counted as volume of HCl. If this happens, the final amount of titrant used will be inaccurate.

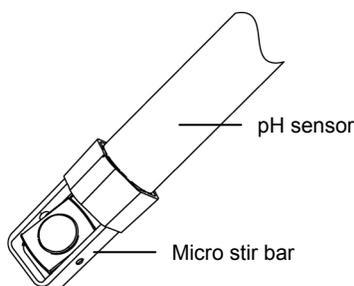
10.  Practice adjusting the stopcock on the buret so that the titrant goes through the drop counter in distinguishable drops that fall at about 1 to 2 drops per second.

**Note:** Good control of the stopcock is important. If you accidentally open the stopcock too far and the HCl flows out (as opposed to coming out in drops), you will have to start over.

11.  Why will it be necessary to start your titration over again if you accidentally allow the titrant to stream out of the stopcock instead of emerging by drops?

The drop counter counts distinct drops. If the drops are not sufficiently distinct from one another, the drop counter will not function properly and the fluid volume will not be accurate.

12.  Add the micro stir bar to the end of the pH sensor.



13.  Why is it necessary to stir the solution during a titration?

Stirring thoroughly mixes the ions in the solution so that the recorded pH is for the entire solution.

14.  Add additional HCl solution to the buret so it is above the zero mark. Allow some of the solution to drip into the waste container until the bottom of the meniscus is lined up with or just below the zero mark and record the starting volume in Table 1.
15.  Remove the waste container.
16.  Cover the beaker containing the remaining titrant solution with a piece of Parafilm<sup>®</sup> or aluminum foil.

## Lab 23: Determination of a Solubility Product

---

Perform the titration of saturated  $\text{Ca}(\text{OH})_2$  solution three times, following the steps below.

17.  Analyte preparation:

- a. Filter about 80 mL of the saturated  $\text{Ca}(\text{OH})_2$  solution through a Büchner funnel (with the filter paper covering the holes) connected to a 250-mL filtering flask and an aspirator pump.
- b. Why do you think the filtration is necessary?

Filtration is necessary to remove the solid particles which, if left in the solution, would react with the titrant and introduce a significant error.

- c. How do you know if the solution is saturated?

If the solution was allowed to equilibrate with solid  $\text{Ca}(\text{OH})_2$ , then it became saturated.

- d. Pipet 50.00 mL of the filtered solution into a clean, 150-mL beaker and place it on the magnetic stirrer. Add enough water to the solution to ensure the bulb of the pH sensor is fully submerged.
- e. Propose a way to precisely measure 50.00 mL of solution.

For the most precise measurement, a 50-mL volumetric pipet should be used. A 50-mL graduated cylinder can provide sufficient accuracy as well.

18.  Turn on the magnetic stirrer at a slow and steady rate.

19.  Connect the drop counter to the data collection system.  $\diamond^{(2.2)}$

20.  Display the pH on the y-axis of a graph and Drop Count on the x-axis.  $\diamond^{(7.1.1)}$

### Collect Data

21.  Clean the lens of the drop counter inside the opening through which the drops are going with water and a cotton swab or tissue.

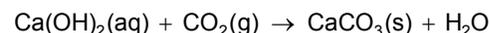
22.  Start recording data.  $\diamond^{(6.2)}$

24.  Turn the buret stopcock carefully, allowing the titrant to drip slowly (1 to 2 drops per second) into the solution.

**Note:** Do not allow the titrated solution to be exposed to air for an extended period of time.

25.  Why do you think prolonged exposure of the titrated solution would introduce an error? (Hint: What can the solution absorb from the air and what chemical reaction can occur as a result?)

Carbon dioxide can be absorbed and turn some of the  $\text{Ca}(\text{OH})_2$  to  $\text{CaCO}_3$ :



**26.**  Do you expect a pH jump or a pH drop at the equivalence point? Explain your answer.

The pH initially was high, since the solution was alkaline. After the equivalence point, when HCl is in excess, the pH should be low. Therefore, at the equivalence point, a pH drop is expected.

**27.**  Continue the titration past the equivalence point until the pH curve flattens.

**28.**  Why is it important to go past the equivalence point?

It is necessary to go past the equivalence point in order to find the point where the slope is the steepest. The curve needs to continue past the steepest point to ensure you can tell when the slope begins to flatten.

**29.**  Stop recording data.  $\diamond^{(6.2)}$

**30.**  In Table 1, record the final drop count and the final volume of the titrant in the buret to a precision of 0.01 mL.

**31.**  Calculate the volume of titrant (final volume minus initial volume) and record this value in Table 1.

Table 1: Titration data

Titration Information	Trial 1	Trial 2	Trial 3
Initial reading of HCl on the buret (mL)	4.00	0.00	0.00
Final reading of HCl on the buret (mL)	29.81	25.72	25.64
Volume of titrant (mL)	25.81	25.72	25.64
Final drop counts	549	558	561

**32.**  Calibrate the drop counter.  $\diamond^{(3.4)}$

**33.**  Set the horizontal axis to the calculated volume.  $\diamond^{(7.1.9)}$

**34.**  In Table 2, record the volume of titrant used to reach the equivalence point.

**35.**  Print the graph.  $\diamond^{(11.2)}$

**36.**  Remove the beaker and dispose of its contents according to the teacher's instructions.

**37.**  Rinse the beaker with distilled water.

## Lab 23: Determination of a Solubility Product

---

- 38.**  Refill the buret over the zero mark with the HCl solutions.
- Fill the buret over the zero mark and allow some of the HCl solution to drip into a waste container until the bottom of the meniscus is lined up with the zero mark or just below.
  - Record the initial reading in Table 1.
- 39.**  Repeat the procedure two more times, beginning with the analyte preparation.

### Data Analysis

---

- 1.**  Calculate the amount of HCl added to reach the equivalence point for each run and enter your answers in Table 2.

For Trial 1:

$$(25.81 \text{ mL titrant}) \left( \frac{0.09600 \text{ mol HCl}^*}{1000 \text{ mL}} \right) = 2.478 \times 10^{-3} \text{ mol HCl}$$

\* A standardized HCl solution with 0.09600 M concentration was used for the test experiments.

- 2.**  How can you determine the amount of  $\text{OH}^-$  ions present in the saturated solution? Enter the number of  $\text{OH}^-$  ions for each run in Table 2.

Based on the stoichiometry of the reaction between  $\text{H}^+$  and  $\text{OH}^-$  ions, the same number of  $\text{OH}^-$  ions was in the  $\text{Ca}(\text{OH})_2$  solution as  $\text{H}_3\text{O}^+$  ions were in HCl.

- 3.**  Calculate the concentration of  $\text{OH}^-$  ions in the solution for each run and enter your answers in Table 2.

For Trial 1:

$$[\text{OH}^-] = \left( \frac{2.478 \times 10^{-3} \text{ mol}}{0.05000 \text{ L}} \right) = 4.956 \times 10^{-2} \text{ M OH}^-$$

- 4.**  Calculate the concentration of  $\text{Ca}^{2+}$  ions in the solution for each run and enter your answers in Table 2.

For Trial 1:

$$[\text{Ca}^{2+}] = \frac{[\text{OH}^-]}{2} = \frac{(4.956 \times 10^{-2} \text{ M})}{2} = 2.478 \times 10^{-2} \text{ M}$$

- 5.**  Calculate the solubility product of calcium hydroxide.

$$K_{\text{sp}} = [\text{Ca}^{2+}][\text{OH}^-]^2 = (2.478 \times 10^{-2})(4.956 \times 10^{-2})^2 = 6.080 \times 10^{-5}$$

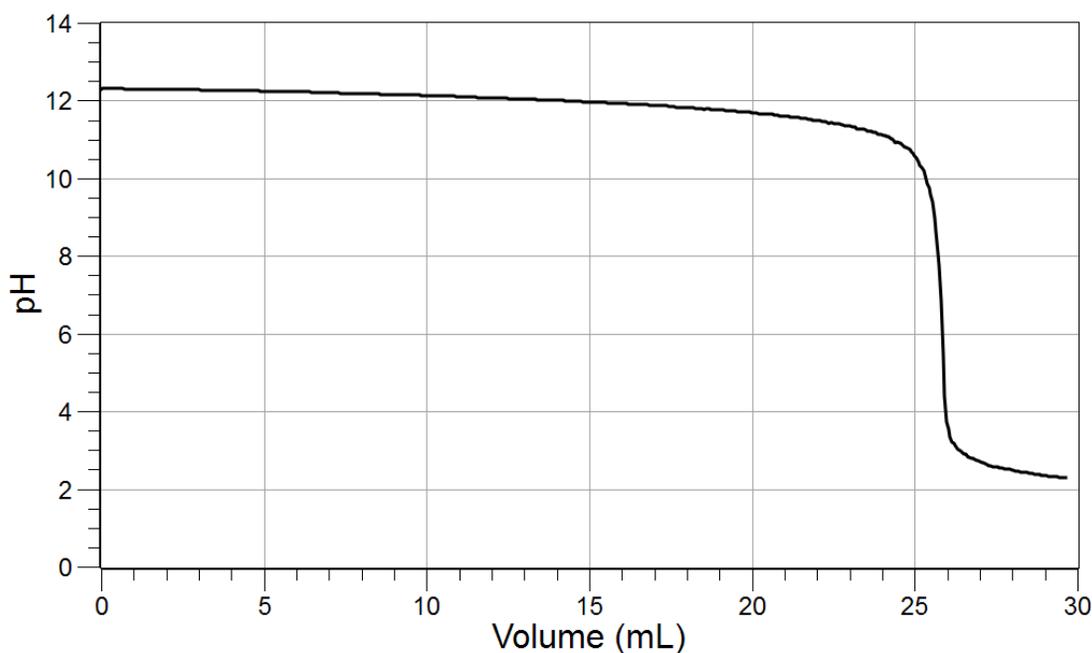
Table 2: Determination of the solubility product of  $\text{Ca}(\text{OH})_2$ 

Parameter	Trial 1	Trial 2	Trial 3
Volume of 0.1 M HCl to reach the equivalence point (mL)	25.81	25.72	25.64
Amount of HCl added to reach the equivalence point (mol)	$2.478 \times 10^{-3}$	$2.470 \times 10^{-3}$	$2.461 \times 10^{-3}$
Amount of $\text{OH}^-$ ions present in the saturated solution (mol)	$2.478 \times 10^{-3}$	$2.470 \times 10^{-3}$	$2.461 \times 10^{-3}$
Concentration of $\text{OH}^-$ ions in the solution (M)	0.04956	0.04938	0.04923
Concentration of $\text{Ca}^{2+}$ ions in the solution (M)	0.02478	0.02469	0.02461
Solubility product	$6.08 \times 10^{-5}$	$6.02 \times 10^{-5}$	$5.96 \times 10^{-5}$
Average value of solubility product	$6.02 \times 10^{-5}$		
Known value of the solubility product	$5.02 \times 10^{-6}$		

6.  How different is your calculated solubility product from the published value? What accounts for the difference?

Significantly higher solubility product than the known value can be attributed to imperfect filtration which resulted in the presence of  $\text{Ca}(\text{OH})_2$  particles. These particles yield higher acid consumption which in turns will cause higher  $\text{OH}^-$  and  $\text{Ca}^{2+}$  concentrations. If the solubility appears to be lower, the solution may not have been saturated.

7.  Sketch or paste your titration curve below.



## Analysis Questions

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**1. How would the experimental value of the solubility product change if the solution was not saturated?**

Lower concentrations would yield a lower solubility product.

**2. Sometimes in the filtered solution white precipitate appears. Propose a possible explanation. (Hint: The filtering flask is connected to a vacuum which may promote faster evaporation of water from the filtered solution.)**

Since the amount of solvent would decrease, the saturated solution would become super-saturated. The excess  $\text{Ca}(\text{OH})_2$  would precipitate out.

**3. Does the filtered precipitate or the solution that is captured by the filter paper have any effect on your results? If so, what?**

No, it does not. We measure the volume of the saturated solution after the filtration and not before.

**4. Prolonged exposure of the saturated solution to air may result in the appearance of a white precipitate other than calcium hydroxide. What could it be? What reaction is taking place? (Hint: What gases are in the air that can initiate a chemical reaction in the solution?)**

$\text{CO}_2$  can dissolve in the solution and produce calcium carbonate,  $\text{CaCO}_3$ , which is less soluble than  $\text{Ca}(\text{OH})_2$  and therefore will precipitate:



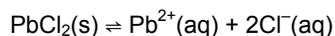
## Synthesis Questions

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Use available resources to help you answer the following questions.

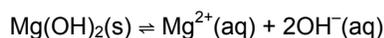
**1. The solubility product of lead chloride,  $\text{PbCl}_2$ , is rather large ( $K_{\text{sp}} = 1 \times 10^{-5}$ ). Propose a method to decrease the solubility of lead ions in a saturated  $\text{PbCl}_2$  solution. (Hint: Use the Le Chatelier principle.)**

Since the saturation process is an equilibrium process, shifting the equilibrium backwards will decrease the lead concentration. According to Le Chatelier's principle, increasing the concentration of the products will shift the equilibrium back. Increasing the  $\text{Cl}^{-}$  ion concentration will provide the desired effect. Therefore, adding  $\text{Cl}^{-}$  from a sodium chloride solution will reduce the lead ion concentration:



**2.  $\text{Mg}(\text{OH})_2$  has a solubility product,  $K_{\text{sp}}$ , of  $5.6 \times 10^{-12}$ . How would you change the pH to help the dissolution of  $\text{Mg}(\text{OH})_2$ ? (Hint: Use the Le Chatelier principle.)**

Lowering the pH would remove some of the  $\text{OH}^{-}$  ions which, in turn, would shift the equilibrium to the right, allowing more  $\text{Mg}(\text{OH})_2$  to dissolve.



**3. Would you have been able to measure the solubility constant for  $\text{Ca(OH)}_2$  if the saturated  $\text{Ca(OH)}_2$  solution was made in a 0.01 M NaOH solution instead of water?**

No. The HCl titrant solution simply measures the  $\text{OH}^-$  concentration. The  $\text{OH}^-$  concentration would no longer be related to the  $\text{Ca}^{2+}$  concentration through stoichiometry if the  $\text{Ca(OH)}_2$  was made in a NaOH solution because  $\text{OH}^-$  ions would come from two sources. We would have no way of measuring or calculating the  $\text{Ca}^{2+}$  concentration, which is necessary for the computation of  $K_{\text{sp}}$ .

**Multiple Choice Questions**

Select the best answer or completion to each of the questions or incomplete statements below.

**1. Solubility of  $\text{Ca}^{2+}$  and  $\text{OH}^-$  ions in a saturated  $\text{Ca(OH)}_2$  solution**

- A. depends on the volume of water that was used to make the solution.
- B. depends on the amount of solid  $\text{Ca(OH)}_2$  that was used to make the solution.
- C. does not depend on the amount of solid  $\text{Ca(OH)}_2$  or the amount of water that was used as long as there was enough solid to make a saturated solution.**
- D. depends on both the amount of solid  $\text{Ca(OH)}_2$  and the amount of water that was used.

**2. The filtration process will introduce an error if**

- A. any of the solution is absorbed by the filter paper.
- B. any of the solid  $\text{Ca(OH)}_2$  is captured on the paper.
- C. the paper is damaged and allows some solid  $\text{Ca(OH)}_2$  particles to go through.**
- D. the room temperature is too low.

**3. Adding NaOH solution to a saturated  $\text{Ca(OH)}_2$  solution will**

- A. have no effect on the solubility of  $\text{Ca(OH)}_2$
- B. result in a lower solubility of  $\text{Ca(OH)}_2$**
- C. result in a higher solubility of  $\text{Ca(OH)}_2$
- D. result in precipitation of NaOH

**4. How would the obtained value for the solubility constant change if you used 100.00 mL of the saturated solution instead of 50.00 mL?**

- A. The value of the solubility constant would double.
- B. The value of the solubility constant would be half of what we obtained with 50.00 mL.
- C. The value of the solubility constant would be the same.**
- D. The value of the solubility constant would be the same if the concentration of the titrant, HCl solution, was also doubled.

### **Extended Inquiry Suggestions**

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The solubility product of  $\text{CaSO}_4$  ( $K_{\text{sp}} = 5 \times 10^{-5}$ ) can be determined using conductivity titration with  $\text{BaCl}_2$  solution. Since the solubility product of  $\text{BaSO}_4$  ( $K_{\text{sp}} = 1.08 \times 10^{-10}$ ) is much less than the solubility product of  $\text{CaSO}_4$ , the excess  $\text{SO}_4^{2-}$  ions will be removed by  $\text{Ba}^{2+}$  ions. The process can be monitored with a conductivity sensor (see Conductometric Titration).

## Lab 24: Determining $K_a$ by Half-Titration of a Weak Acid

### Objectives

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Students determine the equilibrium constant for the ionization of a weak acid ( $K_a$ ) to ascertain the identity of the acid.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Determining the  $K_a$  of a weak acid by measuring the pH of a solution titrated halfway to the equivalence point
- ◆ Using a pH sensor
- ◆ Performing a titration

### Time Requirement

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◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

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#### *For each student or group:*

- |                          |  |
|--------------------------|--|
| ◆ Data collection system | ◆ Graduated cylinder, 100-mL                         |
| ◆ pH sensor              | ◆ Funnel   |
| ◆ Drop counter           | ◆ Magnetic stirrer and stir bar                      |
| ◆ Ring stand             | ◆ 0.20 M Sodium hydroxide (NaOH), 75 mL <sup>1</sup> |
| ◆ Clamp, right-angle     | ◆ Unknown weak acid solution, 50 mL <sup>2</sup>     |
| ◆ Clamp, buret           | ◆ Buffer solutions, pH 4 and pH 10, 10 mL            |
| ◆ Beaker (2), 100-mL     | ◆ Cotton swab or tissue                              |
| ◆ Buret, 50-mL           |  |

<sup>1</sup>To prepare 0.20 M NaOH, refer to the Lab Preparation section.

<sup>2</sup>Use 0.1 M acetic acid as the unknown weak acid for this activity. To prepare this solution, refer to the Lab Preparation section.

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Acids and bases
- ◆ Electrolytes
- ◆ Le Chatelier's Principle
- ◆ Molarity

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 6: Standardizing a Solution of Sodium Hydroxide
- ◆ Lab 7: Acid–Base Titration
- ◆ Lab 30: Determination of the  $K_a$  Values of Two Isomer Multi-Protic Acids

## Using Your Data Collection System

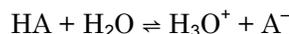
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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a sensor to the data collection system. ◆<sup>(2.1)</sup>
- ◆ Connecting multiple sensors to the data collection system ◆<sup>(2.2)</sup>
- ◆ Calibrating a drop counter ◆<sup>(3.4)</sup>
- ◆ Calibrating a pH sensor ◆<sup>(3.6)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>
- ◆ Changing the variable on the x-axis and y-axis of a graph ◆<sup>(7.1.9)</sup>
- ◆ Finding the slope at a point on a data plot. ◆<sup>(9.3)</sup>

## Background

$K_a$  is the symbol for the equilibrium constant for the ionization of an acid. The following equation describes the ionization of an acid:

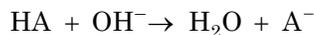


An equilibrium exists, and an acid dissociation constant can be written:

$$K_a = \frac{[\text{H}_3\text{O}^+][\text{A}^-]}{[\text{HA}]} \quad (1)$$

The value of  $K_a$  is an indication of the extent to which an acid dissociates. Strong acids dissociate nearly completely. Weak acids reach equilibrium, where the fraction that has dissociated becomes a constant at a given temperature. The numerical value of the equilibrium constant is unique to the acid and can be used to identify an unknown acid.

Before the titration has begun, the initial pH of the solution is controlled by the auto-dissociation of the acid. At this point, the concentration of  $\text{H}_3\text{O}^+$  and  $\text{A}^-$  are very small compared to the concentration of HA. When the basic titrant solution is added, it is assumed that the  $\text{OH}^-$  ions react completely with the weak acid, HA, to form water and the conjugate base,  $\text{A}^-$ :



The resulting solution now has a smaller amount of HA and a larger amount of  $\text{A}^-$ . Due to stoichiometry, the increase in  $\text{A}^-$  is the same magnitude as the decrease in HA. Solutions that contain a weak acid and also contain the corresponding conjugate base are called buffers.

When the number of moles of added base is equal to the original number of moles of HA, the equivalence point has been reached. A titration curve, in which pH is plotted versus volume of titrant added, can be used to quickly determine the  $K_a$  of the acid.

The acid dissociation constant equation (Equation 1) can be revised to form an expression used to calculate the pH of mixtures of weak acids and their salts:

$$K_a = \frac{[\text{H}_3\text{O}^+][\text{A}^-]}{[\text{HA}]}$$

Taking logarithms,

$$\log K_a = \log[\text{H}_3\text{O}^+] + \log \frac{[\text{A}^-]}{[\text{HA}]}$$

Multiplying by  $-1$ ,

$$-\log K_a = -\log[\text{H}_3\text{O}^+] - \log \frac{[\text{A}^-]}{[\text{HA}]}$$

Substituting  $\text{p}K_a$  for  $-\log K_a$  and  $\text{pH}$  for  $-\log[\text{H}_3\text{O}^+]$ ,

$$\text{p}K_a = \text{pH} - \log \frac{[\text{A}^-]}{[\text{HA}]}$$

## Lab 24: Determining $K_a$ by Half-Titration of a Weak Acid

---

Reordering,

$$\text{pH} = \text{p}K_a + \log \frac{[\text{A}^-]}{[\text{HA}]} \quad (2)$$

Equation 2 is known as the Henderson-Hasselback equation which gives the pH of buffer solutions. Notice that when enough base has been added to reach the point that is halfway between the starting point and the equivalence point, the molarity of  $\text{A}^-$  and HA will be equal. Their ratio will be 1 and since  $\log 1 = 0$ , the pH will be equal to the  $\text{p}K_a$ . The resulting  $K_a$  can be used to determine the identity of the acid.

The acid dissociation constants for several weak acids are shown in Table 1.

Table 1: Acid dissociation constants for 5 weak acids

Name	Formula	$K_a$	$\text{p}K_a$
Acetic acid	$\text{HC}_2\text{H}_3\text{O}_2$	$1.8 \times 10^{-5}$	4.7
Benzoic acid	$\text{HC}_7\text{H}_5\text{O}_2$	$6.4 \times 10^{-5}$	4.2
Formic acid	$\text{HCHO}_2$	$1.8 \times 10^{-4}$	3.7
Nitrous acid	$\text{HNO}_2$	$4.6 \times 10^{-4}$	3.4
Hypochlorous acid	$\text{HOCl}$	$3.5 \times 10^{-8}$	7.5

### Pre-Lab Activity

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#### Setting the stage for the activity

In this lab you will use a method of titration that will bring the analyte halfway to the equivalence point. Halfway to the equivalence point, half of the acid molecules are converted to  $\text{A}^-$ , therefore, for a monoprotic acid

$$[\text{HA}] = [\text{A}^-]$$

In the expression of  $K_a$  (Equation 2),  $[\text{A}^-]$  and  $[\text{HA}]$  cancel out, giving

$$K_a = [\text{H}_3\text{O}^+]$$

$$-\log K_a = -\log [\text{H}_3\text{O}^+]$$

$$\text{p}K_a = \text{pH}$$

The pH of the analyte at this point is equal to the  $\text{p}K_a$ . You will convert this value to  $K_a$  and compare this  $K_a$  value to a table of known  $K_a$  values in order to identify the unknown acid.

You will perform the titration and determine the equivalence point; that is, how much of the titrant is necessary to react with all of the acid in the solution. The pH of the half-titration point is the pH of the solution after adding half the amount of titrant needed to reach the equivalence point.

**Example calculation for students to try**

A sample of 10.00 mL of dilute HNO<sub>2</sub> solution was titrated with 0.1 M NaOH solution. The equivalence point was reached after 10.10 mL. The half-titration point, therefore, was at 5.05 mL. The pH that corresponded to that volume of titrant was 3.34, so the value of  $K_a$  is

$$\begin{aligned} \text{pH} &= 3.34 \\ \text{p}K_a &= 3.34 \\ -\log K_a &= 3.34 \\ K_a &= 4.6 \times 10^{-4} \end{aligned}$$

The published value for the  $K_a$  of nitrous acid is  $4.6 \times 10^{-4}$ .

**1. Would adding NaOH solution to the HA solution increase or decrease the pH of the solution?**

The pH would increase.

**2. Use the Henderson-Hasselbach equation to show that the pH of the solution at the half-titration point is equivalent to the  $\text{p}K_a$  value of the weak acid.**

$$\text{pH} = \text{p}K_a + \log \frac{[\text{A}^-]}{[\text{HA}]}$$

At the half-titration point

$$[\text{A}^-] = [\text{HA}]; \frac{[\text{A}^-]}{[\text{HA}]} = 1; \log \frac{[\text{A}^-]}{[\text{HA}]} = 0$$

Therefore, at the half-titration point

$$\text{pH} = \text{p}K_a$$

**Lab Preparation**

These are the materials and equipment to set up prior to the lab:

- 0.20 M Sodium Hydroxide:** Dissolve 8.00 g of NaOH in some distilled water in a 1-L volumetric flask and fill it to the mark.
- 0.1 M Acetic Acid:** Combine 11.2 mL of glacial acetic acid with some distilled water in a 2-L volumetric flask and fill it to the mark.

**Safety**

Follow all standard laboratory procedures.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

2	4	5	3	1
Then put an accurately measured quantity (50.00 mL) of the unknown solution into a beaker.	Perform the titration and record the half-titration volume.	Calculate the $pK_a$ value for the unknown solution and determine its identity.	Fill a 50-mL buret with the titrant: 0.20 M NaOH, and set the meniscus to the zero mark or right below and record the initial volume.	Set up the titration apparatus with a pH sensor and drop counter. Calibrate the pH sensor.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

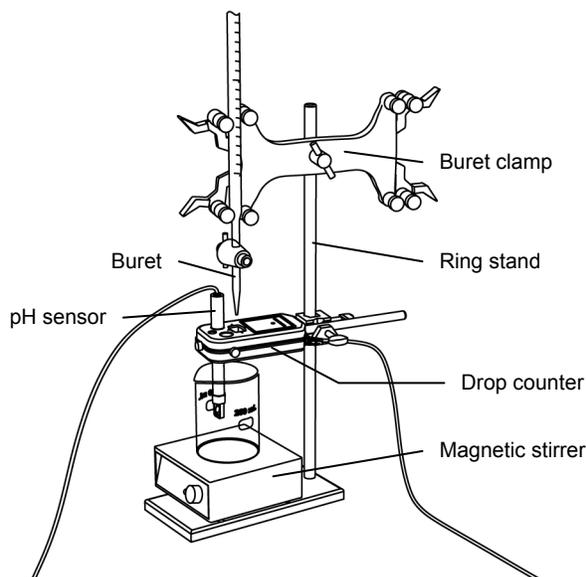
**Note:** When students see the symbol "♦" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

- Start a new experiment on the data collection system. ♦<sup>(1,2)</sup>
- Connect a pH sensor to the data collection system. ♦<sup>(2,1)</sup>
- Calibrate the pH sensor. ♦<sup>(3,6)</sup>

4.  Assemble the titration apparatus, using the steps below and the illustration as a guide.

- a. Position the magnetic stirrer on the base of the ring stand.
- b. Place a waste container on the magnetic stirrer.
- c. Use the buret clamp to attach the buret to the ring stand.
- d. Position the drop counter over the waste container and attach it to the ring stand using the right-angle clamp.



- e. Place the pH sensor through one of the slots in the drop counter.

**Note:** Do not connect the drop counter to the data collection system yet.

5.  Rinse the buret with several milliliters of the 0.20 M NaOH solution:
- a. Ensure that the stopcock is closed and rinse the inside of the buret with several milliliters of the standardized NaOH solution.
  - b. Open the stopcock on the buret and drain the rinse NaOH into the waste container.
  - c. Repeat this process two more times.

6.  Why is it necessary to rinse the buret with the NaOH solution?

If there is any residual water or contaminant in the buret, it will dilute the NaOH and change its concentration. Rinsing eliminates any such contamination.

7.  Make sure the stopcock on the buret is in the “off” position and then use a funnel to fill the buret with about 50 mL of the 0.20 M NaOH solution (titrant).

8.  Drain a small amount of the titrant through the drop counter into the waste beaker to remove any air in the tip of the buret.

9.  Why is it important to remove air from the tip of the buret?

Any air trapped in the buret tip is counted as volume of NaOH. If this happens, the amount of titrant used will be inaccurate.

## Lab 24: Determining $K_a$ by Half-Titration of a Weak Acid

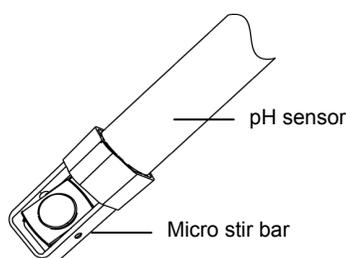
10.  Practice adjusting the stopcock on the buret so that the titrant goes through the drop counter in distinguishable drops that fall at about 1 to 2 drops per second.

**Note:** Good control of the stopcock is important. If you accidentally open the stopcock too far and the NaOH streams out (as opposed to coming out in drops), you will have to start over.

11.  Why will it be necessary to start your titration over again if you accidentally allow the titrant to stream out of the stopcock instead of emerging by drops?

The drop counter counts distinct drops. If the drops are not sufficiently distinct from one another, the drop counter will not function properly and the fluid volume will not be accurate.

12.  Remove the waste container.
13.  Add the micro stir bar to the end of the pH sensor.



14.  Add additional 0.20 M NaOH to the buret so the solution is above the zero mark. Allow some of the NaOH solution to drip into the waste container until the bottom of the meniscus is lined up with or just below the zero mark and record the initial reading in Table 2.
15.  Use the graduated cylinder to pour 50.0 mL of the unknown weak acid solution into a 100-mL beaker and set the beaker on the magnetic stirrer.
16.  Turn on the magnetic stirrer at a gentle rate.
17.  Connect the drop counter to the data collection system.  $\diamond^{(2.2)}$
18.  Display pH versus Drop Count (drops) on a graph.  $\diamond^{(7.1.1)}$

### Collect Data

19.  Clean the lens of the drop counter with water and a cotton swab or tissue.
20.  Start recording data.  $\diamond^{(6.2)}$
21.  Turn the buret stopcock carefully, allowing the titrant to drip slowly (1 to 2 drops per second) into the solution.

**22.**  Continue the titration past the equivalence point until the pH curve flattens.

**23.**  Why is it important to go past the equivalence point?

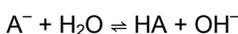
It is necessary to go past the equivalence point in order to find the point where the slope is the steepest. The curve needs to continue past the steepest point to ensure you can tell when the slope begins to flatten.

**24.**  Do you expect a pH drop or a pH rise at the equivalence point? Explain your answer.

Since the pH is low in the initial acid solution and high after the equivalence point when the titrant base solution is in excess, the pH is expected to rise at the equivalence point.

**25.**  Do you expect the pH to be alkaline, neutral, or acidic at the equivalence point? Explain!

At the equivalence point only the salt of the weak acid (its conjugate base) is present. Since the acid is weak, its conjugate base is strong and therefore it will remove some protons from water molecules resulting in  $\text{OH}^-$  ions. Therefore the solution will be slightly alkaline:



**26.**  Stop recording data.  $\diamond^{(6.2)}$

**27.**  Record the final drop count and the final reading of the titrant in the buret to a precision of 0.01 mL in Table 2.

**28.**  Calculate the volume of titrant (final reading minus initial reading) and record this value in Table 2.

Table 2: Titration data

Titration Information	Measurement or Calculation
Initial reading of NaOH in the buret (to 0.01 mL)	0.89
Final reading of NaOH in the buret (to 0.01 mL)	24.69
Volume of titrant (to 0.01 mL)	23.80
Final drop count	714

**29.**  Calibrate the drop counter.  $\diamond^{(3.4)}$

**30.**  Set the horizontal axis to the calculated volume.  $\diamond^{(7.1.9)}$

**31.**  In Table 3, record the volume of titrant used to reach the equivalence point. The equivalence point will be where the slope of the titration curve is the steepest. Find the steepest slope of the data plot to determine this point.  $\diamond^{(9.3)}$

## Lab 24: Determining $K_a$ by Half-Titration of a Weak Acid

- 32.**  Record the pH at the half-titration point (half the volume of titrant used to reach the equivalence point) in Table 3.
- 33.**  Remove the beaker and dispose of its contents according to the teacher's instructions.

### Data Analysis

Table 3: Measurements and determination of the  $pK_a$  of an unknown solution

Parameter	Measured or Calculated Quantity
Volume of 0.20 M NaOH to reach the equivalence point (mL)	23.80
Volume of NaOH to the half-equivalence (or half-titration) point (mL)	11.90
pH at the half-titration point	4.38
Experimental $pK_a$	4.38
Possible solution	Acetic acid
$pK_a$ of the possible solution	4.75
Percent error (%)	7.7

- 1.**  What is the experimental  $pK_a$  of the unknown solution?

The  $pK_a$  is the same as the pH at the half-titration point, so in this example it is 4.38.

- 2.**  How can you identify the unknown solution based on the experimental  $pK_a$ ?

The  $pK_a$  is unique to an acid and can be used to identify an unknown acid. You can use a table that lists acids and their corresponding  $pK_a$  values to identify the unknown solution.

- 3.**  What is the unknown solution?

Acetic acid.

- 4.**  What are sources of error from the titration? Calculate the percent error and record it in Table 3.

The sources of error include inaccuracy of the concentration of either solution, inaccuracy in measuring 50.00 mL of the unknown acid, air bubbles in the buret tip, and parallax error when reading buret volumes.

$$\text{Percent Error} = \frac{|\text{Theoretical Value} - \text{Experimental Value}|}{\text{Theoretical Value}} \times 100$$

$$\text{Percent Error} = \frac{4.75 - 4.38}{4.75} \times 100 = 7.7\%$$

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**Analysis Questions**

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**1. Is the pH of the solution neutral, alkaline, or acidic at the equivalence point?**

The pH at the equivalence point will be alkaline because the conjugate base of a weak acid is stronger than  $\text{OH}^-$ . Therefore it takes a proton away from a water molecule, producing  $\text{OH}^-$  ion:  $\text{A}^- + \text{H}_2\text{O} \rightleftharpoons \text{HA} + \text{OH}^-$

**2. At the half-titration point, the solution is considered to be a buffer. Explain why.**

The solution has a weak acid and the salt of a weak acid (the conjugate base of the weak acid) which makes the solution a buffer.

**3. Would the half-titration volume (the volume of titrant used to reach the half-titration point) be different if propionic acid (which is also a monoprotic weak acid), with the same concentration, had been the unknown weak acid?**

No, the half-titration point would be the same because it depends only on the stoichiometry, which is the same for both acids.

**4. Would the pH of the half-titration point be different if propionic acid (which is also a monoprotic weak acid), with the same concentration, had been the unknown weak acid?**

Yes, the pH of the half-titration point depends on the  $\text{p}K_a$  value of the acid and as such, it depends on the nature of the acid.

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**Synthesis Questions**

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Use available resources to help you answer the following questions.

**1. The acidity constant of formic acid ( $\text{HCOOH}$ ) is  $K_a = 1.8 \times 10^{-4}$ . Would you expect a higher or lower pH if formic acid were used instead of your unknown at the half-titration point? Explain your answer!**

Since the  $K_a$  value is larger, formic acid is a stronger acid. Therefore the  $[\text{H}^+]$  at the half-titration point will be greater, hence the pH will be lower.

**2. If you had to determine the acidity constants of oxalic acid, which is a diprotic acid ( $K_{a1} = 6.5 \times 10^{-2}$ ,  $K_{a2} = 6.1 \times 10^{-5}$ ), what differences would you expect to find in the titration curve?**

The titration curve would have two jumps, one for each of the deprotonation steps.

## Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

- At the equivalence point the solution contains:
  - Only water
  - Half of the untitrated acid and the same amount of salt
  - The product of the titration (salt) and water**
  - Some acid left untitrated
- At the half-titration point:
  - Half of the acid molecules are still untitrated:**
  - Half of the acid molecules are dissociated
  - Half of the acid molecules are overtitrated
  - There are no acid molecules left
- The pH at the half-titration point:
  - Can be calculated from the volume of the titrant necessary to reach the equivalence point
  - Is the same as the pH at the equivalence point
  - Is the same as the  $pK_a$  of the weak acid**
  - Is half of the  $pK_a$  of the weak acid
- The solution is considered a buffer at:
  - The equivalence point
  - The beginning, before the titration
  - Any point after the titration starts and before the equivalence point**
  - Only at the half-titration point

## Extended Inquiry Suggestions

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Provide the students with a sample of an unknown solid acid and have them identify the acid as monoprotic (potassium acid phthalate, potassium bitartrate, or benzoic acid), diprotic (oxalic acid or salicylic acid), or triprotic (citric acid). Have them further identify the acid by determining the molar mass of the acid and comparing it to a list of known acids.

## Lab 25: Order of Reaction

### Objectives

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Students determine the order of reaction by analyzing the reaction rate of crystal violet and NaOH.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Using spectroscopic methods to analyze the reaction rate for a pseudo first-order reaction
- ◆ Calculating the order of reaction using the initial rate method and a graph of the concentration of crystal violet over time
- ◆ Determining the equation needed to obtain a straight-line plot

### Time Requirement

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◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	80 minutes

### Materials and Equipment

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#### *For each student or group:*

- |                          |   |
|--------------------------|---|
| ◆ Data collection system | ◆ Watch glass, 4 in   |
| ◆ Colorimeter            | ◆ 0.1 M Sodium hydroxide (NaOH), 20 mL <sup>1</sup>         |
| ◆ Sensor extension cable | ◆ $1.2 \times 10^{-5}$ M Crystal violet, 20 mL <sup>2</sup> |
| ◆ Cuvette                | ◆ Water, distilled, 30 mL                                   |
| ◆ Beaker, 50-mL (3)      | ◆ Marking pen   |
| ◆ Syringe (3), 5-mL      | ◆ Kimwipes®   |

<sup>1,2</sup>To prepare the solutions, refer to the Lab Preparation section

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Molarity
- ◆ Beer's Law
- ◆ Rate of chemical reactions

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 10: Determining the Equilibrium Constant for a Chemical Reaction
- ◆ Lab 12: Determination of the Rate of the Decomposition of Hydrogen Peroxide
- ◆ Lab 17a: Absorption Spectra

## Using Your Data Collection System

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a sensor to the data collection system ◆<sup>(2.1)</sup>
- ◆ Calibrating the colorimeter ◆<sup>(3.2)</sup>
- ◆ Monitoring live data without recording ◆<sup>(6.1)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Recording a manually sampled data point ◆<sup>(6.3.2)</sup>
- ◆ Finding the values of a point in a graph ◆<sup>(9.1)</sup>
- ◆ Creating calculated data ◆<sup>(10.3)</sup>

## Background

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### Kinetics and Reaction Order

Kinetics is the area of chemistry that deals with how quickly or how slowly reactions take place. By studying the rate of a reaction, valuable information can be gained about how the reaction

proceeds—the reaction mechanism. In general, the rate of a reaction depends on the concentration of the reactants and can be expressed mathematically as the “rate law.”

The rate law for a chemical reaction is an equation that relates the rate of the disappearance of reactants or the rate of appearance of products to the concentration of the reactants. Consider the following reaction:



Generally, the rate law is expressed in terms of the concentrations of reactants and products in the form of

$$\text{Rate} = -\frac{1}{b} \frac{\Delta[B]}{\Delta t} = -\frac{1}{c} \frac{\Delta[C]}{\Delta t} = k[B]^b[C]^c \dots \quad (1)$$

where

$k$  = the rate constant

$b$  and  $c$  = the individual reaction orders for each reactant

The individual reactant reaction orders (that is, the exponents) in Equation 1 must be determined by experiment because they are not necessarily based on the mole ratio in the balanced equation. The sum of the individual orders is the “overall order” of the reaction.

The order of a reaction is commonly zero order, first order, or second order. A zero-order reaction is one in which the rate is *independent* of the concentration of reactants. A first-order reaction is one where the rate depends only on the concentration of one reactant, and in which  $b = 1$ . A second-order reaction can be one in which the reaction rate depends on the concentration of two different reactants (where  $b = 1$  and  $c = 1$ ), or where the reaction rate depends on the concentration of one reactant (where  $b = 2$ ). These options are shown in the "Rate" column in Table 1.

Table 1: Characteristics of reaction order

Overall Reaction Order	Reactants and Products	Rate	Equation to Obtain a Straight-Line Plot (Integrated Rate Equations)
Zero order	$B \rightarrow \text{Products}$	$k$	$[B] = -kt$
First order	$B \rightarrow \text{Products}$	$k[B]$	$\ln[B] = \ln[B]_0 - kt$
Second order	$B + C \rightarrow \text{Products}$	$k[B][C]$	$\ln\left(\frac{[C]/[C]_0}{[B]/[B]_0}\right) = ([C]_0 - [B]_0)kt$
	$2B \rightarrow \text{Products}$	$k[B]^2$	$\frac{1}{[B]} = \frac{1}{[B]_0} + kt$

### Determining the Rate of Reaction

The equation to show the dependence of the concentration of the reactant as a function of time, also called “integrated rate equation,” depends on the order of the reactant. These integrated

## Lab 25: Order of Reaction

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rate equations are shown for different reaction orders in Table 1. (The zero index on the concentrations references initial condition.)

Creating a graph of concentration versus time is one method that can be used to determine the order of a reaction with respect to a particular reactant. If the reaction is zero-order, a plot of concentration versus time will result in a straight line with the slope =  $-k$ . If the reaction is first-order, a straight-line plot will result from a graph of the natural log of concentration versus time, with the slope equal to  $-k$ . If the reaction is second-order, a plot of  $1/\text{concentration}$  versus time will result in a straight line with the slope equal to  $+k$ .

A more rigorous approach is the “initial rate method.” This method relies on the fact that we know the concentrations of the reactants at  $t = 0$  seconds and the order of the reactants can be calculated from the measured rate starting at this time. We will be using the latter approach in this activity.

To measure the rate of a reaction, we monitor the change of the concentration of one of the reactants or products:

$$\text{Rate} = -\frac{1}{b} \frac{\Delta[\text{B}]}{\Delta t}$$

The negative sign accounts for the fact that the change of the concentration of a reactant is negative—the negative sign turns the rate positive. We scale the rate with the stoichiometric coefficient so that the rate calculated for any species involved in the reaction is the same.

For second-order reactions where the rate depends on the concentration of two species and the concentration of one of the species is in large excess, the concentration of the species in excess will not change appreciably and can be considered constant. Therefore, the reaction behaves like a first-order reaction. We designate this as a “pseudo first-order reaction” (this assumes  $[\text{B}] \gg [\text{C}]$ ):

$$\text{Rate} = k[\text{B}][\text{C}] \approx k'[\text{C}]$$

where

$$k' = k[\text{B}], \text{ the pseudo rate constant}$$

## Pre-Lab Activity

### Setting the stage for the activity

In this lab you will monitor the concentration of a reactant in a chemical reaction using a spectroscopic method and applying Beer's Law:

$$A = \epsilon l c \quad (2)$$

where

$A$  = absorption

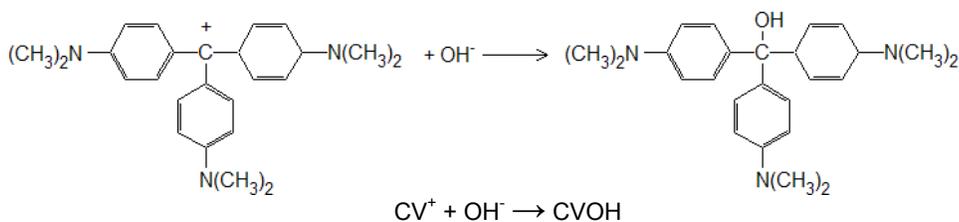
$\epsilon$  = molar absorption coefficient at 592 nm; the published value is  $5 \times 10^4 \text{ cm}^{-1} \text{ M}^{-1}$

$l$  = path length of solution the light passes through (the width of the container with the solution)

$c$  = concentration of the absorbing species

(Please note that the AP version of this equation is expressed as  $A = a b c$ ; where  $a$  and  $b$  correspond to  $\epsilon$  and  $l$ , respectively.)

The chemical reaction is between crystal violet ( $\text{CV}^+$ ), a dye with an intense blue color (therefore it absorbs light in the visible range), and  $\text{OH}^-$  ions:



The product is colorless. The absorption is attributed completely to the reactant present in the solution.

### Initial Rate Method

One way to determine the order of the reactants in a reaction is to utilize the “initial rate method.” Applying this technique, one measures the initial rate of the reaction under carefully chosen conditions and then applies the rate equation (in terms of the rate constant and the concentration of the reactants) under two different conditions.

Applying Equation 1 (with subscripts 1 and 2 to indicate the two sets of conditions) the rate, concentration of  $\text{CV}^+$ , and the concentration of  $\text{OH}^-$  for the two conditions will be:

$$\text{Rate}_1 = k[\text{CV}^+]_1^m [\text{OH}^-]_1^n$$

$$\text{Rate}_2 = k[\text{CV}^+]_2^m [\text{OH}^-]_2^n$$

## Lab 25: Order of Reaction

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In order to eliminate one of the variables, we divide the first equation by the second one:

$$\frac{Rate_1}{Rate_2} = \frac{k[CV^+]_1^m [OH^-]_1^n}{k[CV^+]_2^m [OH^-]_2^n} \quad (3)$$

If the initial concentrations are set so that  $[CV^+]_1 = [CV^+]_2$  and  $[OH^-]_2 = 2[OH^-]_1$ , Equation 3 becomes:

$$\frac{Rate_1}{Rate_2} = \frac{[OH^-]_1^n}{[OH^-]_2^n} = \frac{[OH^-]_1^n}{(2[OH^-]_1)^n} = \frac{1}{2^n} \quad (4)$$

Since  $n$  is the exponent, it is logical to take the logarithm of both sides of Equation 4:

$$\ln\left(\frac{Rate_1}{Rate_2}\right) = \ln\left(\frac{1}{2^n}\right) = \ln(2^{-n}) = -n \ln 2 \quad (5)$$

Multiplying both sides of Equation 5 by  $(-1)$  yields:

$$-\ln\left(\frac{Rate_1}{Rate_2}\right) = n \ln 2 \quad (6)$$

For convenience, we eliminate the negative sign in front of the logarithmic term in Equation 6 with the following two steps:

$$\ln\left(\frac{Rate_1}{Rate_2}\right)^{-1} = n \ln 2$$

$$\ln\left(\frac{Rate_2}{Rate_1}\right) = n \ln 2$$

Equation 6 can then be rearranged to solved for  $n$ :

$$n = \frac{\ln\left(\frac{Rate_2}{Rate_1}\right)}{\ln(2)}$$

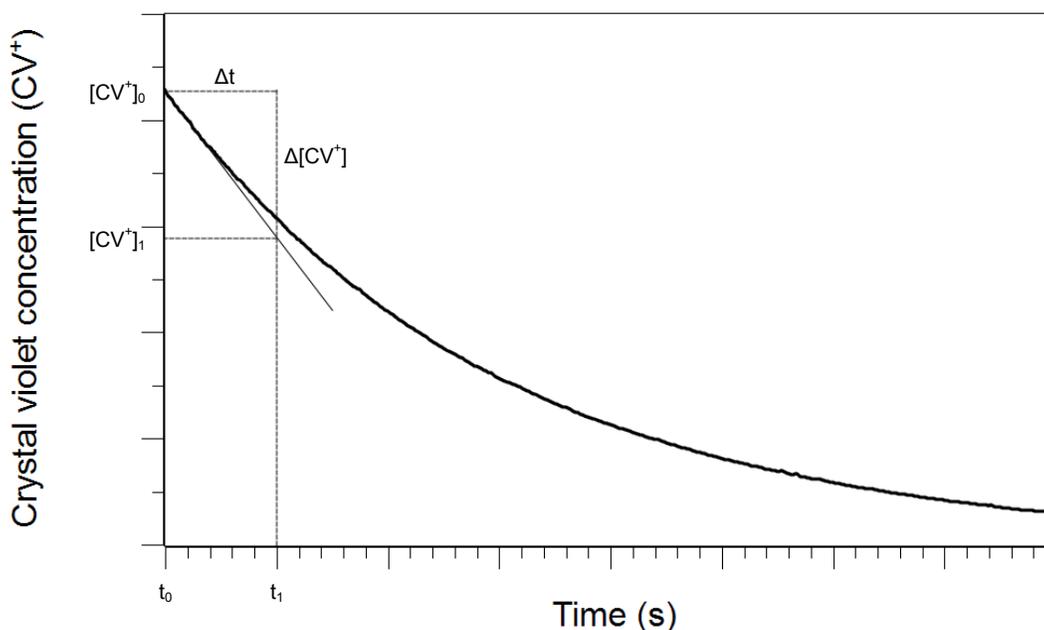
To determine the order  $m$  of  $CV^+$ , we design a third experiment which we will label with the subscript "3," with the initial conditions:  $[CV^+]_3 = 2[CV^+]_1$  and  $[OH^-]_3 = [OH^-]_1$ . The same derivation can be performed as before and the following expression can be obtained for  $m$ :

$$m = \frac{\ln\left(\frac{Rate_3}{Rate_1}\right)}{\ln(2)}$$

By measuring the rates of the three reactions with the concentrations as defined, both  $n$  and  $m$  can be determined.

### Measuring the Reaction Rates

To measure the rate, we will utilize the fact that the rate is the value of the slope at every point of the Crystal violet concentration versus Time graph:



The initial rate is determined as the slope of the line drawn from the initial concentration point to the concentration at the point corresponding to  $t_1$ ,  $[CV^+]_1$ .

Once the orders of the reactants are obtained, we can determine the value of  $k$  by using the appropriate rate representation from Table 1.

#### Example calculation to try

In an experiment, the following concentrations were prepared for  $CV^+$  and  $OH^-$ :

Table 2: Concentration of reactants for 3 trials

Trial	$[CV^+]$ (M)	$[OH^-]$ (M)
1	$3.0 \times 10^{-6}$	$2.5 \times 10^{-2}$
2	$6.0 \times 10^{-6}$	$5.0 \times 10^{-2}$
3	$6.0 \times 10^{-6}$	$2.5 \times 10^{-2}$

## Lab 25: Order of Reaction

For each run, the reactants were mixed and the reaction mixture was transferred into a spectroscopic cell. Without any delay, the cell was placed into a colorimeter. The absorbance data was collected at 592 nm until the absorbance dropped below 0.1. The absorbance data was then converted to concentration data in a spreadsheet program:

Repeating Equation 2,

$$A = \epsilon l c$$

and solving for  $c$  (concentration),

$$[CV^+] = \frac{A}{\epsilon l}$$

From the slopes of the curves on the  $[CV^+]$  versus Time graph, the initial rates were determined (shown by the straight lines) and listed in Table 3.

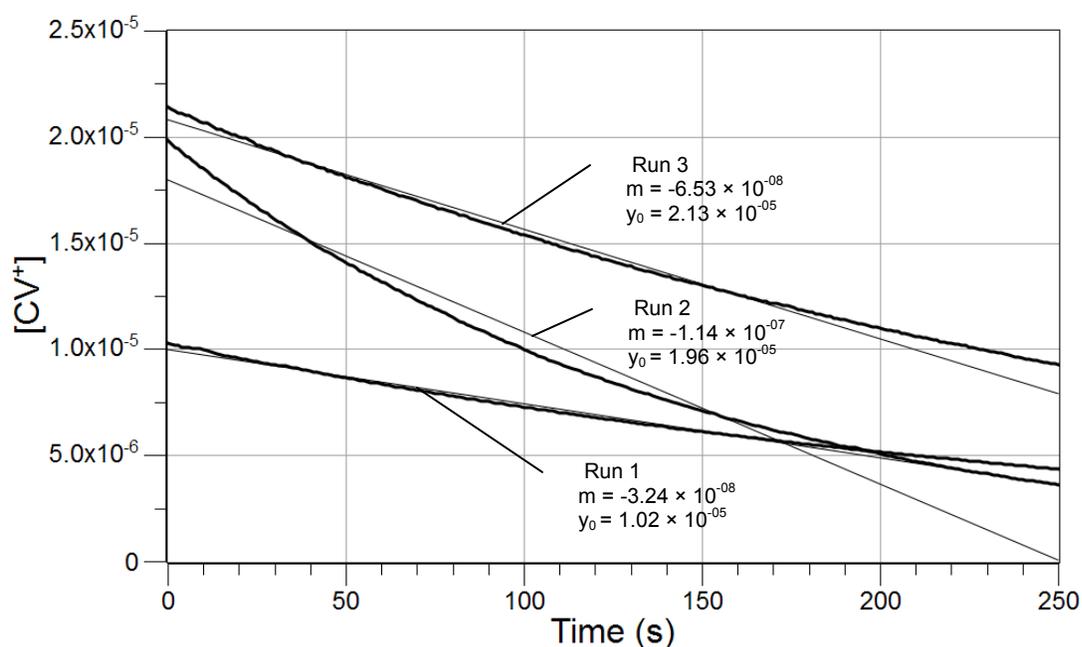


Table 3: Initial rates

Trial	Initial Rate (M/s)
1	$3.2 \times 10^{-8}$
2	$1.1 \times 10^{-7}$
3	$6.5 \times 10^{-8}$

In Trial 3, the concentration of  $\text{CV}^+$  was prepared to be twice as much as in Trial 1:

$$[\text{CV}^+]_3 = 2[\text{CV}^+]_1:$$

$$\frac{\text{Rate}_1}{\text{Rate}_3} = \frac{[\text{CV}^+]_1^m}{[\text{CV}^+]_3^m} = \frac{[\text{CV}^+]_1^m}{(2[\text{CV}^+]_1)^m} = \frac{1}{2^m}$$

Taking the logarithm of this equation and multiplying both sides by  $(-1)$  yields:

$$\ln\left(\frac{\text{Rate}_1}{\text{Rate}_3}\right) = \ln\left(\frac{1}{2^m}\right) = \ln(2^{-m}) = -m \ln 2$$

$$\ln\left(\frac{\text{Rate}_3}{\text{Rate}_1}\right) = m \ln 2$$

Solving this equation for  $m$  gives the following results:

$$m = \frac{\ln\left(\frac{\text{Rate}_3}{\text{Rate}_1}\right)}{\ln 2} = \frac{\ln\left(\frac{6.5 \times 10^{-8} \text{ M}^{-1} \text{ s}^{-1}}{3.2 \times 10^{-8} \text{ M}^{-1} \text{ s}^{-1}}\right)}{\ln 2} = \frac{0.708}{0.693} = 1.010 \approx 1.0$$

In Trial 2, the concentration of  $\text{OH}^-$  was twice as much as in Trial 3:  $[\text{OH}^-]_2 = 2[\text{OH}^-]_3$ :

$$\frac{\text{Rate}_3}{\text{Rate}_2} = \frac{[\text{OH}^-]_3^n}{[\text{OH}^-]_2^n} = \frac{[\text{OH}^-]_3^n}{(2[\text{OH}^-]_3)^n} = \frac{1}{2^n}$$

Taking the logarithm of this equation and multiplying both sides by  $(-1)$  yields:

$$\ln\left(\frac{\text{Rate}_3}{\text{Rate}_2}\right) = \ln\left(\frac{1}{2^n}\right) = \ln(2^{-n}) = -n \ln 2$$

$$\ln\left(\frac{\text{Rate}_2}{\text{Rate}_3}\right) = n \ln 2$$

Solving this equation for  $n$  gives the results:

$$n = \frac{\ln\left(\frac{\text{Rate}_2}{\text{Rate}_3}\right)}{\ln 2} = \frac{\ln\left(\frac{1.1 \times 10^{-7} \text{ M}^{-1} \text{ s}^{-1}}{6.5 \times 10^{-8} \text{ M}^{-1} \text{ s}^{-1}}\right)}{\ln 2} = \frac{0.526}{0.693} = 0.759 \approx 1$$

It appears that the order of both reactants is 1, so the overall order (the sum of the individual orders) is 2. Since the concentration of  $\text{OH}^-$  is much greater than the concentration of  $\text{CV}^+$ , the concentration of  $\text{OH}^-$  will not change appreciably, which makes the reaction a *pseudo first-order reaction*:

$$\text{Rate} = k[\text{OH}^-][\text{CV}^+] \approx k'[\text{CV}^+]$$

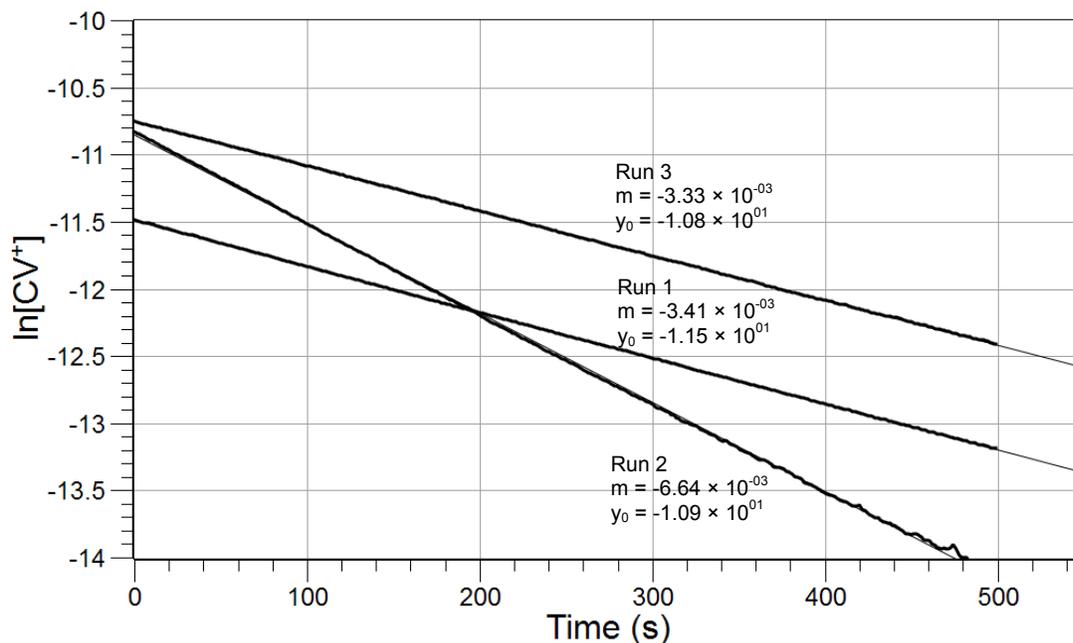
Therefore, the applicable rate law is

$$\ln[\text{CV}^+] = \ln[\text{CV}^+]_0 - k't$$

## Lab 25: Order of Reaction

The plot of  $\ln[\text{CV}^+]$  versus Time gave a straight line for each run. From the slope, the rate constant  $k$  can be calculated:

$$k = \frac{k'}{[\text{OH}^-]}$$



The following data was obtained and the rate constant calculated.

Table 4: Determination of the reaction rate constant

Trial	$k'$ ( $\text{M s}^{-1}$ )	$[\text{OH}^-]$ (M)	$k$ ( $\text{s}^{-1}$ )
1	$3.4 \times 10^{-3}$	$2.5 \times 10^{-2}$	0.14
2	$6.6 \times 10^{-3}$	$5.0 \times 10^{-2}$	0.13
3	$3.3 \times 10^{-3}$	$2.5 \times 10^{-2}$	0.13

Using the results from Trial 1:

$$k = \frac{k'}{[\text{OH}^-]} = \frac{3.4 \times 10^{-3} \text{ s}^{-1}}{2.5 \times 10^{-2} \text{ M}} = 0.13 \text{ M}^{-1} \text{ s}^{-1}$$

### 1. What does the value of the intercept represent on the $\ln[\text{CV}^+]$ versus time graph?

The intercept represents  $\ln[\text{CV}^+]_0$ .

**2. Why do the initial rates have to be measured (as opposed to measuring the rate at any other time point) in order to calculate the order of the reactants? (Hint: what are the concentrations of the reactants in the beginning of the reaction and what are they later?)**

The rates have to be determined in the beginning because that is the only point where we know the concentrations of the reactants. As the reaction progresses, we no longer know the concentrations of the reactants to use in the equations to calculate  $n$  or  $m$ .

**3. If at  $t_0 = 0$  seconds  $[\text{CV}^+]_0 = 5.0 \times 10^{-6}$  M and at  $t_1 = 10$  seconds,  $[\text{CV}^+]_1 = 2.0 \times 10^{-6}$  M, what is the initial rate of the reaction?**

$$\text{Rate} = -\frac{\Delta[\text{CV}^+]}{\Delta t} = -\frac{[\text{CV}^+]_1 - [\text{CV}^+]_0}{t_1 - t_0} = -\frac{(2.0 \times 10^{-6} \text{ M}) - (5.0 \times 10^{-6} \text{ M})}{(10 \text{ s}) - (0 \text{ s})} = 3.0 \times 10^{-7} \frac{\text{M}}{\text{s}}$$

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

- 1. 0.1 M NaOH:** Dissolve 4.0 g of NaOH in some distilled water in a 100-mL volumetric flask. Fill the flask to the mark and mix well.
- 2.  $1.2 \times 10^{-5}$  M Crystal violet:** Dissolve 0.0472 g of crystal violet in some distilled water in a 100-mL volumetric flask. Fill the flask to the mark and mix the solution well. Make sure there are no solid particles on the bottom after the solution rests for a few minutes. Pipet 10.00 mL of this solution into a 1-L volumetric flask. Fill the flask to the mark and mix the solution well.

## Safety

Add these important safety precautions to your normal laboratory procedures:

- ◆ In case of contact with skin, NaOH should be washed off with a large amount of water.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

1	2	5	4	3
Set up the data collection system. Calibrate the colorimeter.	Draw the calculated amount of the two solutions and the water into the syringes. Start recording data.	Repeat the experiment with the other two sets of concentrations. Determine the order of the reactants.	Record data until the absorbance drops below 0.1.	Fill a cuvette with the NaOH solution and water. Add the CV <sup>+</sup> solution and immediately put the cuvette in the colorimeter.

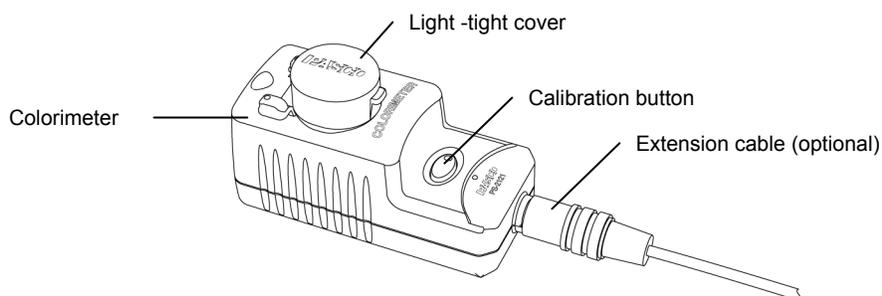
## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "◆" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

- Start a new experiment on the data collection system ◆<sup>(1,2)</sup>
- Connect the colorimeter to the data collection system using the extension cable. ◆<sup>(2,1)</sup>
- Calibrate the colorimeter. ◆<sup>(3,2)</sup>



- Put about 20 mL of the crystal violet solution, the sodium hydroxide solution, and the distilled water in separate 50-mL beakers. Label the beakers.
- Cover the NaOH solution with a watch glass.

6.  Why is it necessary to cover the NaOH solution with a watch glass?

NaOH absorbs CO<sub>2</sub> from air and forms NaHCO<sub>3</sub>.

7.  Label one syringe “CV”, one “NaOH”, and one “Water”.
8.  For the first run, fill the three syringes with the volume of liquids specified in Table 5, Trial 1.
9.  Eliminate the air bubbles from the syringes by holding the syringe over a sink, pointing upward, knocking gently on the syringe and pushing the plunger in until all the air is expelled. Draw in more solution as needed.

Table 5: Initial volumes and concentrations

Trial	Initial Concentration (M)		Volume (mL)			
	[CV <sup>+</sup> ]	[OH <sup>-</sup> ]	Crystal Violet 1.2 × 10 <sup>-5</sup> M	NaOH 0.10 M	Water	Total
1	3.0 × 10 <sup>-6</sup>	2.5 × 10 <sup>-2</sup>	1.50	1.50	3.00	6.00
2	6.0 × 10 <sup>-6</sup>	5.0 × 10 <sup>-2</sup>	3.00	3.00	0.0	6.00
3	6.0 × 10 <sup>-6</sup>	2.5 × 10 <sup>-2</sup>	3.00	1.50	1.50	6.00

### Collect Data

10.  Perform the experiment for each of the three runs as follows:
- Deliver the water and sodium hydroxide solutions from the syringes into a clean cuvette.
  - Start data recording. <sup>(6.2)</sup>
  - As quickly as possible, add the crystal violet solution to the cuvette, close the cuvette, invert it a few times, wipe it dry and clean and place it into the colorimeter.
- Note:** This should be done as quickly as possible to minimize the error arising from the delay of collecting data after mixing the reactants.
- Immediately close the lid on the colorimeter and start data recording. <sup>(6.2)</sup>
  - Why do you think the cuvette should be clean on the outside?

The wall should be free of fingerprints and other marks because contamination introduces error in the detection of absorption.

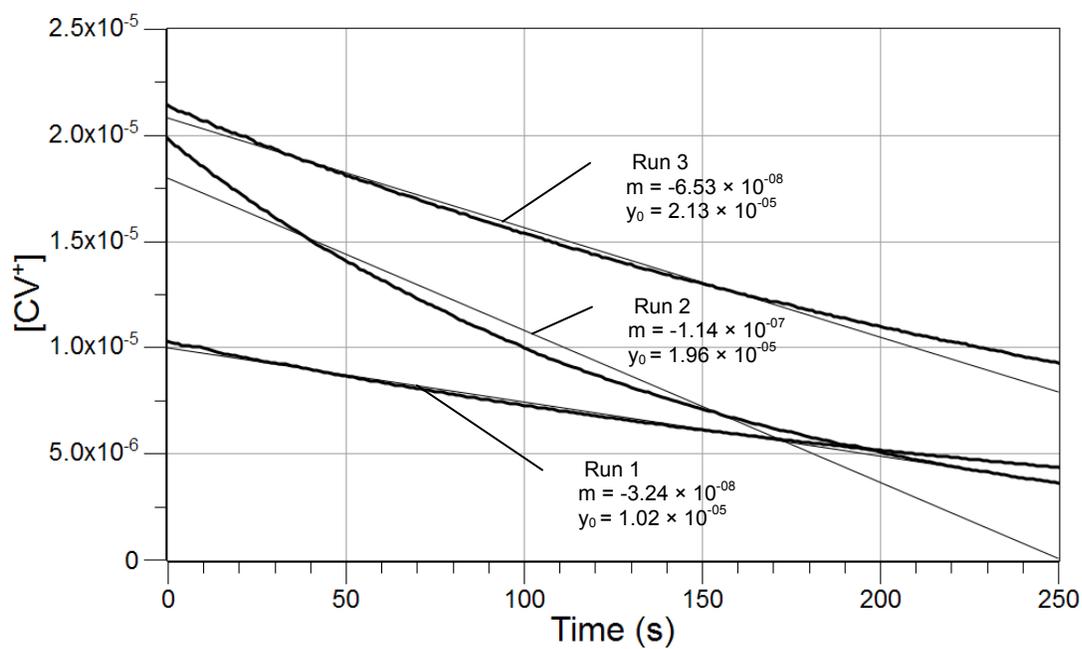
- Continue taking readings until the absorbance drops below 0.1.

## Lab 25: Order of Reaction

- g.** Stop data recording  $\diamond^{(6.2)}$  and clean the cuvette.
- h.** For the next run, fill the three syringes with the volume of liquids specified for the next trial (in Table 5) and eliminate air bubbles, as before. Repeat the process for each of the next two runs.
- 11.**  Print the graph.  $\diamond^{(11.2)}$
- 12.**  Save your experiment.  $\diamond^{(11.1)}$  and clean up according to your teacher's directions.

## Data Analysis

- 1.**  In the data collection system, create a calculated data set to obtain the concentrations as a function of time for each trial using Beer's Law  $\diamond^{(10.3)}$  knowing the path  $l = 1.65$  cm and  $\epsilon = 5 \times 10^4 \text{ M}^{-1} \text{ s}^{-1}$ .
- 2.**  Display the data set on the y-axis with Time on the x-axis.  $\diamond^{(7.1.1)}$
- 3.**  Find the slope and intercept of the best-fit line.  $\diamond^{(9.6)}$
- 4.**  Print the graph.  $\diamond^{(11.2)}$
- 5.**  Sketch or attach the Concentration versus Time graph, showing the slopes.



6.  Find the slope of the best-fit line over the first 20 to 25 points to determine the initial rate on your graphs. <sup>(9.6)</sup> Consider those points which are still lined up on a straight line. Record the slopes of those lines (which actually are the rates for the reactions) in Table 6.

Table 6: Initial rates

Trial	Initial Rate (M/s)
1	$3.2 \times 10^{-8}$
2	$1.1 \times 10^{-7}$
3	$6.5 \times 10^{-8}$

7.  With these values of the rates, calculate  $m$  from the rates of Trial 1 and Trial 3.

$$m = \frac{\ln\left(\frac{\text{Rate}_3}{\text{Rate}_1}\right)}{\ln 2} = \frac{\ln\left(\frac{6.5 \times 10^{-8} \text{ M}^{-1} \text{ s}^{-1}}{3.2 \times 10^{-8} \text{ M}^{-1} \text{ s}^{-1}}\right)}{\ln 2} = 1$$

8.  With these values of the rates, calculate  $n$  from the rates of Trial #2 and Trial #3.

$$n = \frac{\ln\left(\frac{\text{Rate}_2}{\text{Rate}_3}\right)}{\ln 2} = \frac{\ln\left(\frac{1.1 \times 10^{-7} \text{ M}^{-1} \text{ s}^{-1}}{6.5 \times 10^{-8} \text{ M}^{-1} \text{ s}^{-1}}\right)}{\ln 2} = \frac{0.526}{0.693} = 0.759 \approx 1$$

9.  With these values of  $n$  and  $m$ , what is the rate expression in terms of the concentrations of the reactants  $n$  and  $m$ ?

$$\text{Rate} = k[\text{CV}^+][\text{OH}^-]$$

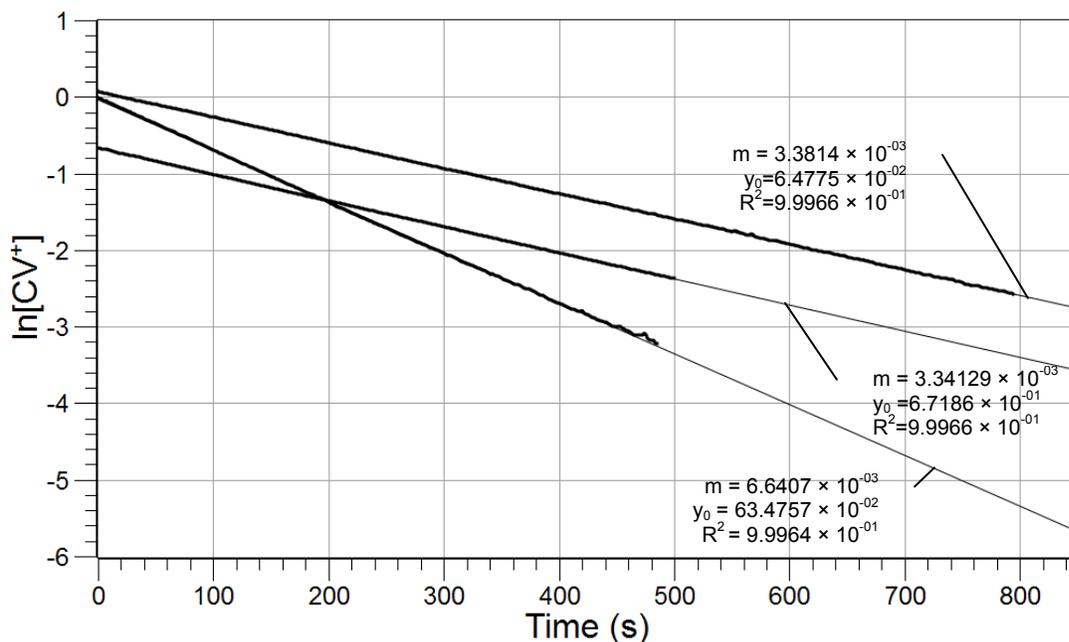
10.  Consider that  $[\text{OH}^-] \gg [\text{CV}^+]$  and won't change appreciably during the reaction. The  $\text{OH}^-$  concentration can be considered "constant" and can be combined with  $k$  to form  $k'$ , which is a pseudo rate constant. What is the value of the pseudo rate constant in terms of the rate constant and the concentration of  $\text{OH}^-$ ?

$$k' = k[\text{OH}^-]$$

11.  What is the rate expression using the pseudo rate constant instead of the rate constant and the concentration of  $\text{OH}^-$ ?

$$\text{Rate} = k'[\text{CV}^+]$$

12.  Based on the order determined for the two reactants, display a graph with the appropriate representation of your data ( $\ln[\text{CV}^+]$  or  $1/[\text{CV}^+]$  versus Time). Sketch or paste the resulting curve for each trial below.



13. □ From the graph above, calculate the rate constant,  $k$ , from the values of the pseudo rate constant,  $k'$ .

For Trial 1:

$$k = \frac{k'}{[\text{OH}^-]}$$

$$k = \frac{3.4 \times 10^{-3} \text{ M s}^{-1}}{2.5 \times 10^{-2} \text{ M}} = 0.14$$

Table 7: Determination of the reaction rate constant

Trial	$k'$ ( $\text{M s}^{-1}$ )	$[\text{OH}^-]$ (M)	$k$ ( $\text{s}^{-1}$ )
1	$3.4 \times 10^{-3}$	$2.5 \times 10^{-2}$	0.14
2	$6.6 \times 10^{-3}$	$5.0 \times 10^{-2}$	0.13
3	$3.3 \times 10^{-3}$	$2.5 \times 10^{-2}$	0.13

## Analysis Questions

1. What values did you get for  $n$  and  $m$ ? What is the overall order of the reaction?

Both  $n$  and  $m$  should be 1. Therefore, the overall order of the reaction is second order.

2. What is the average value of the rate constant that you obtained with standard deviation?

$$k = 0.14 \pm 0.01 \text{ s}^{-1}$$

**3. How did the color of the solution change during the reaction? Explain!**

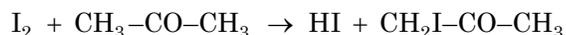
The color intensity decreased since the concentration of the absorbing species was decreasing.

**4. How do you think the rate would change if we doubled the concentration of both reactants?**

The rate would increase four fold.

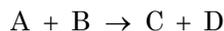
**Synthesis Questions**

Use available resources to help you answer the following questions.

**1. Iodine reacts with acetone to form iodo-acetone according to the following reaction:**

Iodine is brown; the acetone, hydrogen iodide, and iodo-acetone are colorless. The order of both reactants is 1. Propose a method to measure the rate constant for this reaction.

The reaction can be monitored by measuring the absorbance of iodine. From the absorbance, the iodine concentration can be calculated the same way we calculated it in this experiment for the crystal violet.

**2. When only one species is absorbing it is easy to follow the reaction. However, if there are two species involved in absorption, the situation is somewhat more complicated. Consider the following hypothetical reaction:**

where A absorbs in the blue range and C absorbs in the yellow range of the visible spectrum. Do you think the reaction can be monitored by monitoring the absorption of the solution? If so, how? Explain!

Since the colors are substantially different, both species can be monitored simultaneously. The blue species absorbs around 440 nm while the yellow absorbs around 500 nm.

**3. How would your strategy be different from this activity if you were to monitor the concentration of C in the previous example? (Hint: how would the absorption of C change as the reaction progresses?)**

Instead of the consumption of a reactant being monitored, one could monitor the accumulation of one of the products which is numerically the same (because of the stoichiometry). However, they have opposite signs.

## Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

- 1. The rate of the reaction between  $\text{CV}^+$  and  $\text{OH}^-$  depends:**
  - A.** Only on the concentration of the reactants.
  - B.** Only on the rate constant,  $k$ .
  - C. On the concentrations of the reactants and the rate constant,  $k$ .**
  - D.** On the ratio of the concentrations of the reactants and the rate constant,  $k$ .
- 2. In the reaction between  $\text{CV}^+$  and  $\text{OH}^-$ , doubling the concentration of  $\text{CV}^+$ :**
  - A.** Will not change the reaction rate.
  - B.** Will double the rate constant.
  - C. Will double the reaction rate.**
  - D.** Will not have an affect on the reaction at all.
- 3. Generally in a chemical reaction, changing the concentration of a reactant:**
  - A.** Will always change the rate of the reaction.
  - B. Will always change the rate of the reaction except if the order of that reactant is zero-order.**
  - C.** Will always change the rate of the reaction except if the order of that reactant is first-order.
  - D.** Will always change the rate of the reaction except if the order of that reactant is second-order.
- 4. The absorbance is:**
  - A.** Not related to the concentration of the absorbing species.
  - B.** Proportional to the concentration of the absorbing species only.
  - C. Proportional to the concentration of the absorbing species and the path length of solution the light passes through.**
  - D.** Proportional only to the path length through which the light has to pass.

## Extended Inquiry Suggestions

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An Arrhenius analysis can be performed with this reaction. If the cuvette is kept in a water bath at various temperatures, the reaction can be performed at different temperatures to determine the activation energy of the reaction:

$$k = Ae^{-E_a/RT}$$

$$\ln k = \ln A - \frac{E_a}{R} \frac{1}{T}$$

## Lab 26: Conductometric Titration

### Objectives

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Students determine the titration equivalence point with a conductometric titration method.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Using a conductivity sensor and drop counter to perform titration measurements
- ◆ Determining equivalence points and solution concentrations

### Time Requirement

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◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

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#### *For each student or group:*

- ◆ Data collection system
- ◆ Conductivity sensor
- ◆ Drop counter with micro stir bar
- ◆ Magnetic stirrer
- ◆ Buret, 50-mL
- ◆ Beaker (2), 100-mL
- ◆ Beaker, 50-mL
- ◆ Volumetric pipet, 50-mL
- ◆ Ring stand
- ◆ Clamp, right-angle
- ◆ Clamp, buret
- ◆ 0.0200 M H<sub>2</sub>SO<sub>4</sub> solution, 50-mL<sup>1</sup>
- ◆ Barium hydroxide (Ba(OH)<sub>2</sub>), unknown concentration, 50-mL<sup>1</sup>
- ◆ Deionized water, 50 mL
- ◆ Wash bottle with deionized water
- ◆ Cotton swab or tissue

<sup>1</sup>To prepare the solutions, refer to the Lab Preparation section.

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Acids and bases
- ◆ Electrolytes
- ◆ Molarity
- ◆ Titration

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 6: Standardizing a Solution of Sodium Hydroxide
- ◆ Lab 7: Acid–Base Titration
- ◆ Lab 8: Oxidation–Reduction Titration
- ◆ Lab 9: Mole Relationships in a Chemical Reaction
- ◆ Lab 23: Determination of a Solubility Product
- ◆ Lab 24; Determining  $K_a$  by Half-Titration of a Weak Acid

## Using Your Data Collection System

---

Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a sensor to the data collection system. ◆<sup>(2.1)</sup>
- ◆ Connecting multiple sensors to your data collection system ◆<sup>(2.2)</sup>
- ◆ Calibrating the drop counter ◆<sup>(3.4)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>
- ◆ Adjusting the scale of a graph ◆<sup>(7.1.2)</sup>
- ◆ Changing the variable on the x-axis and y-axis of a graph ◆<sup>(7.1.9)</sup>

- ◆ Finding coordinates of a point on a graph ◆<sup>(9.1)</sup>
- ◆ Saving your experiment ◆<sup>(11.1)</sup>
- ◆ Printing the graph. ◆<sup>(11.2)</sup>

## Background

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As you may recall from titrations done in earlier labs, a titration determines the amount, or concentration, of a reactant in a chemical reaction. You might have used different methods to detect the equivalence point, which is the point at which stoichiometric quantities of reactants combine.

During this experiment, you will determine the equivalence point with a conductometric method, making use of the fact that solutions conduct electricity when ions are present. Higher ion concentration, in general, yields higher conductivity.

Consider a strong electrolyte of unknown concentration. The solution conducts electricity due to the presence of ions. Adding another strong electrolyte that reacts with those ions produces a precipitate or a non-conducting molecule (such as H<sub>2</sub>O) or a gas which leaves the solution. Because of this, the ion concentration and conductivity decreases.

Continued addition of the second electrolyte continues to lower the ion concentration and the conductivity until the equivalence point is reached. At the equivalence point, the two electrolytes are combined in their stoichiometric ratio. That is, all ions are removed and conductivity is at its minimum.

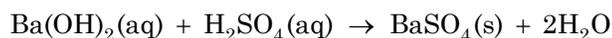
Conductivity increases if more of the second electrolyte is added after the equilibrium point. Although no chemical reaction occurs at this point, ions are being added to the solution from that second electrolyte.

## Pre-Lab Activity

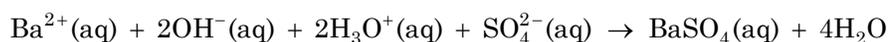
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### *Setting the stage for the activity*

During this activity, you will determine the concentration of a Ba(OH)<sub>2</sub> solution. You will titrate the Ba(OH)<sub>2</sub> solution with a H<sub>2</sub>SO<sub>4</sub> solution, which react according to the following equation:



The net ionic equation of the reaction is:

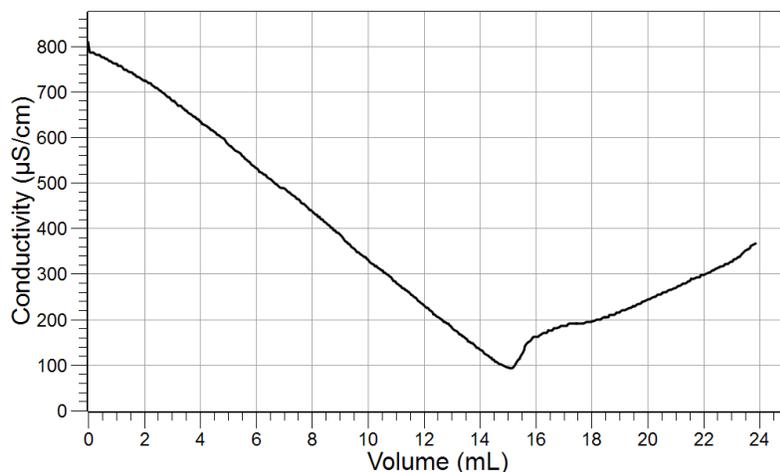


Before the equivalence point, the Ba<sup>2+</sup> and OH<sup>-</sup> ions are responsible for the conductivity. After the equivalence point, the excess H<sub>3</sub>O<sup>+</sup> and SO<sub>4</sub><sup>2-</sup> ions are responsible for the conductivity. At the equivalence point, none of these ions are present in any significant concentration.

## Lab 26: Conductometric Titration

### Example calculation to try

A sample of 50.00 mL  $\text{Ba}(\text{OH})_2$  solution with a concentration of about  $5.00 \times 10^{-3}$  M is pipetted into a 100-mL beaker. A conductivity sensor is placed into the solution and a drop counter is installed over the solution. The unknown solution is titrated with 0.0200 M  $\text{H}_2\text{SO}_4$ . The titration curve obtained is shown below:



The equivalence point (the point with the lowest conductivity) was reached after adding 15.12 mL of 0.0200 M  $\text{H}_2\text{SO}_4$ . The molarity of the barium hydroxide solution can be obtained knowing the volume and concentration of the sulfuric acid solution:

$$\begin{aligned} 15.12 \text{ mL} & \left( \frac{0.0200 \text{ mol H}_2\text{SO}_4}{1000 \text{ mL H}_2\text{SO}_4} \right) \left( \frac{1 \text{ mol Ba}(\text{OH})_2}{1 \text{ mol H}_2\text{SO}_4} \right) \left( \frac{1}{0.0500 \text{ L Ba}(\text{OH})_2} \right) \\ & = 6.05 \times 10^{-3} \text{ M Ba}(\text{OH})_2 \end{aligned}$$

The concentration of the unknown  $\text{Ba}(\text{OH})_2$  solution was  $6.05 \times 10^{-3}$  M.

### 1. Why is the conductivity curve not symmetrical around the equivalence point?

The ions present before and after the equivalence point have different abilities to conduct electricity.

### 2. What will happen in the titrated solution as the titrant solution is being added?

Increasing amounts of white precipitate will appear.

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

- $4.0 \times 10^{-3}$  M  $\text{Ba}(\text{OH})_2$ :** Dissolve 1.262 g of  $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$  with water in a 1-L volumetric flask and fill it to the mark.
- 0.0200 M  $\text{H}_2\text{SO}_4$ :** Combine 1.10 mL of 98%  $\text{H}_2\text{SO}_4$  solution to about 300 mL of deionized water in a 1-L volumetric flask and fill it to the mark.

**Note:** Standardize the solution and report the actual concentration to the students.

## Safety

Follow all standard laboratory procedures.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

3	2	4	5	1
Fill a 50 mL buret with the titrant, 0.0200 M $\text{H}_2\text{SO}_4$ , and set the meniscus to the zero mark.	Then put an accurately measured quantity of the $\text{Ba}(\text{OH})_2$ solution into a beaker. Place it onto the magnetic stirrer.	Perform the titration. Add the titrant solution until the conductivity drops and rises again. Record the volume.	Determine the concentration of the $\text{Ba}(\text{OH})_2$ solution.	Set up the titration apparatus with a stirrer, drop counter, and conductivity sensor.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

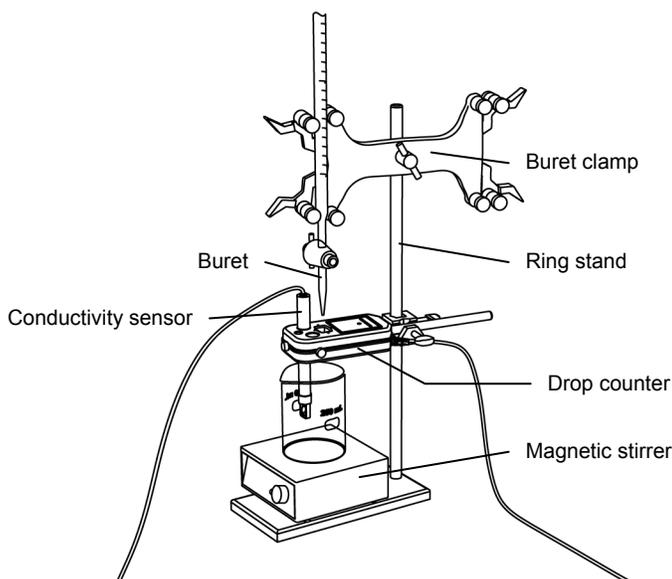
**Note:** When students see the symbol "♦" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

- Start a new experiment on the data collection system. ♦<sup>(1,2)</sup>
- Connect the conductivity sensor to the data collection system. ♦<sup>(2,1)</sup>

## Lab 26: Conductometric Titration

3.  Assemble the titration apparatus, using the steps below and the illustration as a guide.
- Position the magnetic stirrer on the base of the ring stand.
  - Place a waste container (100-mL beaker) on the magnetic stirrer.
  - Use the buret clamp to attach the buret to the ring stand.
  - Position the drop counter over the waste container and attach it to the ring stand using the right-angle clamp.
  - Place the conductivity sensor through one of the slots in the drop counter.



**Note:** Do not connect the drop counter to the data collection system yet.

4.  Rinse the buret with several milliliters of the standardized 0.0200  $\text{H}_2\text{SO}_4$  solution:
- Ensure that the stopcock is closed and rinse the inside of the buret with several milliliters of the standardized  $\text{H}_2\text{SO}_4$  solution.
  - Open the stopcock on the buret and drain the rinse  $\text{H}_2\text{SO}_4$  solution into the waste container.
  - Repeat this process two more times.
5.  Why is it necessary to rinse the buret with the  $\text{H}_2\text{SO}_4$  solution?

If there is any residual water or contaminant in the buret, it will dilute the  $\text{H}_2\text{SO}_4$  solution and change its concentration. Rinsing eliminates any such contamination.

6.  Make sure the stopcock on the buret is in the “off” position and then use a funnel to fill the buret with about 50 mL of the  $\text{H}_2\text{SO}_4$  solution (titrant).
7.  Drain a small amount of the titrant through the drop counter into the waste beaker to remove any air in the tip of the buret.
8.  Why is it important to remove air from the tip of the buret?

Any air trapped in the buret tip is counted as volume of  $\text{H}_2\text{SO}_4$ . If this happens, the amount of titrant used will be inaccurate.

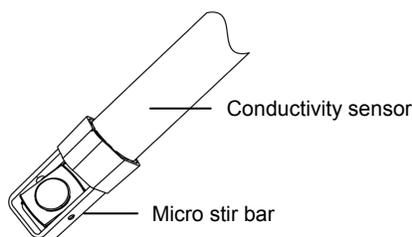
9.  Practice adjusting the stopcock on the buret so that the titrant goes through the drop counter in distinguishable drops that fall at about 1 to 2 drops per second.

**Note:** Good control of the stopcock is important. Each drop should result in a blink of the LED on the drop counter. If the LED is continuously lit, you have opened the stopcock too far and you will have to start over.

10.  Why will it be necessary to start your titration over again if you accidentally allow the titrant to stream out of the stopcock instead of emerging by drops?

The drop counter counts distinct drops. If the drops are not sufficiently distinct from one another, the drop counter will not function properly and the fluid volume will not be accurate.

11.  Add the micro stir bar to the end of the conductivity sensor.



12.  Why is it necessary to stir the solution during a titration?

Stirring thoroughly mixes the ions in the solution so that the recorded pH reflects the pH of the entire solution.

13.  Add additional 0.0200 M  $\text{H}_2\text{SO}_4$  to the buret so the solution is above the zero mark. Allow some of the  $\text{H}_2\text{SO}_4$  solution to drip into the waste container until the bottom of the meniscus is lined up with or just below the zero mark and record the initial reading in Table 1.

14.  Remove the waste container.

15.  Use the volumetric pipet to transfer 50.00 mL of the  $\text{Ba}(\text{OH})_2$  solution with the unknown concentration into a 100-mL beaker.

16.  Add enough deionized water to the beaker so the tip of the conductivity sensor is covered with solution.

17.  Tap the sensor a few times to make sure any trapped air is expelled from the sensor.

18.  Turn on the magnetic stirrer at a slow and steady rate.

19.  Connect the drop counter to the data collection system.  $\diamond^{(2.2)}$

20.  Display the Conductivity on the y-axis of a graph and Drop Count on the x-axis.  $\diamond^{(7.1.1)}$

## Lab 26: Conductometric Titration

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### Collect Data

21.  Clean the lens of the drop counter inside the opening the drops go through with water and a cotton swab or tissue.

22.  Start recording data.  $\diamond^{(6.2)}$

23.  Adjust the scale of the graph.  $\diamond^{(7.1.2)}$

24.  Turn the buret stopcock carefully, allowing the titrant to drip slowly (1 to 2 drops per second) into the solution.

25.  What do you expect to happen in the beaker during the titration?

There will be precipitation since  $\text{BaSO}_4$  is not water soluble.

26.  Continue data collection until the curve passes its minimum point by about 3 mL.

27.  How can you recognize the equivalence point?

The conductivity should be at its minimum as all the ions from the reactants are removed at that point.

28.  At the minimum, is the conductivity at 0? Explain your answer.

The conductivity will not be zero, since not all the ions are removed from the solution

29.  Stop recording data.  $\diamond^{(6.2)}$

30.  In Table 1, record the final drop count and the final reading of the titrant in the buret to a precision of 0.01 mL.

31.  Calculate the volume of titrant (final reading minus initial reading) and record this value in Table 1.

Table 1: Titration data for drop counter calibration

Titration Information	Trial 1	Trial 2
Initial reading of $\text{H}_2\text{SO}_4$ solution on the buret (mL)	0.00	0.00
Final reading of $\text{H}_2\text{SO}_4$ solution on the buret (mL)	21.10	22.80
Volume of titrant (mL)	21.10	22.80
Final drop count	441	482

32.  Calibrate the drop counter.  $\diamond^{(3.4)}$

- 33.**  On the graph, set the horizontal axis to the calculated volume. ♦<sup>(7.1.9)</sup>
- 34.**  Print the graph. ♦<sup>(11.2)</sup>
- 35.**  Find the volume of  $\text{H}_2\text{SO}_4$  solution (to a precision of 0.01 mL) used to reach the equivalence point. ♦<sup>(9.1)</sup> In Table 2, record the volume.
- Note:** The lowest point of the curve represents the volume of the  $\text{H}_2\text{SO}_4$  solution that was required to reach the equivalence point.
- 36.**  Refill the buret above the zero mark with the  $\text{H}_2\text{SO}_4$  solution.
- Fill the buret above the zero mark and allow some of the  $\text{H}_2\text{SO}_4$  solution to drip into a waste container until the top of the meniscus is lined up with the zero mark or just below.
  - Record the starting point in Table 1.
- 37.**  Rinse the conductivity sensor tip with deionized water.
- 38.**  Remove the beaker and dispose of its contents according to the teacher's instructions.
- 39.**  Rinse the beaker with distilled water.
- 40.**  Begin the second titration of  $\text{Ba}(\text{OH})_2$  as follows:
- Use the volumetric pipet to transfer 50.00 mL of the  $\text{Ba}(\text{OH})_2$  solution with the unknown concentration into a 100-mL beaker.
  - Add enough deionized water to the beaker so the tip of the conductivity sensor is covered with solution.
  - Tap the sensor a few times to make sure any trapped air is expelled from the sensor.
  - Turn on the magnetic stirrer at a slow and steady rate.
  - Display the Conductivity on the y-axis of a graph and Drop Count on the x-axis. ♦<sup>(7.1.1)</sup>
  - Repeat the steps following the Collect Data subheading to complete the titration.
- 41.**  Save your experiment ♦<sup>(11.1)</sup> and clean up according to your teacher's instructions.

## Data Analysis

- Obtain the concentration of the  $\text{H}_2\text{SO}_4$  solution from your teacher and record it in Table 2.
- Calculate the number of moles of  $\text{H}_2\text{SO}_4$  that was necessary to reach the equivalence point (the stoichiometrically necessary amount to react with the  $\text{Ba}(\text{OH})_2$ ).

For Trial 1:

$$15.08 \text{ mL} \left( \frac{0.0200 \text{ mol H}_2\text{SO}_4}{1000 \text{ mL}} \right) = 3.02 \times 10^{-4} \text{ mol H}_2\text{SO}_4$$

- Calculate the number of moles of  $\text{Ba}(\text{OH})_2$  that was in the beaker.

Since  $\text{H}_2\text{SO}_4$  reacts in a 1:1 stoichiometric ratio with  $\text{Ba}(\text{OH})_2$ , the number of moles of  $\text{Ba}(\text{OH})_2$  is the same as the number of moles of added  $\text{H}_2\text{SO}_4$  up to the equivalence point:  $3.02 \times 10^{-4}$  mol.

- Calculate the concentration of the original  $\text{Ba}(\text{OH})_2$  from the number of moles of  $\text{Ba}(\text{OH})_2$  that was present in the beaker and the volume of the  $\text{Ba}(\text{OH})_2$  solution. Record the results in Table 2.

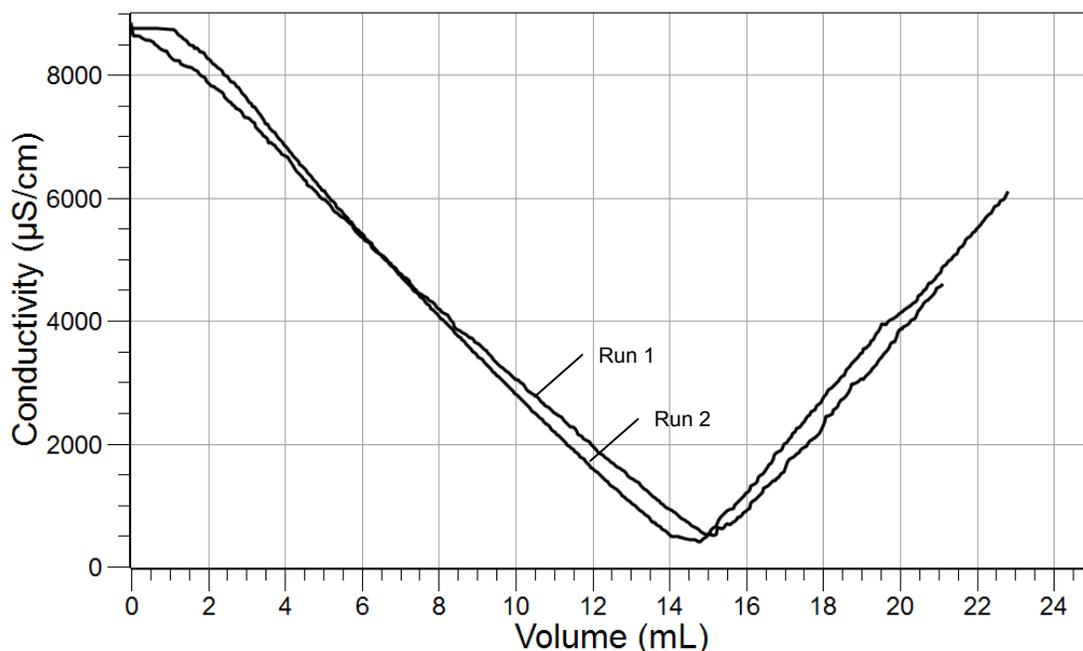
For Trial 1:

$$\left( \frac{3.02 \times 10^{-4} \text{ mol Ba}(\text{OH})_2}{0.05000 \text{ L}} \right) = 6.04 \times 10^{-3} \text{ M Ba}(\text{OH})_2$$

Table 2: Determination of the concentration of the  $\text{Ba}(\text{OH})_2$  solution

	Trial 1	Trial 2
Concentration of standardized $\text{H}_2\text{SO}_4$ solution (M)	0.0200	
Volume of $\text{Ba}(\text{OH})_2$ solution (mL)	50.00	50.00
Volume of $\text{H}_2\text{SO}_4$ to the equivalence point (mL)	15.08	14.76
Concentration of the $\text{Ba}(\text{OH})_2$ solution (M)	$6.04 \times 10^{-3}$	$5.90 \times 10^{-3}$

5.  Sketch or attach your titration curves below, indicating the data run.



### Analysis Questions

1. Is the resulting titration curve what you expected?

Students may mention that, at the equivalence point, the conductivity was not zero.

2. Why would the titration curve be different than you expected?

Even at the equivalence point, there are ions present from the auto-dissociation of water.

### Synthesis Questions

Use available resources to help you answer the following questions.

1. How would the titration curve be different if  $\text{Na}_2\text{SO}_4$  is used instead of  $\text{H}_2\text{SO}_4$ ? (Hint: Check which ions would be removed and which ones would not.)

The  $\text{Na}^+$  and  $\text{OH}^-$  ions would remain in the solution. Therefore, the conductivity would have been significantly higher, even at the equivalence point.

2. What solution would allow you to measure the concentration of an unknown  $\text{AgNO}_3$  solution?

Any strong electrolyte with  $\text{Cl}^-$  ions would suffice:  $\text{Ag}^+(\text{aq}) + \text{Cl}^-(\text{aq}) \rightarrow \text{AgCl}(\text{s})$

## Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

- At the equivalence point in this lab, what does the titrated solution contain?
  - Only water and  $\text{BaSO}_4$
  - Only  $\text{Ba}^{2+}$  and  $\text{OH}^-$  ions
  - Only  $\text{H}^+$  and  $\text{SO}_4^{2-}$  ions
  - $\text{Ba}^{2+}$ ,  $\text{OH}^-$ ,  $\text{H}^+$ , and  $\text{SO}_4^{2-}$  ions
- Which statement is *not* true?
  - The conductivity of solutions depends on the concentration of ions.
  - The conductivity of solutions depends on the nature of the ions that are present.
  - The conductivity of solutions depends on the concentration of undissociated molecules present in the solution.**
  - The conductivity is lowest at the equivalence point.
- What does doubling the volume of a solution by adding water do?
  - It doubles the ion concentration and therefore increases the conductivity.
  - It increases the conductivity since the ion concentration is lowered.
  - It decreases the conductivity since the ion concentration is increased
  - It lowers the conductivity since the ion concentration is lowered.**

## Extended Inquiry Suggestions

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Consider filtering, drying, and measuring the mass of the resulting  $\text{BaSO}_4$  to see how well the actual amount matches the proposed amount. The proposed amount can be calculated from the actual concentration of the  $\text{Ba}(\text{OH})_2$  solution.

## Lab 27: Identifying an Unknown Metal

### Objectives

Students identify an unknown metal by applying and relating the Ideal Gas Law, the three possible oxidation numbers of metals, and the periodic table of elements.

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Employing a data collection system to monitor temperature and pressure in order to calculate the number of moles of hydrogen produced by a chemical reaction
- ◆ Using an electronic balance
- ◆ Applying the calculated number of moles of hydrogen gas produced to determine three possible atomic weights of the starting material.
- ◆ Using the calculated atomic weights and the periodic table to identify the unknown metal

### Time Requirement

◆ Preparation time	15 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

#### *For each student or group:*

- |   |  |
|---|--|
| ◆ Data collection system  | ◆ Beaker, 1500-mL                                  |
| ◆ Absolute pressure sensor with quick-release connectors and plastic tubing | ◆ Balance (1 per class)                            |
| ◆ Stainless steel temperature sensor  | ◆ Rubber stopper with one hole                     |
| ◆ Graduated cylinder, 10-mL or 25-mL  | ◆ 3 M Hydrogen chloride (HCl), 100 mL <sup>1</sup> |
| ◆ Graduated cylinder, 250-mL  | ◆ Unknown metal (3 pieces), 0.2 g <sup>2</sup>     |
| ◆ Erlenmeyer flask, 250-mL  | ◆ Electrical tape, roll                            |

<sup>1</sup> To prepare 3.0 M HCl, refer to the Lab Preparation section.

<sup>2</sup> Use magnesium ribbon as the unknown metal for this activity.

## **Concepts Students Should Already Know**

---

Students should be familiar with the following concepts:

- ◆ Definition of an ideal gas
- ◆ Ideal Gas Law
- ◆ Standard temperature and pressure (STP) condition
- ◆ Avogadro's Law
- ◆ Dalton's Law
- ◆ Chemical formula
- ◆ Balancing chemical equations
- ◆ Stoichiometric calculations

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 3: Determine the Molar Mass of a Volatile Liquid
- ◆ Lab 5: Molar Volume of a Gas
- ◆ Lab 29: Exploring Gas Laws

## **Using Your Data Collection System**

---

Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a temperature sensor and a pressure sensor to your data collection system ◆<sup>( 2.2)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>

## Background

Metals that have a more negative reduction potential than  $H^+$  ions can reduce  $H^+$  ions to  $H_2$ :



where “Me” refers to a metal.

A sample of the unknown metal can be reacted with an acid and the hydrogen formed can be collected. The amount of hydrogen collected can be calculated from its pressure, volume, and temperature. From the amount of hydrogen and the mass of the unknown metal sample, an approximation of the atomic weight of the metal can be calculated based on all three stoichiometric possibilities.

## Pre-Lab Activity

### Setting the stage for the activity

To identify an unknown metal reacted with an acid, the released  $H_2$  gas is first captured in a closed container with known volume and known temperature. The Ideal Gas Law can be used to calculate the number of moles of hydrogen:

$$n = \frac{pV}{RT}$$

where

$n$  = number of moles of hydrogen released in the reaction (mol)

$p$  = partial pressure of hydrogen in the container {Pa or  $N/m^2$ }

$T$  = temperature of the hydrogen gas (K)

$R$  = universal gas constant ( $J \text{ mol}^{-1} \text{ K}^{-1}$ )

From the number of moles of hydrogen released and applying each of the possible three stoichiometric ratios, three atomic weights can be calculated. Based on the three calculations and the physical properties of the unknown, a match can be found in the periodic table.

Knowing the amount of hydrogen gas produced, the number of moles of the metal can be calculated for the three stoichiometric possibilities:

$$n \text{ mol } H_2 \left( \frac{x \text{ mol Me}}{y \text{ mol } H_2} \right) = \left( n \frac{x}{y} \right) \text{ mol Me}$$

where  $n$  = number of moles of hydrogen collected and the three possibilities for  $x$  and  $y$  are shown in Table 1.

Table 1: Stoichiometric ratios, from Equations 1, 2, and 3, of metal ions to hydrogen gas

Possibility	$x$	$y$
1	2	1
2	1	1
3	2	3

The atomic weight of the unknown metal, then, is

$$AW_{\text{Me}} = \frac{m}{n \frac{x}{y}} \quad (4)$$

where

$m$  = mass of the unknown metal sample (g)

$AW_{\text{Me}}$  = atomic weight of the unknown metal (g/mol)

$n$  = number of moles of hydrogen collected

$x$  = assumed stoichiometric coefficient for the unknown metal

$y$  = assumed stoichiometric coefficient for hydrogen gas

Along with the three calculations, the apparent physical and chemical properties, such as color and reactivity with air, can also help to identify the metal.

### Example calculation to try

Pennies used to be made of copper; currently however, pennies are made from a grey metal with a shiny, very thin, copper coating. We filed 0.340 g of a penny into a measuring dish and transferred it into a 250-mL Erlenmeyer flask. For the purpose of this experiment, we ignore the very thin copper coating.

Twenty milliliters of 3 M HCl solution was transferred into the Erlenmeyer flask. The flask was closed quickly with a two-holed rubber stopper containing a temperature sensor and a hose connected to a pressure sensor. The final pressure in the flask was 154,435 Pa. The atmospheric pressure in the lab was 101.0 kPa and the temperature of the gas was 298 K.

The volume of the Erlenmeyer flask was determined by filling the empty flask with water up to the rubber stopper and was found to be 261 mL. The number of moles of  $\text{H}_2$  was:

$$n = \frac{pV}{RT}$$

$$n = \frac{\left(154,435 \frac{\text{N}}{\text{m}^2} - 101,000 \frac{\text{N}}{\text{m}^2}\right) \left[ \left(2.61 \times 10^{-4} \text{ m}^3\right) - \left(2.00 \times 10^{-5} \text{ m}^3\right) \right]}{\left(8.314 \frac{\text{Nm}}{\text{mol K}}\right) (298 \text{ K})} = 5.20 \times 10^{-3} \text{ mol H}_2$$

Using Equation 4 and applying the stoichiometric ratio of Possibility 2 (substituting  $x = 1$  and  $y = 1$ ) the atomic weight will be

$$AW_{\text{Me}} = \frac{0.340 \text{ g}}{(5.20 \times 10^{-3} \text{ mol H}_2) \times \left(\frac{1 \text{ mol Me}}{1 \text{ mol H}_2}\right)} = 65.4 \text{ g/mol}$$

Repeating the above calculation for the other two possibilities for  $x$  and  $y$  results in Table 2.

Table 2: Calculated atomic weight for each possible stoichiometric ratio

Possibility	$x$	$y$	AW(g/mol)	Possible Oxidation Number of Me
1	2	1	32.8	+1
2	1	1	65.5	+2
3	2	3	98.1	+3

The atomic weight of sulfur is 32 g/mol and the next element in the periodic table, chlorine, is 35.5 g/mol; neither of these are metals, so the results using Possibility 1 can be omitted. The element corresponding to the atomic weight resulting from applying the stoichiometric considerations of Possibility 2 would be zinc, a feasible option since zinc combines as  $\text{Zn}^{2+}$  in compounds.

For Possibility 3, the closest elements are molybdenum, 96.0 g/mol, technetium 97.9 g/mol, and ruthenium, 101.07 g/mol. All three are rather uncommon, and not appropriate for coins. None is electronegative enough to reduce  $\text{H}^+$  ions. Technetium is also radioactive.

Therefore, we concluded that the penny is made from zinc. The fact that zinc is cheap and commonly available also suggests the likelihood of its use in coins.

**1. Why do you have to subtract the atmospheric pressure from the final pressure to obtain the pressure of the formed hydrogen gas?**

We do this because the final pressure includes the atmospheric pressure and we need the partial pressure of hydrogen for the calculations.

**2. Would it matter if the volume of the Erlenmeyer flask used in the experiment had been 250 mL?**

No, because the number of moles of hydrogen depends only on the number of moles of unknown metal.

**3. Why did we subtract the volume of the HCl solution from the volume of the flask?**

The gas cannot occupy the portion of the flask where the HCl solution is; the available volume for the gas molecules is that much less.

## Lab Preparation

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These are the materials and equipment to set up prior to the lab:

1. **3 M HCl:** Dilute concentrated hydrochloric acid solution in a 1:4 ratio with distilled water. The exact concentration is not critical in this experiment.

## Safety

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Add these important safety precautions to your normal laboratory procedures:

- ◆ If you get hydrochloric acid on your skin, wash it off with large amounts of water.
- ◆ Use goggles and rubber gloves for this experiment.
- ◆ Handle the Erlenmeyer flask very carefully when it is pressurized. A knock on the glass can cause it to crack. Because of the pressure, a slight explosion can occur.

## Sequencing Challenge

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The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

1	2	5	4	3
Obtain and measure the mass of a small piece (about 0.2 g) of unidentified metal.	Put the metal into an Erlenmeyer flask and add hydrogen chloride to the flask with the metal.	Calculate the number of moles of hydrogen created. Use that to determine the atomic weight of the metal. Identify the metal.	Measure the temperature of the water bath. After the reaction, record the temperature, pressure and volume.	Immediately plug the flask with a stopper connected to a pressure sensor. Keep the flask in a water bath.

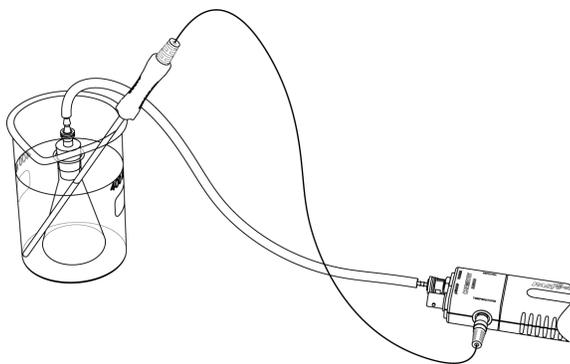
## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "◆" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

- Start a new experiment on the data collection system. ◆<sup>(1.2)</sup>
- Connect a stainless steel temperature sensor and a pressure sensor to the data collection system. ◆<sup>(2.2)</sup>
- Place the barbed connector tightly into the rubber stopper. Connect the barbed connector to the pressure port of the sensor with a piece of tubing.



- Wrap the Erlenmeyer flask with 10 to 15 rounds of electric tape. This is a preventive measure in case the flask cracks. The tape keeps the glass pieces together.
- Mount the 250-mL Erlenmeyer flask in a water bath in a 1500-mL beaker. The water should cover as much of the flask as possible.
- Place the temperature sensor in the water bath.
- Why is it necessary to immerse the flask as much as possible in the water bath?

It is necessary because we need to know the temperature of the gas in the flask. If the flask is not completely immersed, the temperature of the gas will not be the same as the temperature of the water bath and we will not know what the temperature of the gas is.

**Collect Data**

8.  Perform following the steps. Repeat this part of the procedure three times.
- a. Measure between 0.150 g and 0.180 g of the unknown metal to the nearest milligram. Record the mass of the sample in Table 3.
  - b. Using a graduated cylinder, measure 20.0 mL of 3.0 M HCl solution and transfer it into the 250-mL Erlenmeyer flask.
  - c. Start data recording.  $\diamond^{(6.2)}$
  - d. Record the initial pressure in Table 3.
  - e. What does the pressure reading on the sensor represent at this point?

The pressure sensor shows the atmospheric pressure of air.

- f. Drop the measured piece of unknown metal into the flask and immediately insert the rubber stopper airtight.

**Important:** Make sure that the stopper is sitting firmly as pressure will build in the flask and a loose stopper may pop out. If that happens, you will need to repeat the experiment.

- g. Continue to monitor the pressure. Once the hissing in the flask is over and the pressure has leveled off, record the final pressure and temperature readings in Table 3.
- h. Stop data recording.  $\diamond^{(6.2)}$  You do not need to save your data.
- i. What does the final pressure reading represent? (Hint: What components contribute to the final pressure?)

The final pressure reading represents the atmospheric pressure plus the pressure from the hydrogen gas produced.

- j. Remove the stopper carefully and dispose of the spent acid solution properly.
  - k. Repeat the experiment for a total of three times.
9.  Fill the Erlenmeyer flask to the top with water and, over a sink or waste container, insert the stopper. Using the 250-mL graduated cylinder, measure the volume of the water to obtain the volume of the flask and record the value in Table 3.

## Data Analysis

1.  Calculate the number of moles of H<sub>2</sub> for each trial. Record your answers in Table 3.

For Trial 1:

$$n = \frac{pV}{RT}$$

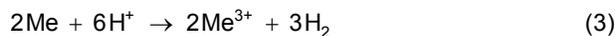
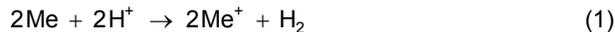
$$n = \frac{\left(44,000 \frac{\text{N}}{\text{m}^2}\right) \left(2.42 \times 10^{-4} \text{ m}^3\right)}{\left(8.314 \frac{\text{Nm}}{\text{molK}}\right) (302 \text{ K})} = 4.24 \times 10^{-3} \text{ mol H}_2$$

Table 3: Number of moles  $n$  of H<sub>2</sub> calculated from the measured values

	Trial 1	Trial 2	Trial 3
Mass of unknown metal (g)	0.106	0.104	0.106
<b>Pressure</b>			
Initial pressure (kPa)	102	102	102
Initial pressure (Pa or N/m <sup>3</sup> )	102000	102000	102000
Final pressure (kPa)	146	147	149
Final pressure (Pa or N/m <sup>3</sup> )	146000	147000	149000
Pressure of H <sub>2</sub> (Pa or N/m <sup>3</sup> )	44000	45000	47000
<b>Temperature</b>			
Mean temperature of water bath (K)	302	302	302.9
<b>Volume</b>			
Volume of flask (mL)	262		
Volume of flask (m <sup>3</sup> )	$2.62 \times 10^{-4}$		
Volume H <sub>2</sub> can occupy (mL)	242		
Volume H <sub>2</sub> can occupy (m <sup>3</sup> )	$2.42 \times 10^{-4}$		
<b>Calculated Number of Moles of H<sub>2</sub></b>			
Calculated number of moles of H <sub>2</sub> (mol)	$4.24 \times 10^{-3}$	$4.34 \times 10^{-3}$	$4.52 \times 10^{-3}$

## Lab 27: Identifying an Unknown Metal

2.  Using the mass of the unknown metal in Table 3, calculate the atomic weight for each run for each stoichiometric possibility and record calculations into Table 4.



$$AW_{\text{Me}} = \frac{m}{n \frac{x}{y}}$$

For Trial 1, Equation 1 (Table 4: Possibility 1):

$$AW_{\text{Me}} = \frac{0.106 \text{ g}}{(4.24 \times 10^{-3} \text{ mol H}_2) \times \left(\frac{2 \text{ mol Me}}{1 \text{ mol H}_2}\right)} = 12.5 \text{ g/mol}$$

For Equation 2 (Table 4: Possibility 2):

$$AW_{\text{Me}} = \frac{0.106 \text{ g}}{(4.24 \times 10^{-3} \text{ mol H}_2) \times \left(\frac{2 \text{ mol Me}}{1 \text{ mol H}_2}\right)} = 25.0 \text{ g/mol}$$

For Equation 3 (Table 4: Possibility 3):

$$AW_{\text{Me}} = \frac{0.106 \text{ g}}{(4.24 \times 10^{-3} \text{ mol H}_2) \times \left(\frac{2 \text{ mol Me}}{3 \text{ mol H}_2}\right)} = 37.5 \text{ g/mol}$$

Table 4: Calculation of atomic weight

Possibility	x	y	AW (g/mol)				Oxidation Number of Me
			Trial 1	Trial 2	Trial 3	Average	
1	2	1	12.5	12.0	11.7	12.1	+1
2	1	1	25.0	24.0	23.5	24.1	+2
3	2	3	37.5	35.9	35.2	36.2	+3

## Analysis Questions

1. What elements can be assigned to Possibility 1 based on the obtained atomic weight? Argue if it is or is not a feasible option.

The atomic weight of 12.1 g/mol falls into the non-metal section between boron and carbon, which makes this an unlikely option.

**2. What elements can be assigned to Possibility 2 based on the obtained atomic weight? Argue if it is or is not a feasible option.**

The atomic weight of 24.1 g/mol suggests magnesium, which is indeed feasible since it forms compounds with a +2 oxidation state.

**3. What elements can be assigned to Possibility 3 based on the obtained atomic weight? Argue if it is or is not a feasible option.**

The atomic weight of 35.2 g/mol suggests chlorine, a non-metal. Even if we allow for some experimental error, chlorine falls on the wrong side of the periodic table. Therefore, Possibility 3 can also be ruled out.

**Synthesis Questions**

Use available resources to help you answer the following questions.

**1. Identify a common metal that usually has a +3 oxidation state and explain why you didn't consider it in Possibility 3?**

Aluminum has a +3 oxidation state. However, it has the atomic weight of 27.0 g/mol—too much less than the atomic weight determined for Possibility 3: 35.2 g/mol to be considered.

**2. If your unknown metal was zinc, would you have generated more or less hydrogen gas than the unknown metal you had (assuming you measured the same amount of the sample)?**

Zinc has a substantially higher atomic weight than magnesium does, so the same mass of zinc would have been fewer moles. Fewer moles of zinc would have generated less hydrogen gas.

**Multiple Choice Questions**

Select the best answer or completion to each of the questions or incomplete statements below.

**1. There were three options for identifying the unknown metal because:**

- A.** The unknown can be any metal.
- B.** There are three possible combinations of stoichiometry by which the unknown metal can react with the  $\text{H}^+$  ions.
- C.** The experiment could have been performed three different ways.
- D.** Metals can have three different oxidation states: +1, +2, and +3.

**2. The reaction between the unknown metal and HCl was highly exothermic, resulting in a warm reaction mixture. Did this temperature change introduce an experimental error?**

- A.** Yes, but not too significant and we neglected it.
- B.** Yes, since higher temperature would cause a higher pressure reading.
- C.** No, as long as equilibrium was established between the gas and the water bath.
- D.** No, temperature doesn't affect pressure

## Lab 27: Identifying an Unknown Metal

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- 3.** Would we have introduced an error if we used 50 mL of the HCl solution instead of 20 mL?
- A.** Yes, a greater volume of HCl would have resulted in higher pressure.
  - B.** No, it would not have introduced an error since the number of moles of hydrogen generated would have been the same.
  - C.** Possibly we would have introduced an error as there may not have been enough HCl solution.

### Extended Inquiry Suggestions

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Have students carry out the experiment presented as the example in the Pre-Lab Activity section. This experiment reinforces the metal activity series, as  $H^+$  can oxidize zinc but not copper. Old pennies are made from copper only; therefore, those pennies will not dissolve in HCl. This provides an excellent topic for discussion.

## Lab 28: Molecular Interaction in Ethanol and Acetone

### Objectives

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Students determine and relate the heat of vaporization of substances to the interactions between molecules.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Studying the molecular interaction of ethanol and of acetone by monitoring their vapor pressure as a function of temperature.
- ◆ Using pressure and temperature sensors

### Time Requirement

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◆ Preparation time	10 minutes
◆ Pre-lab discussion and activity	10 minutes
◆ Lab activity	50 minutes

### Materials and Equipment

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*For each student or group:*

- ◆ Data collection system
- ◆ Stainless steel temperature sensor
- ◆ Absolute pressure sensor with quick-release connectors and plastic tubing
- ◆ Sensor extension cable
- ◆ Beaker, 1500-mL
- ◆ Beaker, 50-mL
- ◆ Erlenmeyer flask, 250-mL
- ◆ Graduated cylinder, 50-mL
- ◆ Hot plate with magnetic stirrer and stirring bar
- ◆ Clamp, utility
- ◆ Ring stand
- ◆ 100% Ethanol (C<sub>2</sub>H<sub>5</sub>OH), 50 mL
- ◆ Acetone ((CH<sub>3</sub>)<sub>2</sub>CO), 50 mL
- ◆ Rubber stopper, 2-hole
- ◆ Glycerin, 2 drops
- ◆ Water, 1200 mL

## **Concepts Students Should Already Know**

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Students should be familiar with the following concepts:

- ◆ Molar volume of a gas
- ◆ Molar mass
- ◆ Gas laws
- ◆ Intermolecular interactions

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 3: Determine the Molar Mass of a Volatile Liquid
- ◆ Lab 5: Molar Volume of a Gas
- ◆ Lab 27: Identifying an Unknown Metal
- ◆ Lab 29: Exploring Gas Laws

## **Using Your Data Collection System**

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a temperature sensor and a pressure sensor to your data collection system ◆<sup>(2.2)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Displaying data on a graph ◆<sup>(7.1.1)</sup>
- ◆ Displaying two data runs on a graph ◆<sup>(7.1.3)</sup>
- ◆ Creating calculated data ◆<sup>(10.3)</sup>
- ◆ Printing ◆<sup>(11.2)</sup>

## Background

Many of the physical properties of substances and solutions are determined by the nature of the interactions between molecules. There are three fundamental interactions which collectively are known as “van der Waals” interactions: dipole-dipole (DD), ion-dipole (ID), and London-type dispersion forces (LD).

The DD forces exist between two dipole molecules—for example, between the water molecules and the polar ethanol molecule. A special type of DD interaction is the hydrogen bond which exists between a partially negatively-charged atom (usually oxygen or nitrogen) and a hydrogen atom which is attached to an atom with high electronegativity. Hydrogen bonds are relatively strong; for example, hydrogen bonding is responsible for the very high boiling point of water.

The ion-dipole forces exist between ions and polar molecules, such as  $\text{Na}^+$  ions and water molecules in a NaCl solution. The LD forces are rather weak and based on temporary, induced polarity between two otherwise non-polar molecules— $\text{CCl}_4$  and  $\text{I}_2$  molecules, for example.

The strength of the bonds between molecules determines the melting point, boiling point, and vapor pressure of a substance at a given temperature. Greater bond strength results in higher melting and boiling points as more thermal energy is needed to break the bonds. Also, greater strength between molecules results in lower vapor pressure at a given temperature as fewer molecules will possess the necessary kinetic energy to escape into the gas phase.

Another factor in molecular interactions is the size of the molecules. Larger molecules need more thermal energy to be able to escape, therefore, the vapor pressure of larger molecules at a given temperature is less. For the same reason, the melting and boiling points of substances with larger molecules are higher. The greater amount of energy needed for molecules to escape from liquid phase to gas phase is reflected by the heat of vaporization.

Heat of vaporization is the energy required for a known amount of substance to escape from the liquid to gas phase. The mathematical relationship between the heat of vaporization and the temperature is given by the Clausius-Clapeyron equation:

$$\ln p = -\frac{\Delta H_{\text{vap}}}{R} \frac{1}{T} + C \quad (1)$$

where

$p$  = vapor pressure of the substance

$\Delta H_{\text{vap}}$  = heat of vaporization (J/mol)

$T$  = temperature (K)

$C$  = a constant

$R = 8.314 \text{ J}/(\text{mol K})$

Equation 1 shows that a plot of  $\ln p$  versus  $1/T$  should give a straight line with a slope of  $-(\Delta H_{\text{vap}}/R)$ .

## Pre-Lab Activity

### Setting the stage for the activity

In this activity, you will compare the molecular interactions between ethanol molecules and then between acetone molecules. Their formulas and properties are listed in Table 1.

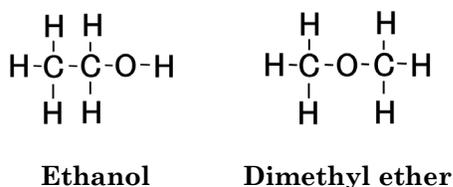
Table 1: Properties of acetone and ethanol

Properties	$\begin{array}{c} \text{O} \\    \\ \text{CH}_3 - \text{C} - \text{CH}_3 \end{array}$ Acetone	$\text{CH}_3 - \text{CH}_2 - \text{OH}$ Ethanol
Possible interactions	London-type of dispersion	Hydrogen bond
Strength of strongest interactions	Weak	Strong
Formula weight (g/mol)	58.0	46.0
Boiling point (°C)	56.5	78.4

The ethanol molecule is much lighter and its boiling point is much higher than the boiling point of acetone because of the existing hydrogen bond between the ethanol molecules.

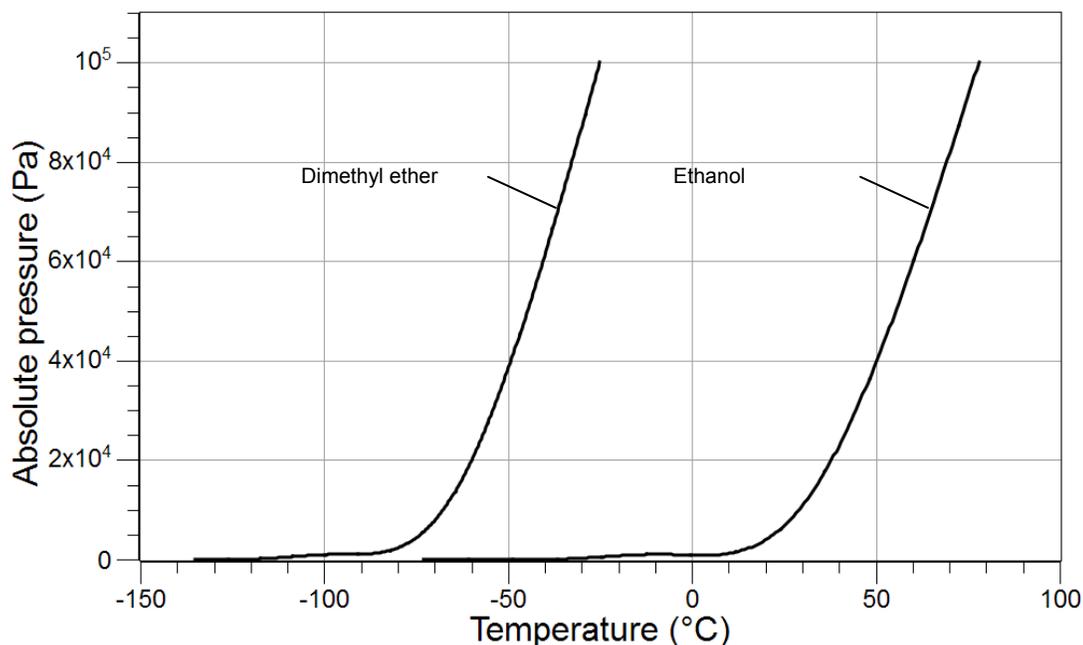
### Example calculation to try

In an experiment, the molecular interactions between ethanol and dimethyl ether were compared. They have the same formula but different molecular structures:

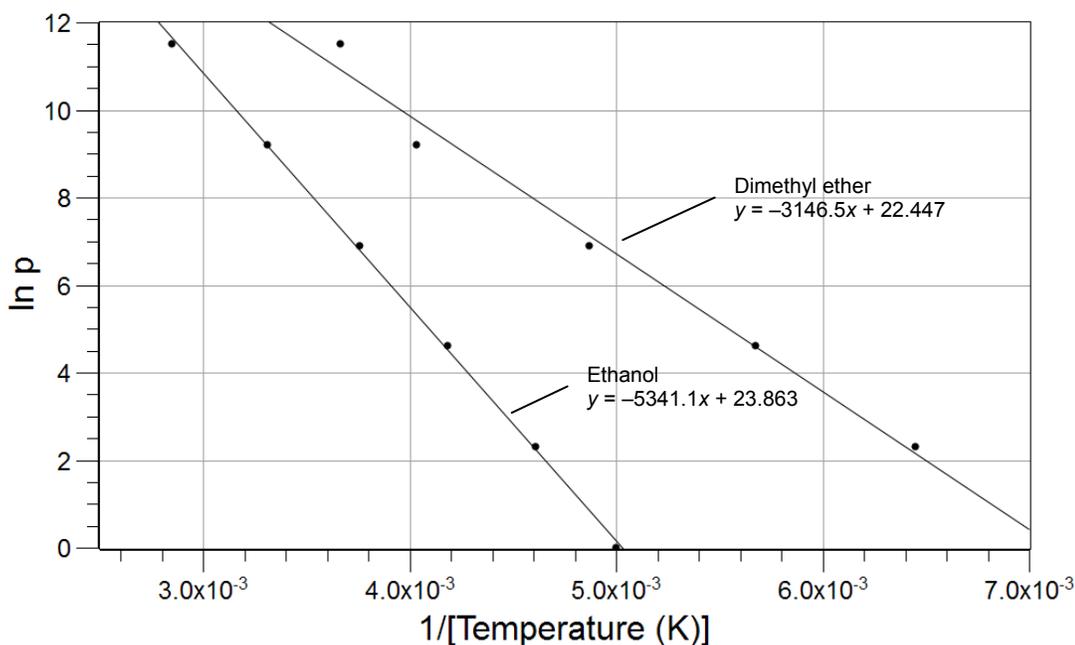


Boiling point:	78.4 °C	-23.6 °C
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Ethanol molecules can form hydrogen bonds between the oxygen and the hydrogen atoms of other ethanol molecules. This explains its relatively high boiling point. Dimethyl ether, on the other hand, can form only weak London-type interactions between molecules, which explains its low boiling point. The following data was obtained as the vapor pressures of these two substances were compared:



As you can see, at any given temperature where both compounds are in the liquid phase more of the dimethyl ether molecules escape to the gas phase, building higher pressure than ethanol molecules, due to the weaker interactions between dimethyl ether molecules. The heat of vaporization of ethanol, therefore, is higher than it is for dimethyl ether. The heat of vaporization can be calculated from the data above with the Clausius-Clapeyron equation:



## Lab 28: Molecular Interaction in Ethanol and Acetone

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Table 2: Determination of the heat of vaporization

	Slope (K)	Heat of Vaporization (J/mol)
Ethanol	-5341.1	44,406
Dimethyl ether	-3146.5	26,160

Calculation of the heat of vaporization for Ethanol from the slope:

$$(\text{slope}) = -\frac{\Delta H_{\text{vap}}}{R}$$

$$\Delta H_{\text{vap}} = -(\text{slope})R = -(5341.1\text{K})\left(8.314\frac{\text{J}}{\text{mol K}}\right) = 44,406\frac{\text{J}}{\text{mol}}$$

The same calculation applies for the dimethyl ether.

The lower heat of vaporization for dimethyl ether demonstrates the weaker intermolecular interactions between the dimethyl ether molecules.

### 1. Explain how the boiling point and heat of vaporization are related.

They both depend on intermolecular interactions. They both increase with the increase in the strength of the intermolecular interaction.

### 2. Explain how the vapor pressure of a liquid is related to its boiling point and its intermolecular interactions.

Stronger intermolecular interactions result in higher boiling points because molecules need higher thermal energy to escape into the gas phase. It also results in fewer molecules in the gas phase, which means that the vapor pressure will be lower.

## Lab Preparation

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Although this activity requires no specific lab preparation, allow 10 minutes to assemble the equipment needed to conduct the lab.

## Safety

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Add these important safety precautions to your normal laboratory procedures:

- ◆ Organic solvents are extremely flammable; have no open flame in the lab during the experiment.
- ◆ Do not inhale the fumes of volatile organic liquids.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

4	2	3	5	1
Collect the data on a graph until the temperature is less than 30 °C and follow the same procedure with the acetone.	Add 50 mL of ethanol to the 250-mL Erlenmeyer flask immersed in the hot water bath.	Once the liquid boils, allow a few minutes boiling, then insert the rubber stopper with the pressure and temperature sensors.	Calculate the heat of vaporization for the ethanol and the acetone.	Connect the sensors to the rubber stopper, set up a hot water bath, and immerse the Erlenmeyer flask securely in it.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "♦" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

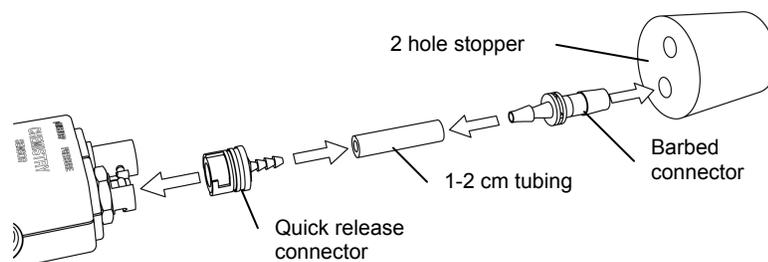
### Set Up

- Set a 1500-mL beaker, 3/4 full with water, on the hot plate with magnetic stirrer and gently place a stirring bar in the beaker.
- Use the ring stand and utility clamp to mount a 250-mL Erlenmeyer flask in the water bath so it is immersed as much as possible without allowing water to enter in the top.
- Turn on the hot plate. Heat the water bath until the temperature is about 80 °C (bubbles start to form on the bottom of the beaker)
- Start a new experiment on the data collection system. ♦<sup>(1,2)</sup>

## Lab 28: Molecular Interaction in Ethanol and Acetone

5.  Place the barbed connector of the absolute pressure sensor tightly into the rubber stopper and connect it to the pressure port of the sensor with a piece of tubing.

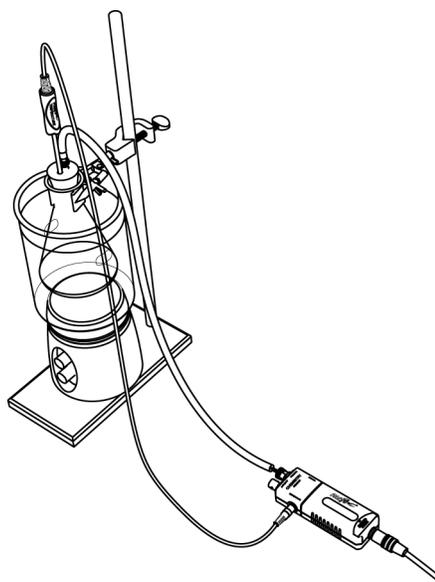
**Note:** If necessary, add a drop of glycerin onto the end of the connector that goes into the hole in the rubber stopper.



6.  Insert the temperature sensor into the other hole in the rubber stopper. If necessary, add a drop of glycerin.
7.  Connect the absolute pressure sensor to the data collection system using a sensor extension cable. <sup>(2.2)</sup>
8.  Display Pressure on the y-axis with Time on the x-axis. <sup>(7.1.1)</sup>
9.  Place the stopper into the Erlenmeyer flask to test for a tight fit.
10.  Remove the stopper.
11.  Transfer 50 mL of ethanol into the Erlenmeyer flask in the water bath.

### Collect Data

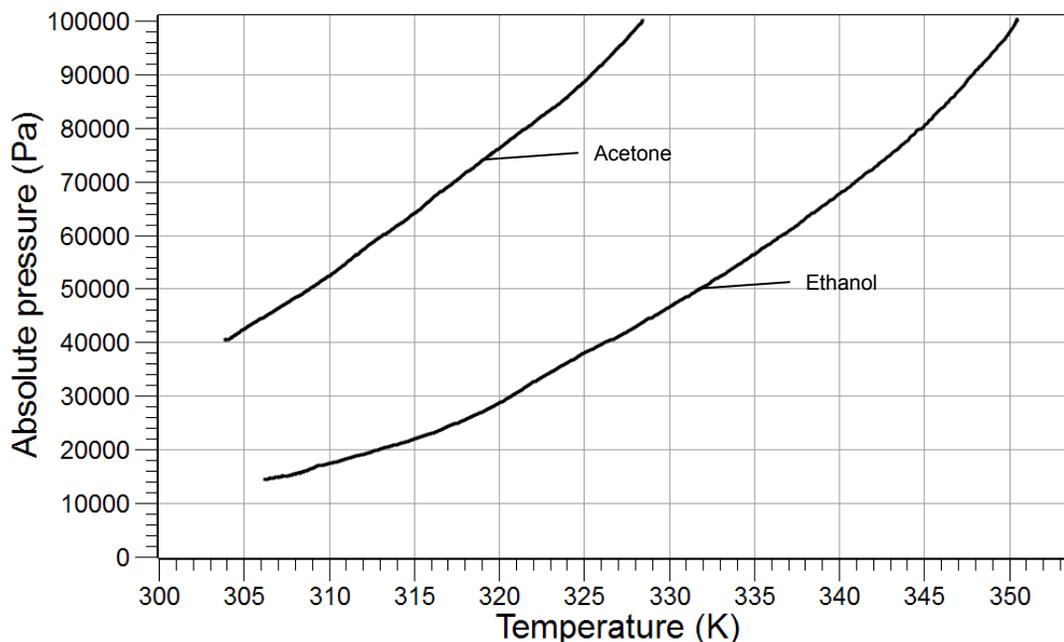
12.  Once the ethanol starts to boil, allow a few minutes for the ethanol fumes to fill the flask.
13.  What is going to fill the flask at this point?  
The air is replaced by ethanol molecules.
14.  Remove the flask from the water bath and immediately insert the stopper tightly.
15.  Start recording data. <sup>(6.2)</sup>
16.  Continue the data collection until the temperature drops to about 30 °C.



17.  Stop recording data.  $\diamond^{(6.2)}$
18.  How do you predict the pressure will change? Explain your answer.  
The pressure will decrease as the temperature decreases; the vapor pressure at lower temperature is lower.
19.  Discard the ethanol as instructed by your teacher and rinse the flask.
20.  Repeat the procedure with a 60 °C water bath and 50 mL of acetone. Allow the flask to cool to about 25 °C.
21.  Why do you think 60 °C is a sufficiently high temperature for the water bath for acetone?  
Acetone has a boiling point at about 56 °C.
22.  Display both data runs.  $\diamond^{(7.1.3)}$
23.  Print the graph.  $\diamond^{(11.2)}$
24.  Save your experiment  $\diamond^{(11.1)}$  and clean up according to your teacher's instructions.

## Data Analysis

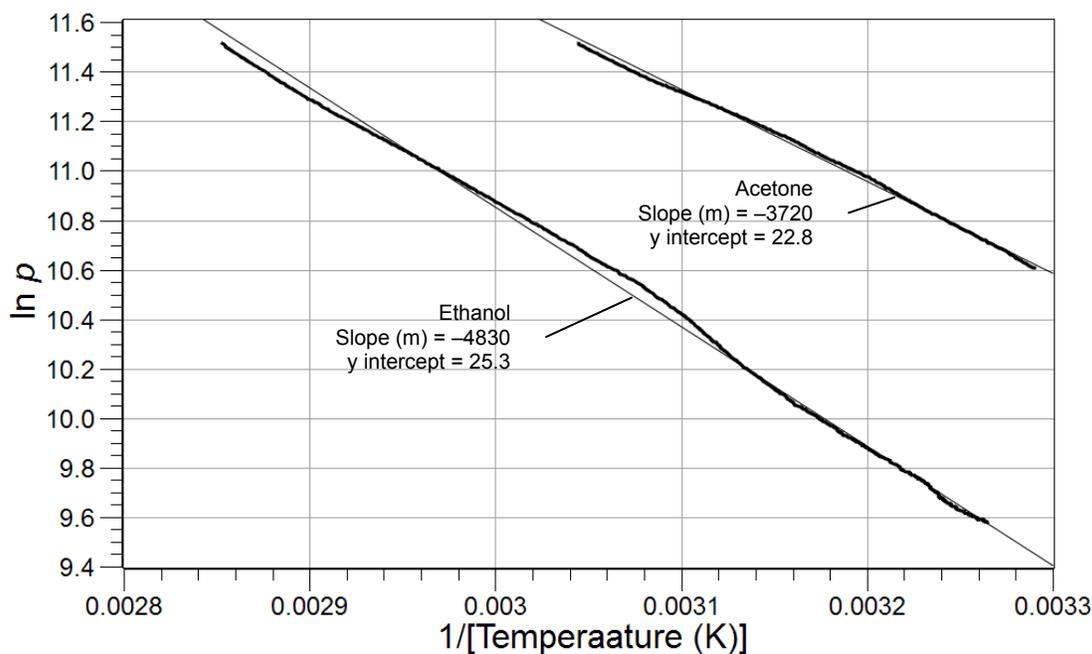
1.  Sketch or attach the Pressure versus Temperature graph below.



2.  Generate the Clausius-Clapeyron plot: Generate two calculated datasets: one for  $\ln p$  and one for  $1/T$ .  $\diamond^{(10.3)}$

## Lab 28: Molecular Interaction in Ethanol and Acetone

3.  Display  $\ln p$  on the y-axis of a graph with  $1/T$  on the x-axis. ♦(7.1.1)
4.  Print the graph. ♦(11.2)
5.  Sketch or attach the graph of  $\ln p$  versus  $1/T$ .



6.  Calculate the heat of vaporization for both substances from the slope of the Clausius-Clapeyron plot.

Using the results for ethanol:

$$(\text{slope}) = -\frac{\Delta H_{\text{vap}}}{R}$$

$$\Delta H_{\text{vap}} = -(\text{slope})R = -(-4830\text{K})(8.314 \frac{\text{J}}{\text{mol K}}) = 4.016 \times 10^4 \frac{\text{J}}{\text{mol}} = 40.16 \frac{\text{kJ}}{\text{mol}}$$

Table 3: Heat of vaporization determined for ethanol and acetone

Substance	Slope (K)	Heat of Vaporization (J/mol)
Ethanol	-4830	$4.016 \times 10^4$
Acetone	-3720	$3.093 \times 10^4$

### Analysis Questions

1. Did it matter how much of the liquid phase was present? Explain your answer!

The actual amount present is not relevant as long as there was enough to establish the vapor pressure.

**2. Compare the curves of the pressure versus temperature graph for ethanol and acetone. Compare the pressures at 35 °C and 50 °C. What conclusions can you draw about the intermolecular interactions between the ethanol and acetone molecules?**

At both temperatures the vapor pressure of acetone is significantly higher, which means that there are more acetone molecules than ethanol molecules in the gas phase. More molecules in the gas phase at the same temperature indicate weaker intermolecular interactions.

**3. Compare the slopes of the Clausius-Clapeyron curves for ethanol and acetone. What conclusion can you draw from the difference between the slopes?**

The line for ethanol is steeper, indicating a higher heat of vaporization.

**4. Compare the calculated heats of vaporization. Do your results support your predictions?**

The significantly larger heat of vaporization for ethanol supports the assumption that the ethanol molecules have much stronger intermolecular interactions than the acetone molecules.

## Synthesis Questions

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Use available resources to help you answer the following questions.

**1. Knowing the heat of vaporization for ethanol and acetone, predict how the heat of fusion (the amount of heat necessary to melt 1 mol of substance) of ethanol and acetone would compare.**

Because of the stronger interactions between the ethanol molecules, the heat of fusion for ethanol would be significantly more than for acetone.

**2. Based on what you have learned, predict the heat of vaporization for ethylene glycol (HO-CH<sub>2</sub>-CH<sub>2</sub>-OH), a common ingredient of car coolant liquids. Explain your predictions!**

Since there are two polar hydrogen atoms and two oxygen atoms in each ethylene glycol molecule, each molecule can form two hydrogen bonds. A higher number of hydrogen bonds will most likely result in a higher heat of vaporization for ethylene glycol. Indeed, the heat of vaporization for ethylene glycol is 63,200 J/mol.

## Multiple Choice Questions

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Select the best answer or completion to each of the questions or incomplete statements below.

**1. High vapor pressure at room temperature is an indication of:**

- A.** High heat of vaporization.
- B.** High heat of fusion.
- C.** Strong intermolecular interactions.
- D.** Weak intermolecular interactions.

- 2. The vapor pressure of acetone was:**
- A. Higher at any temperature than the vapor pressure of ethanol.**
  - B. Lower at any temperature than the vapor pressure of ethanol.**
  - C. Not significant below 30 °C.**
  - D. About the same at any temperature as the vapor pressure of ethanol.**
- 3. The heat of vaporization is greater for ethanol than for dimethyl ether because:**
- A. There are stronger intermolecular interactions between the dimethyl ether molecules.**
  - B. There are weaker intermolecular interactions between the dimethyl ether molecules.**
  - C. The ethanol molecule is larger.**
  - D. The dimethyl ether molecule is larger.**
- 4. The heat of vaporization of methanol (CH<sub>3</sub>-OH) is most likely:**
- A. Less than the heat of vaporization of ethanol because the molecule is smaller.**
  - B. Greater than the heat of vaporization of ethanol because the molecule is smaller.**
  - C. Less than the heat of vaporization of acetone because the molecule is smaller.**
  - D. The same as the heat of vaporization of ethanol because there are hydrogen bonds in methanol just like in ethanol.**

### Extended Inquiry Suggestions

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A modeling activity could be performed by students to "study" the molecular interactions between ethylene glycol molecules. The actual experiment is not easily done since ethylene glycol does not have significant vapor pressure below 100 °C and at higher temperatures safety becomes a concern. Instead, provide students with a set of vapor pressure versus temperature data. The data can be readily generated from the Antoine equation for ethylene glycol:

$$\ln(p) = A - \frac{B}{T} + C \ln(T) + DT^E$$

where

$$A = 84.09$$

$$B = 10411$$

$$C = -8.1976$$

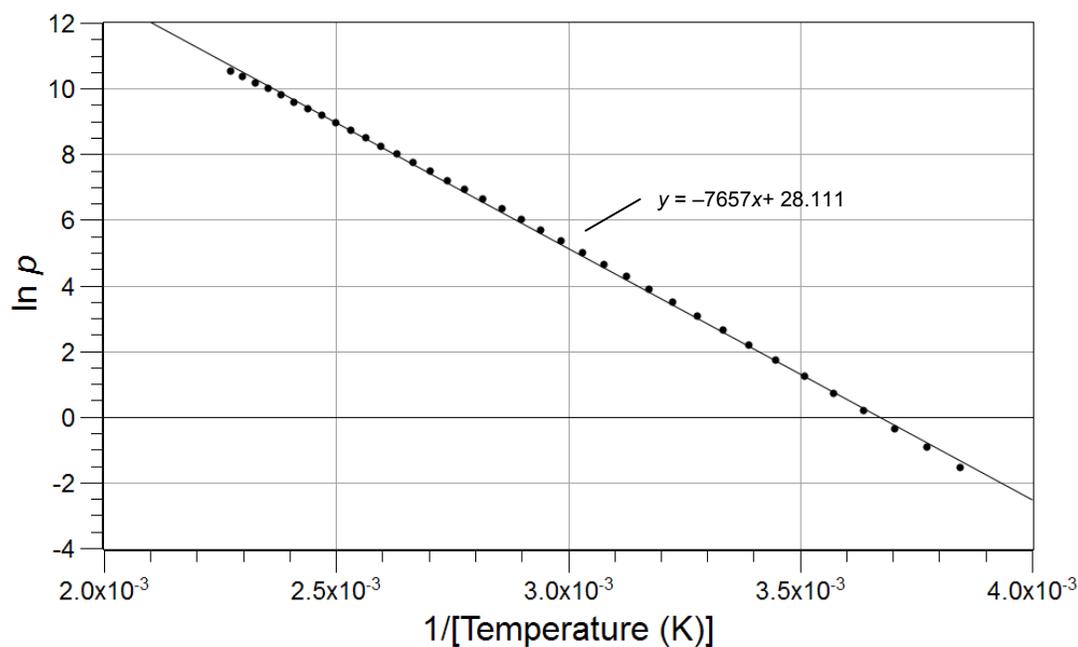
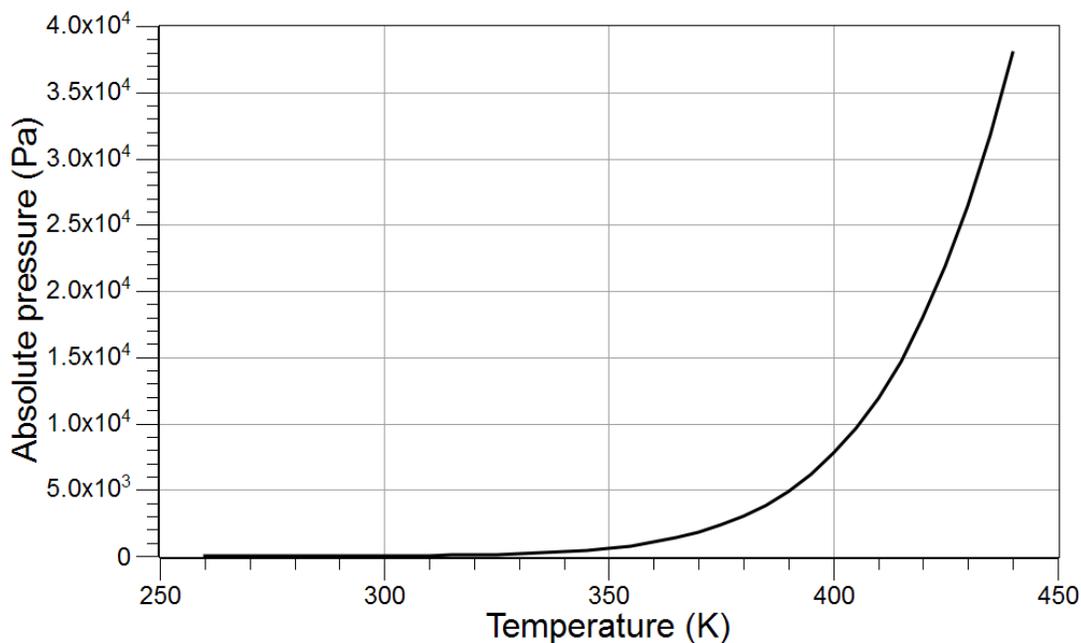
$$D = 1.6536 \times 10^{-18}$$

$$E = 6$$

Please note, no units are provided.

The pressure versus temperature graph and the Clausius-Clapeyron plot can be obtained from the modeled data, shown below, clearly demonstrating a higher heat of vaporization than that of

ethanol. Hold a discussion to relate the high heat of vaporization (higher than water) to the fact that ethylene glycol is used as a major ingredient in various automotive systems for two reasons: high boiling point (heat of vaporization) and low freezing point.



Determination of the heat of vaporization

Substance	Slope (1/K)	Heat of vaporization (J/mol)
Ethylene glycol	-7657.0	63,660



## Lab 29: Exploring Gas Laws

### Objectives

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Students explore the relationship between pressure and volume, and pressure and temperature, for a gas.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Assembling an experimental setup
- ◆ Using pressure and temperature sensors to relate the temperature and pressure of a gas (Gay-Lussac's Law)
- ◆ Using the pressure sensor to relate the pressure and volume of a gas (Boyle's Law)
- ◆ Recording pressure and temperature data

### Time Requirement

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- |                                   |            |
|-----------------------------------|------------|
| ◆ Preparation time                | 15 minutes |
| ◆ Pre-lab discussion and activity | 10 minutes |
| ◆ Lab activity                    | 50 minutes |

### Materials and Equipment

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*For each student or group:*

- |   |  |
|---|--|
| ◆ Data collection system  | ◆ Erlenmeyer flask, 250-mL                     |
| ◆ Absolute pressure sensor with quick-release connectors and plastic tubing | ◆ Syringe, 60-mL                               |
| ◆ Sensor extension cable  | ◆ Hot plate with magnetic stirrer and stir bar |
| ◆ Stainless steel temperature sensor  | ◆ Rubber stopper, 2-hole                       |
| ◆ Ring stand  | ◆ Glycerin, several drops                      |
| ◆ Clamp, utility  | ◆ Electrical tape, roll                        |
| ◆ Beaker, 1500-mL   | ◆ Water, 1200 mL                               |

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Measuring physical quantities
- ◆ Direct and inverse proportionality relationships
- ◆ Interpreting graphs

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 3: Determine the Molar Mass of a Volatile Liquid
- ◆ Lab 5: Molar Volume of a Gas
- ◆ Lab 27: Identifying an Unknown Metal
- ◆ Lab 28: Molecular Interaction in Ethanol and Acetone

## Using Your Data Collection System

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting multiple sensors to your data collection system ◆<sup>(2.2)</sup>
- ◆ Putting the data collection system into manual sampling mode with manually entered data ◆<sup>(5.2.1)</sup>
- ◆ Changing the units of a measurement ◆<sup>(5.3)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Starting a manually sampled new data set ◆<sup>(6.3.1)</sup>
- ◆ Recording a manually sampled data point ◆<sup>(6.3.2)</sup>
- ◆ Stopping a manually sampled set ◆<sup>(6.3.3)</sup>
- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>
- ◆ Adjusting the scale of a graph ◆<sup>(7.1.2)</sup>

- ◆ Creating calculated data ◆<sup>(10.3)</sup>
- ◆ Save your experiment ◆<sup>(11.1)</sup>
- ◆ Printing ◆<sup>(11.2)</sup>

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## Background

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In solids and liquids the atoms or molecules are very close to each other, leaving no room between them. For this reason, solids and liquids cannot be measurably compressed. Gases, on the other hand, have relatively large distances between the atoms or molecules as they bounce into each other and into the walls of their container. This fact allows gases to be compressed.

Gas pressure is related to the frequency gas molecules bounce into surfaces. When the volume of a container of gas is changed, the distance between the walls changes and the amount of time it takes for a particle to get from one wall to another changes. This results in a different number of collisions per second which causes a different pressure. Decreasing the volume, therefore, increases the pressure and vice versa. This relationship was discovered by Robert Boyle in 1661.

If a gas is allowed to expand, increasing the temperature will force the gas to expand, maintaining constant pressure. This relationship was discovered by Jacques Charles in 1789. Also, if the temperature of a gas increases, the average kinetic energy of the molecules increases and results in 1) more energetic collisions between molecules and 2) more energetic collisions between the molecule and the wall of the container, which results in higher pressure if the gas is not allowed to expand. This relationship was discovered by Joseph Gay-Lussac in 1802.

Since temperature is a measure of the average kinetic energy (and therefore the average speed) of gas molecules, a change in temperature will change the time it takes for molecules to move from wall to wall in a container. This implies that if the molecules stopped moving, they would no longer hit the walls and the pressure would be zero. The colder something becomes, the slower the molecules move, thus the temperature at which all motion stops must be the coldest temperature possible. This temperature is called "absolute zero".

In this lab you will study the relationship between the pressure and volume (Boyle's Law) at constant temperature and the relationship between the pressure and temperature (Gay-Lussac's Law) at constant volume.

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## Pre-Lab Activity

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### *Setting the stage for the activity*

In this activity, you will perform experiments to study the properties of gases. In the first experiment, you will relate the pressure to the volume of gases at room temperature. Connecting a syringe to a pressure sensor, you will monitor pressure changes as the volume of the captured gas changes (the plunger is pushed in, decreasing the volume). In the second experiment, you will keep the volume of a gas sample constant and monitor the change in the pressure of the gas as the temperature is increased.

### *Example calculation to try*

## Lab 29: Exploring Gas Laws

### Boyle's Law

We used to use hand-driven pumps to inflate tires on bicycles and cars. An average car tire requires 33.0 psi pressure. The pump we are using has an inner diameter of 1.00 inch and the length of 70.0 cm.

How far from the top of the pump must the piston be pushed in to build enough pressure to force the air into a tire with 33 psi pressure?

We will employ Boyle's Law, which states that the pressure is inversely proportional to the volume while the number of molecules and the temperature are kept constant. For example, reducing the volume by half will double the pressure. This also means that the product of pressure and volume will always be constant:

$$p_1 V_1 = p_2 V_2 = \text{a constant value}$$

The necessary conversions are

$$1 \text{ atm} = 14.5 \text{ psi} = 101,325 \text{ Pa}$$

$$1 \text{ inch} = 2.54 \text{ cm}$$

$$(33.0 \text{ psi}) \left( \frac{1 \text{ atm}}{14.5 \text{ psi}} \right) \left( \frac{101,325 \text{ Pa}}{1 \text{ atm}} \right) = 2.31 \times 10^5 \text{ Pa}$$

The area of the base of the piston is

$$\left( \frac{2.54 \text{ cm}}{2} \right)^2 \left( \frac{1 \text{ m}}{100 \text{ cm}} \right)^2 (3.14) = 5.07 \times 10^{-4} \text{ m}^2$$

The volume of the pump is

$$V_1 = \Delta l A$$

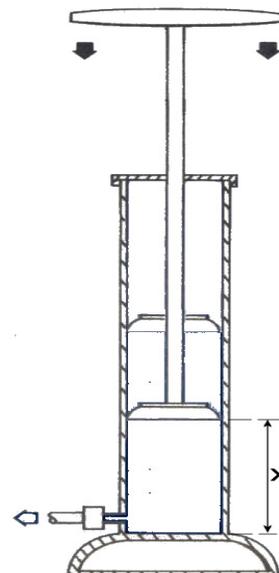
where

$\Delta l$  = displacement of the piston (m)

$A$  = surface area of the piston ( $\text{m}^2$ )

Substituting the appropriate values yields:

$$V_1 = (0.700 \text{ m}) (5.07 \times 10^{-4} \text{ m}^2) = 3.55 \times 10^{-4} \text{ m}^3$$



The initial pressure is the atmospheric pressure ( $p_1 = 1.01 \times 10^5$  Pa). In order to achieve the desired pressure of 33 psi, the final volume should be:

$$p_1 V_1 = p_2 V_2$$

$$V_2 = \frac{p_1 V_1}{p_2}$$

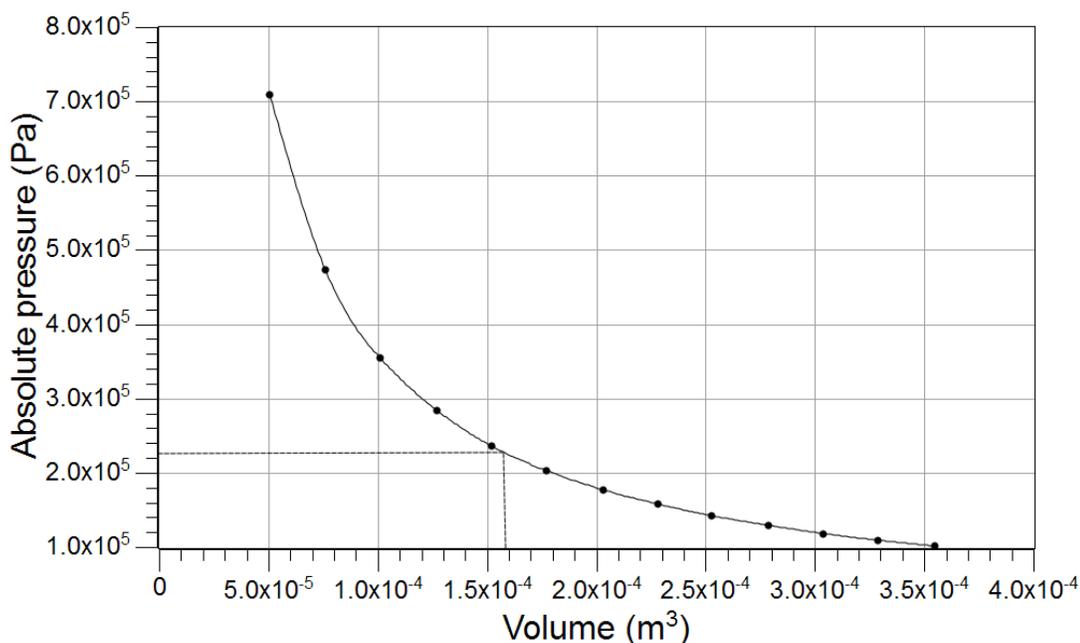
$$V_2 = \left( \frac{(1.01 \times 10^5 \text{ Pa})(3.55 \times 10^{-4} \text{ m}^3)}{(2.31 \times 10^5 \text{ Pa})} \right) = 1.55 \times 10^{-4} \text{ m}^3$$

The distance  $x$  of the piston from the bottom is

$$x = \frac{(1.55 \times 10^{-4} \text{ m}^3)}{(5.07 \times 10^{-4} \text{ m}^2)} = 0.308 \text{ m}$$

Therefore, the pump has to be pushed down until it is at least 0.309 m from the bottom—a little more than halfway down. We can actually push the piston in much further, which is why we can build much higher pressure to drive air into the tire effectively.

If you monitor the increase in pressure as the volume is decreasing, you obtain the following graph:



## Lab 29: Exploring Gas Laws

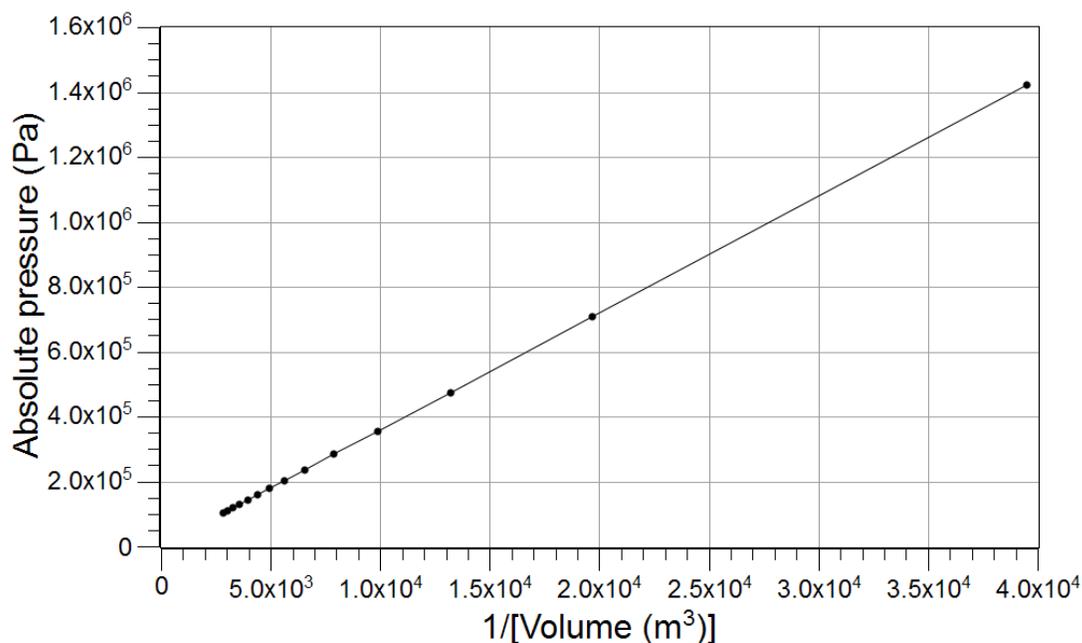
Notice that the pressure necessary to drive air into the tire is marked on the graph with the corresponding volume. The inverse relationship can be shown more clearly if we consider that an inverse relationship means that one quantity is directly proportional to the inverse of the other quantity:

$$pV = \text{constant}$$

$$p = \frac{\text{constant}}{V}$$

$$p \propto \frac{1}{V}$$

Therefore, the plot of  $p$  versus  $1/V$  is linear:



### Gay-Lussac's Law

In another example, a 50-m<sup>3</sup> gas container is pressurized with methane gas at room temperature ( $T_1 = 25\text{ }^\circ\text{C}$ ) to an initial pressure ( $p_1 = 50\text{ atm}$ ). To what value would the safety valve be set in order to release the gas if the temperature rises above a critical value ( $T_2 = 40\text{ }^\circ\text{C}$ )?

To solve this example we have to employ Gay-Lussac's Law. This law states that the pressure of a gas with a constant number of molecules and constant volume is proportional to the temperature:

$$\frac{p_1}{T_1} = \frac{p_2}{T_2}$$

The necessary conversions are

$$T_1 = 25^\circ\text{C} = 298\text{K}$$

$$T_2 = 40^\circ\text{C} = 313\text{K}$$

$$p_1 = 50\text{atm} = 5.07 \times 10^6\text{Pa}$$

The critical pressure will be

$$p_2 = \frac{(5.07 \times 10^6\text{Pa})(313\text{K})}{(298\text{K})} = 5.32 \times 10^6\text{Pa} = 52.5\text{atm}$$

(The conversion to pascals turns out not to have been necessary.)

**1. Explain why the decreasing volume results in higher pressure.**

A decreasing volume increases the frequency of the collisions among the molecules as well as of the collisions between molecules and the wall of the container. In this case, the increased number of collisions between the molecules and the container wall is responsible for the increasing pressure.

**2. Explain why increasing temperature increases the pressure.**

Higher temperature will increase the average kinetic energy which will increase the average speed of molecules. Higher speed results in more energetic and more frequent collisions with the wall and with other molecules, which results in higher pressure.

## Lab Preparation

Although this activity requires no specific lab preparation, allow 10 minutes to assemble the equipment needed to conduct the lab.

## Safety

Add these important safety precautions to your normal laboratory procedures:

- ◆ Organic solvents are extremely flammable; have no open flames in the lab during the experiment.
- ◆ Do not inhale the fumes of volatile organic liquids.

## Sequencing Challenge

The steps below are part of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

3	4	1	5	2
Now set up the dcs to monitor pressure and temperature and set up the apparatus to investigate Gay-Lussac's Law.	After the water bath temperature reaches 80 °C, stop recording data.	Set up the data collection system (dcs) with the absolute pressure sensor and a syringe to investigate Boyle's Law.	From your graphs, relate pressure, volume, temperature, and the number of molecules in a closed system.	Push in the syringe plunger 5 mL at a time. Record the volume and pressure until the pressure exceeds 4 atm.

## Procedure with Inquiry

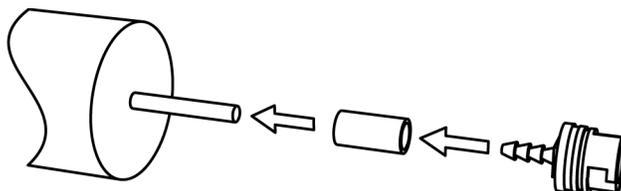
After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "♦" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

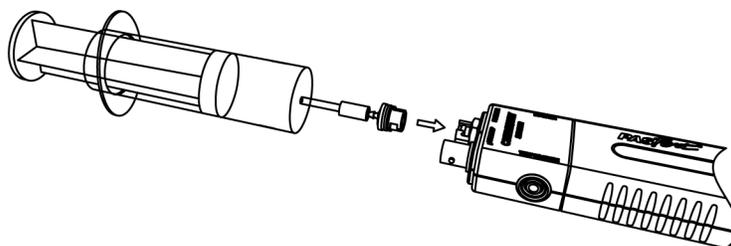
### Part 1 – Boyle's Law

#### Set Up

- Start a new experiment on the data collection system. ♦<sup>(1,2)</sup>
- Connect the absolute pressure sensor to the data collection system using a sensor extension cable. ♦<sup>(2,2)</sup>
- Put a drop of glycerin on the barbed end of a quick-release connector and put that end into one end of a short piece (about 2.5 cm) of plastic tubing that comes with the sensor.
- Put a drop of glycerin on the end of the syringe. Connect the end of the syringe to the other end of the small piece of plastic tubing.



5.  Adjust the plunger so there is 60.0 mL of air in the syringe.
6.  Align the quick-release connector on the end of the plastic tubing with the pressure port of the absolute pressure sensor. Push the connector onto the port, and then turn the connector clockwise until it clicks (about one-eighth turn).

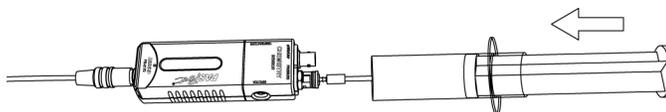


7.  Configure the data collection system to manually collect pressure and volume. Define “volume” as a manually entered data set with units of milliliters.  $\diamond^{(5.2.1)}$
8.  Change the units of the pressure measurement to Pa.  $\diamond^{(5.3)}$
9.  What is the pressure at this time in the syringe?

The pressure is the same as the atmospheric pressure.

### Collect Data

10.  Start a new, manually sampled data set.  $\diamond^{(6.3.1)}$
11.  Before pushing the plunger (set at 60 mL), record the pressure and enter the volume.  $\diamond^{(6.3.2)}$
12.  One student should push the plunger in 5 mL at a time while another student records the pressure and manually enters the volume.  $\diamond^{(6.3.2)}$



13.  Continue to take readings at 5 mL intervals. Stop the data set when the pressure exceeds 4 atm (404 kPa).  $\diamond^{(6.3.3)}$

CAUTION: To minimize the risk of injury or damage to the equipment, avoid over-compressing the air in the syringe.

14.  Display Pressure on the y-axis of a graph and Volume on the x-axis.  $\diamond^{(7.1.1)}$

## Lab 29: Exploring Gas Laws

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15.  Print the graph. ♦<sup>(11.2)</sup>

16.  Save your experiment. ♦<sup>(11.1)</sup>

17.  How does it feel to push the plunger in as the volume decreases?

It becomes increasingly more difficult to push the plunger in as the volume decreases.

18.  Can you push the plunger in all the way? Explain your answer.

No, because the volume of the gas molecules cannot be zero. Mathematically, as the volume approaches zero, the pressure will approach infinity.

### Part 2 – Guy-Lussac's Law

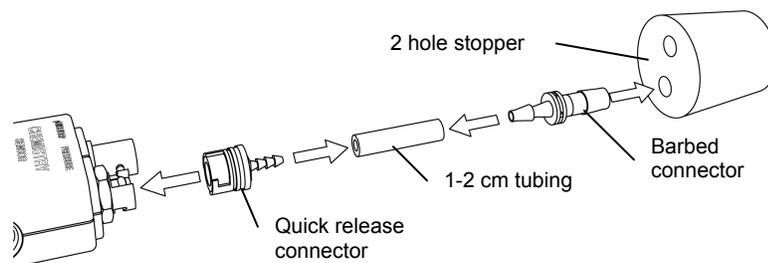
#### Set Up

19.  Start a new experiment on the data collection system. ♦<sup>(1.2)</sup>

20.  Display Pressure on the y-axis and Temperature on the x-axis. ♦<sup>(7.1.1)</sup>

**Note:** Change the units of the temperature measurement to Kelvin. ♦<sup>(5.3)</sup>

21.  Place the barbed connector of the pressure sensor tightly into one hole of the rubber stopper and connect it to the pressure port of the sensor with a piece of tubing. Use a drop of glycerin if necessary.



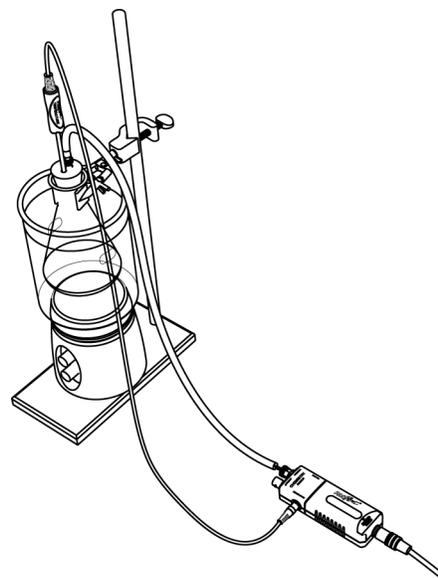
22.  Insert the temperature sensor into the other hole in the rubber stopper. If necessary, add a drop of glycerin. Wrap the flask 15 to 20 times with electric tape if available.

23.  Set a 1500-mL beaker, 3/4 full with water, on the hot plate with magnetic stirrer on the ring stand.

24.  Place the stirring bar in the beaker.
25.  Mount a 250-mL Erlenmeyer flask in the water so that it is covered with water as much as possible.
26.  Why is it essential to immerse the Erlenmeyer flask as far as possible into the water bath?

The temperature of the glass wall of the flask will be the temperature of the gas inside the flask, which is needed for calculations. If the temperature of the wall is not homogenous, we will not know the temperature of the gas inside of the flask.

27.  Place the stopper tightly into the Erlenmeyer flask.
28.  Connect the pressure sensor and the temperature sensor to the data collection system.  $\diamond^{(2.2)}$



### Collect Data

29.  Start recording data.  $\diamond^{(6.2)}$
30.  Turn on the hot plate.
31.  Monitor the pressure as a function of temperature until the water bath temperature reaches 80 °C.
32.  Stop recording data.  $\diamond^{(6.2)}$
33.  Display Pressure on the y-axis of a graph and Temperature on the x-axis.  $\diamond^{(7.1.1)}$
34.  Print the graph.  $\diamond^{(11.2)}$
35.  Save your experiment.  $\diamond^{(11.1)}$
36.  What is the highest temperature you could achieve with this setup? Explain your answer.

The highest temperature would be the boiling point of water, 100 °C.

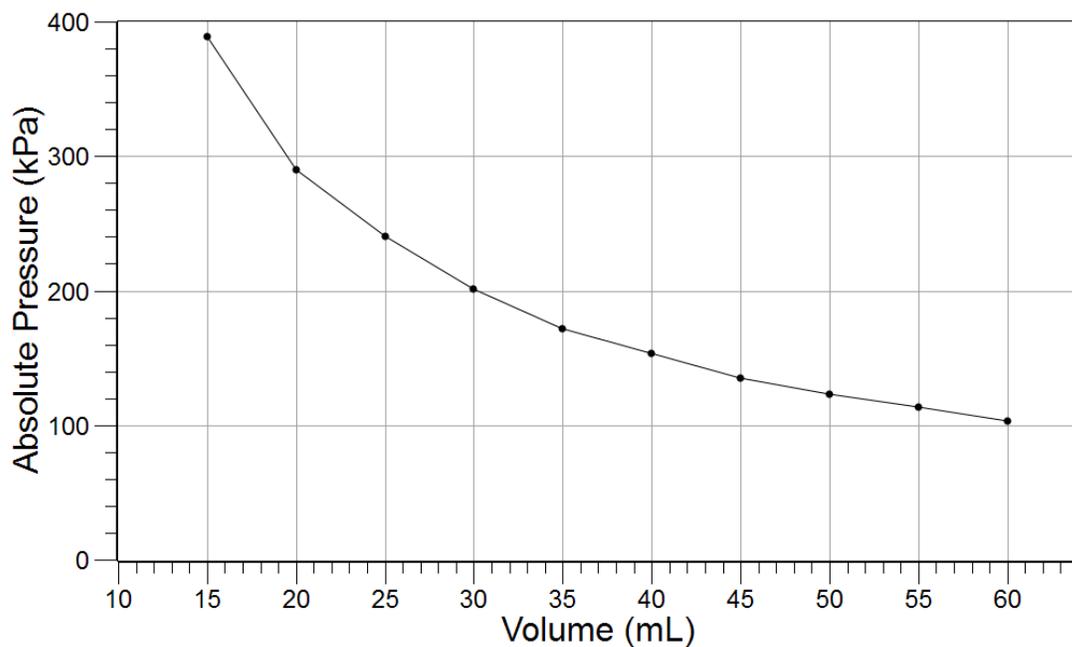
37.  How does the pressure change as the temperature increases?

The pressure increases as the temperature increases.

## Data Analysis

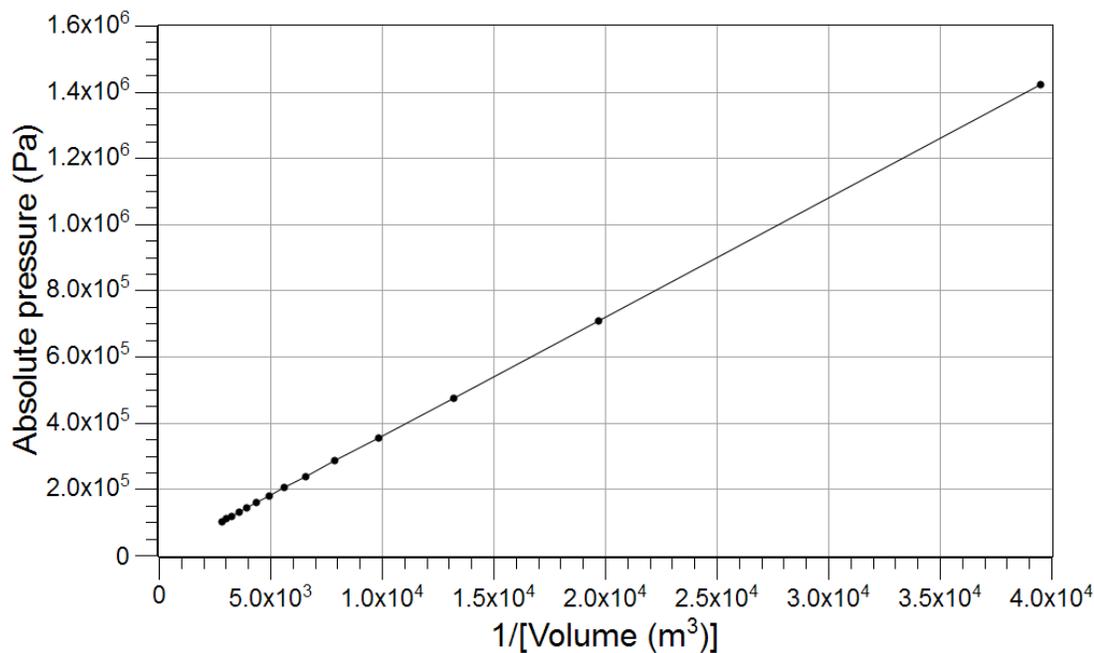
### Part 1 – Boyle's Law

- Open the file that you saved earlier in Part 1 of the procedure. ♦<sup>(1.1)</sup>
- Sketch or attach the Pressure versus Volume graph below.



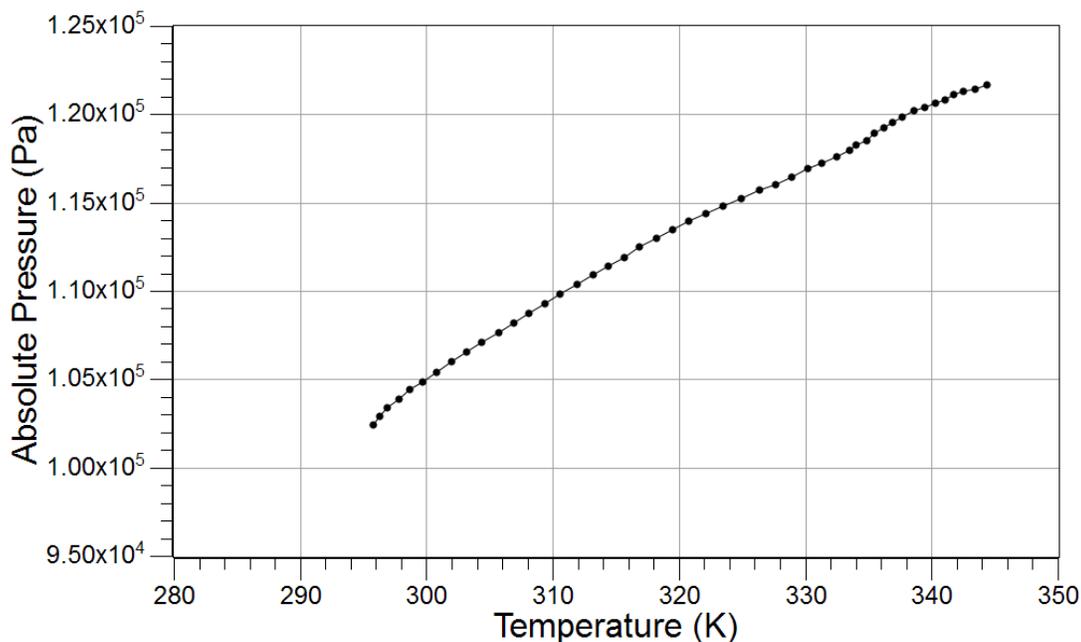
- Load the data set of pressure versus volume) and create a calculated data set of  $1/V$  using the volume data. ♦<sup>(10.3)</sup>

4.  Sketch or attach the pressure versus inverse volume graph below.



### Part 2 – Gay-Lussac's Law

5.  Open the file you saved earlier in Part 2 of the procedure.  $\diamond^{(1.1)}$
6.  Sketch or attach the Pressure versus Temperature graph below.



## Analysis Questions

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- 1. Did the pressure change as expected as you pushed the plunger in? How did the pressure change influence how hard you were pushing the plunger?**

The pressure increased as the plunger was pushed in. The increasing pressure increased the resistance, making it harder to push the plunger further into the syringe.

- 2. Did you find the pressure directly proportional or inversely proportional to the volume based on the graph?**

Inversely proportional.

- 3. What kind of relationship did you find based on the graph between the pressure and inverse volume? Express this relationship mathematically!**

Pressure was directly proportional to the inverse volume:  $p \propto \frac{1}{V}$

- 4. What kind of relationship did you find between the pressure and temperature based on the graph? Express this relationship mathematically!**

Pressure was directly proportional to the temperature:  $p \propto T$

- 5. Combine the two equations into one. (Hint: if a quantity is proportional to two other quantities, it is proportional to their products as well.)**

$$p \propto \frac{T}{V}$$

- 6. Predict what the intercept should be on the vertical axis and explain your prediction.**

The pressure should be zero at 0 K. That is where theoretically molecules have no kinetic energy and therefore their velocity is zero. Since molecules do not move, the pressure should be zero.

## Synthesis Questions

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Use available resources to help you answer the following questions.

- 1. Consider the Erlenmeyer flask with the stopper tightly inserted. How do you think the pressure would change if you doubled the number of molecules (moles) in the flask? Explain your prediction!**

Doubling the number of molecules increases the number of collisions, resulting in an increase of pressure.

- 2. Based on your answer to the previous question, what kind of mathematical relationship is there between the pressure and number of moles  $n$  of a gas? What is the mathematical representation of that relationship?**

Direct proportionality

$$p \propto n$$

**3.** Combine the relationship between pressure and the number of moles of the gas with the relationship you obtained for pressure, temperature, and volume.

$$p \propto n \frac{T}{V}$$

**4.** Direct proportionality can be turned into an equation by using a constant. For example, if  $A \propto B$ , then with a constant, for example,  $k$ , the relationship turns into a mathematical equation:  $A = kB$ . Using the constant  $R$ , turn the proportionality relationship between  $p$ ,  $n$ ,  $T$ , and  $V$  into an equation!

$$p = R \frac{nT}{V}$$

**5.** The equation you just derived is referred to as the "Ideal Gas Law" which relates pressure, temperature, number of moles, and volume for gases. The constant  $R$  is referred to as the universal gas constant and has the value of 8.314 J/(mol K). Arrange the Ideal Gas Law so you have  $p$  and  $V$  on the left and the other terms on the right. This is a more common form of this law.

$$pV = nRT$$

## Multiple Choice Questions

Select the best answer or completion to each of the questions or incomplete statements below.

**1.** Assume that the plunger of the syringe is at the 30 mL mark. Close the tip of the syringe with your thumb and pull the plunger to the 60 mL mark. How will the pressure change in the syringe?

- A.** The pressure will not change.
- B.** The pressure will double.
- C.** The pressure will be half of the original value.
- D.** We need the temperature to be able to calculate it.

**2.** The pressure and volume are inversely proportional to each other, which means:

- A.** They are also directly proportional to each other.
- B.** Pressure is directly proportional to the inverse volume.
- C.** The pressure is proportional to the temperature.
- D.** The pressure is proportional with the number of moles of gas molecules.

**3.** The relationship between the pressure and temperature is such that:

- A.** Increasing temperature will yield increasing pressure.
- B.** Increasing temperature will yield decreasing pressure.
- C.** Increasing temperature will yield increasing inverse pressure.
- D.** They are inversely proportional to each other.

4. Increasing the number of moles of gas two-fold in the Erlenmeyer flask will:

- A. Double the volume.
- B. Double the temperature.
- C. Double the gas constant.
- D. Double the pressure.

### Extended Inquiry Suggestions

As an extended inquiry, students can determine the theoretical values of the slope of the “ $p$  versus  $1/V$ ” and “ $p$  versus  $T$ ” graphs, based on the Ideal Gas Law, and compare them with the experimental values.

To determine the theoretical value of the slope of the  $p$  versus  $1/V$  graph, we need to rearrange the Ideal Gas Law:

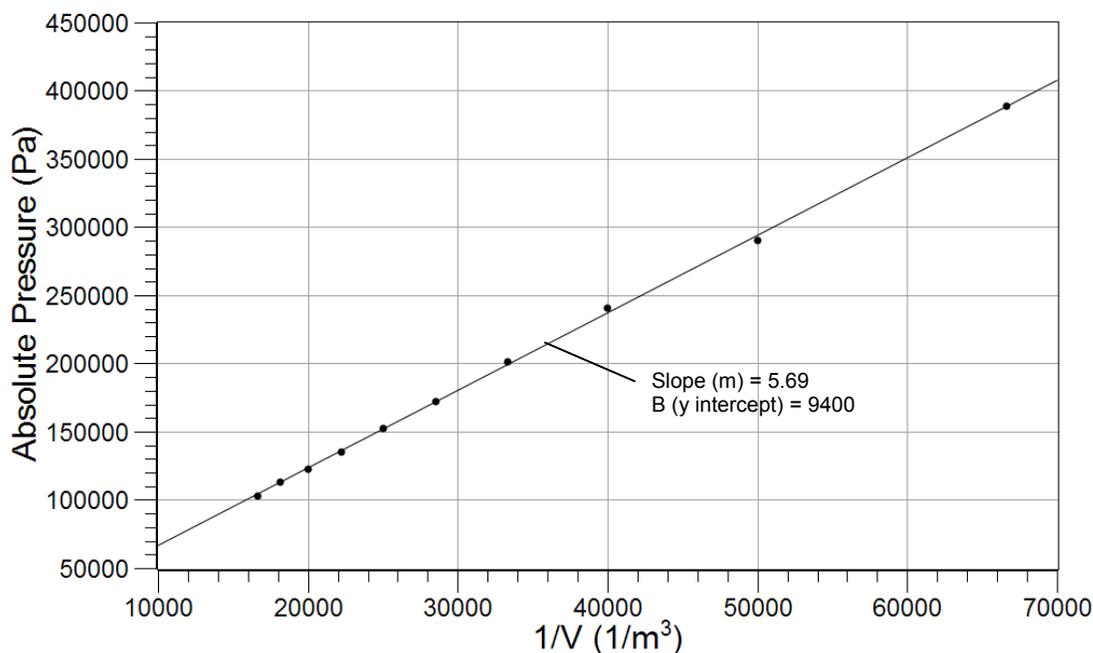
$$p = nRT \frac{1}{V}$$

The value of  $n$  can also be calculated from the Ideal Gas Law, based on the initial conditions:

$$n = \frac{pV}{RT} = \frac{\left(101325 \frac{\text{N}}{\text{m}^2}\right)(6.00 \times 10^{-5} \text{ m}^3)}{\left(8.314 \frac{\text{N m}}{\text{mol K}}\right)(298 \text{ K})} = 2.45 \times 10^{-3} \text{ mol}$$

The theoretical value of the slope, then, is:

$$nRT = (2.45 \times 10^{-3} \text{ mol}) \left(8.314 \frac{\text{J}}{\text{mol K}}\right) (298 \text{ K}) = 6.08 \text{ J}$$



The experimental value from the equation from the graph is 5.69 J; therefore, the error is:

$$\left| \frac{6.08 \text{ J} - 5.59 \text{ J}}{6.08 \text{ J}} \right| \times 100 = 8.1\%$$

To calculate the theoretical value of the slope of the Pressure versus Temperature graph, the Ideal Gas Law has to be rearranged as follows:

$$p = \frac{nR}{V}T$$

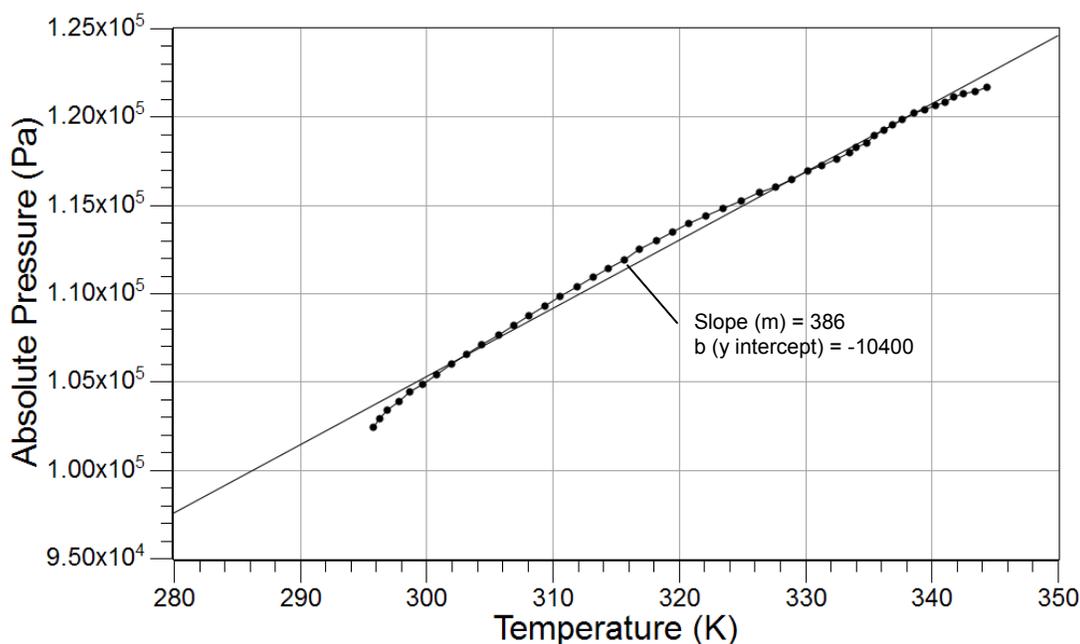
Students have to realize that along with  $n$ , they need the volume of the Erlenmeyer flask. The total volume of a 250-mL flask is more than 250 mL. Students must measure the entire volume (by filling it with water).

In this experiment,  $V = 256 \text{ mL}$ . When calculating  $n$ , they have to realize that after the stopper is placed in the flask,  $n$  doesn't change:

$$n = \frac{pV}{RT} = \frac{\left(101325 \frac{\text{N}}{\text{m}^2}\right)(2.56 \times 10^{-4} \text{ m}^3)}{\left(8.314 \frac{\text{N m}}{\text{mol K}}\right)(298 \text{ K})} = 1.05 \times 10^{-2} \text{ mol}$$

Therefore, the value of the slope should be

$$\frac{nR}{V} = \frac{(1.05 \times 10^{-2} \text{ mol})\left(8.314 \frac{\text{N m}}{\text{mol K}}\right)}{(2.56 \times 10^{-4} \text{ m}^3)} = 3.40 \times 10^2 \frac{\text{N}}{\text{m}^2 \text{ K}}$$



## Lab 29: Exploring Gas Laws

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The experimental value from the equation from the graph appears to be  $386.0 \text{ N}/(\text{m}^2\text{K})$ . The error is

$$\left| \frac{386.0 \frac{\text{N}}{\text{m}^2 \text{K}} - 340.0 \frac{\text{N}}{\text{m}^2 \text{K}}}{340.0 \frac{\text{N}}{\text{m}^2 \text{K}}} \right| \times 100 = 13.5\%$$

## Lab 30: Determination of the $K_a$ Values of Two Isomeric Multi-Protic Acids

Adapted from the work of Dr. Frazier Nyasulu

### Objectives

Students determine the acidity constants of two isomeric multi-protic acids, use these values to identify the acids, and provide an explanation for the difference in values based on molecular force considerations.

### Procedural Overview

Students will gain experience conducting the following procedures:

- ◆ Determining the  $pK_a$  of the multiple equivalence points of two isomeric multi-protic acids (fumaric acid and maleic acid) through titration
- ◆ Using a pH sensor and drop counter to perform titration measurements and determine equivalence points and solution concentrations

### Time Requirement

◆ Preparation time	50 minutes
◆ Pre-lab discussion and activity	30 minutes
◆ Lab activity	120 minutes

### Materials and Equipment

#### *For each student or group:*

- |                                    |  |
|------------------------------------|--|
| ◆ Data collection system           | ◆ Graduated cylinder, 100-mL                             |
| ◆ pH sensor                        | ◆ Magnetic stirrer                                       |
| ◆ Drop counter with micro stir bar | ◆ Unidentified fumaric acid solution, 50 mL <sup>1</sup> |
| ◆ Ring stand                       | ◆ Unidentified maleic acid solution, 50 mL <sup>2</sup>  |
| ◆ Clamp, right-angle               | ◆ 0.500 M Sodium hydroxide (NaOH), 150 mL <sup>3</sup>   |
| ◆ Clamp, buret                     | ◆ Funnel   |
| ◆ Beaker (2), 250-mL               | ◆ Buffers, pH 4 and pH 10, 10 mL                         |
| ◆ Beaker (2), 25-mL                | ◆ Wash bottle with deionized water                       |
| ◆ Buret, 50-mL                     | ◆ Cotton swab or tissue                                  |

<sup>1-3</sup> To prepare the solutions, refer to the Lab Preparation section.

## **Concepts Students Should Already Know**

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Students should be familiar with the following concepts:

- ◆ Titration
- ◆ Acid-base reactions
- ◆ Stoichiometry of chemical reactions
- ◆ Molarity
- ◆ Acidity constant:  $K_a$  and  $pK_a$

## **Related Labs in This Guide**

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Labs conceptually related to this one include:

- ◆ Lab 6: Standardizing a Solution of Sodium Hydroxide
- ◆ Lab 7: Acid–base Titration
- ◆ Lab 19: Properties of Buffers
- ◆ Lab 23: Determining of a Solubility Product
- ◆ Lab 24: Determining  $K_a$  by Half-Titration of a Weak Acid

## **Using Your Data Collection System**

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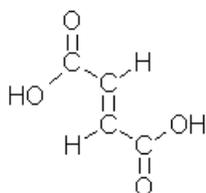
Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a sensor to the data collection system ◆<sup>(2.1)</sup>
- ◆ Connecting multiple sensors to the data collection system ◆<sup>(2.2)</sup>
- ◆ Calibrating a drop counter ◆<sup>(3.4)</sup>
- ◆ Calibrating a pH sensor ◆<sup>(3.6)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>
- ◆ Changing the variable on the x-axis and y-axis of a graph ◆<sup>(7.1.9)</sup>

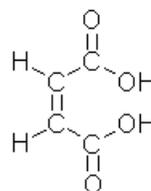
- ◆ Finding the coordinates of a point on a graph ◆<sup>(9.1)</sup>
- ◆ Finding the slope at a point on the data plot ◆<sup>(9.3)</sup>
- ◆ Saving your experiment ◆<sup>(11.1)</sup>
- ◆ Printing the graph. ◆<sup>(11.2)</sup>

## Background

Multi-protic acids are those acids that have more than one acidic proton. Among the organic molecules, those considered to be multi-protic have more than one carboxylic group (COOH). Maleic and fumaric acids are both multi-protic:



Fumaric acid



Maleic acid

Furthermore, these two acids are structural isomers, which means they have the same formula, but the 3-dimensional orientations of the atoms in the molecule are different. These kinds of differences usually result in some significant differences in chemical and physical properties.

Looking at fumaric acid first, the two acidic hydrogen atoms are identical; therefore, they are bound with the same strength to the rest of the molecule. This means their acidity is the same. Once the first hydrogen is removed, however, the molecule is negatively charged. The second positively charged hydrogen then has to be removed by a negatively charged ion. The second acidity constant, therefore, is somewhat smaller than the first:

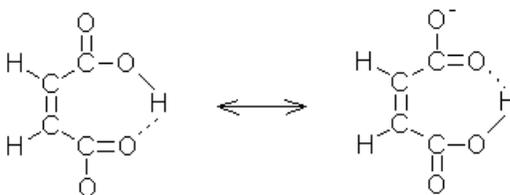
$$K_{a1} = 9.33 \times 10^{-4}, \quad \text{p}K_{a1} = 3.03$$

$$K_{a2} = 3.63 \times 10^{-5}, \quad \text{p}K_{a2} = 4.44$$

where 1 and 2 refer to the first and second acidic hydrogen ions.

When fumaric acid is titrated, both acidic protons detach at nearly the same time and the two equivalence points are not easily detected separately. Usually the equivalence points show up as a single equivalence point somewhere between the two values.

Maleic acid behaves differently. The first hydrogen detaches easily. However, there is a stabilizing effect that prevents the second hydrogen from detaching:



## Lab 30: Determination of the $K_a$ Values of Two Isomeric Multi-Protic Acids

The now negatively-charged oxygen binds to the other acidic hydrogen atom through a hydrogen bond. The hydrogen is now firmly bonded in a six-member ring, which is usually a very stable geometrical arrangement among organic molecules. The result is that the second hydrogen has very little ability to dissociate, and has a very small acidity constant.

$$K_{a1} = 1.26 \times 10^{-2}, \quad pK_{a1} = 1.90$$

$$K_{a2} = 8.51 \times 10^{-7}, \quad pK_{a2} = 6.07$$

### Pre-Lab Activity

#### Setting the stage for the activity

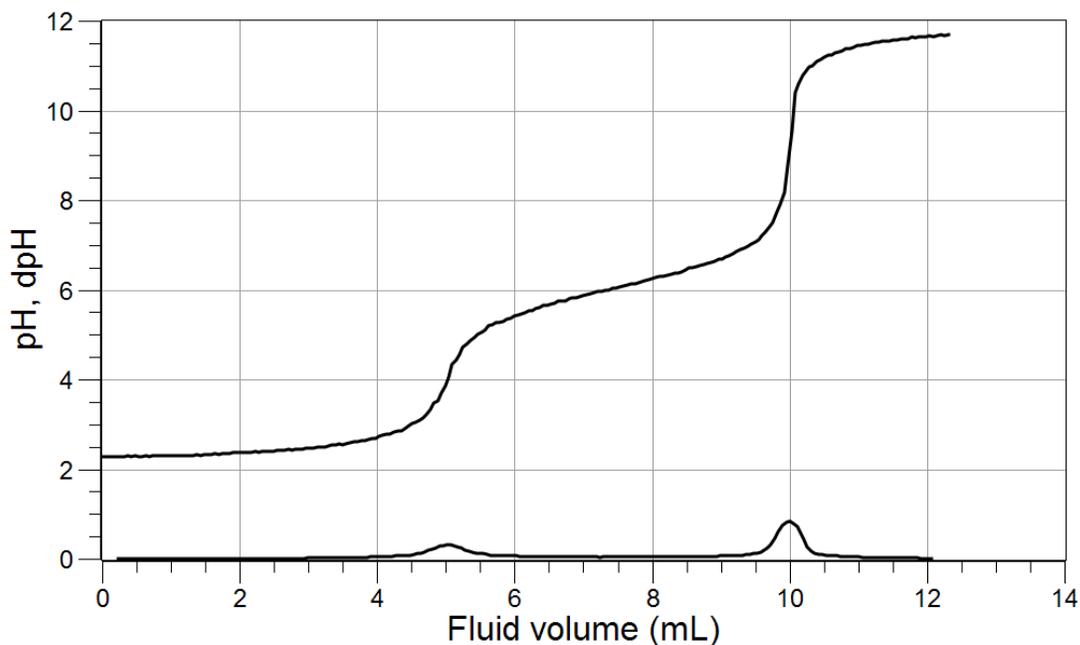
In this activity, you will titrate a sample of both acids and determine their acidity constants. The equivalence point will be detected using a pH electrode and the  $pK_a$  values will be determined from the half-titration point.

#### Example calculation to try

Both fumaric acid and maleic acid are made from maleic anhydride through hydrolysis. We analyzed two samples in order to identify maleic acid as one of the samples and fumaric acid as the other sample.

#### Sample I

A solution made from Sample I was analyzed first: 50.00 mL of solution was titrated with 0.100 M NaOH and the following titration curve was obtained:



The second trace (close to the x-axis) is the derivative of the pH graph; the maximum of the derivative shows the equivalence points. The presence of two equivalence points suggests that Sample I is maleic acid. The data in Table 1 can be obtained from the graph.

Table 1: Titration results of Sample I

Parameter	Equivalence Point #1	Equivalence Point #2
Equivalence point (mL)	5.00	10.00
Half-titration point (mL)	2.50	$5.00 + (10.00 - 5.00)/2 = 7.50$
pH at half-titration point	2.20	6.00
$pK_a$	2.20	6.00

The  $pK_a$  values determined from the graph are close to the theoretical values, confirming the presence of maleic acid. The percent error of this determination is

$$\text{Percent Error} = \left| \frac{\text{Theoretical Value} - \text{Experimental Value}}{\text{Theoretical Value}} \right| \times 100$$

$$\text{Percent error} = \left| \frac{1.90 - 2.20}{1.90} \right| \times 100 = 16\%$$

$$\text{Percent error} = \left| \frac{6.07 - 6.00}{6.07} \right| \times 100 = 1.2\%$$

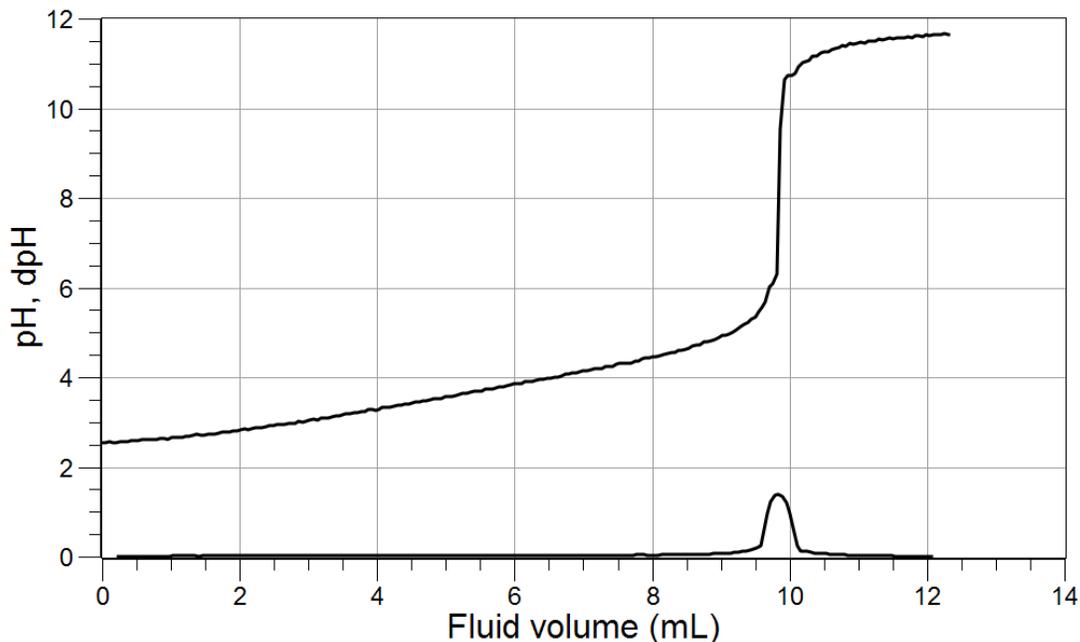
The concentration of that solution, using the first equivalence point, was

$$5.00 \text{ mL NaOH} \left( \frac{0.100 \text{ mol NaOH}}{1000 \text{ mL NaOH}} \right) \left( \frac{1 \text{ mol maleic acid}}{1 \text{ mol NaOH}} \right) \left( \frac{1}{5.000 \times 10^{-2} \text{ L}} \right) = 0.0100 \text{ M}$$

## Lab 30: Determination of the $K_a$ Values of Two Isomeric Multi-Protic Acids

### Sample II

A solution made from Sample II was analyzed next. 50.00 mL of solution was titrated with 0.100 M NaOH and the following titration curve was obtained:



The presence of only one equivalence point suggests that Sample II is fumaric acid. The data in Table 2 can be obtained from the graph.

Table 2: Titration results of Sample II

Parameter	Equivalence Point Values
Equivalence point (mL)	9.90
Half titration point (mL)	4.95
pH at half titration point	3.50
$pK_a$	3.50

The expected value of  $pK_a$  for fumaric acid is between  $pK_{a1}$  and  $pK_{a2}$ :  $(4.44 + 3.03)/2 = 3.74$ , so the percent error is

$$\text{Percent Error} = \frac{|3.74 - 3.50|}{3.74} \times 100 = 6.4\%$$

The concentration of Sample II, using the equivalence point, was:

$$9.90 \text{ mL NaOH} \left( \frac{0.100 \text{ mol NaOH}}{1000 \text{ mL NaOH}} \right) \left( \frac{1 \text{ mol fumaric acid}}{2 \text{ mol NaOH}} \right) \left( \frac{1}{5.000 \times 10^{-2} \text{ L}} \right) = 9.90 \times 10^{-3} \text{ M}$$

The concentration of the solution of Sample II was also  $9.90 \times 10^{-3}$  M.

**1. Explain the way the position of the second half-titration point for maleic acid was calculated!**

The first equivalence point was at 5.00 mL. The NaOH solution used between 5.00 mL and 10.00 mL was to obtain the second equivalence point for the second hydrogen. The halfway point (where the pH will be equal to the  $pK_a$ ) is

$$(10.00 \text{ mL} - 5.00 \text{ mL})/2 = 2.50 \text{ mL}$$

after the first equivalence point, so the point at which this happens is

$$5.00 \text{ mL} + 2.50 \text{ mL} = 7.50 \text{ mL}$$

**2. Would we get a different result for the concentration of maleic acid if we used the second equivalence point instead of the first for the calculation?**

No, we would have gotten the same results:

$$10.00 \text{ mL NaOH} \left( \frac{0.100 \text{ mol NaOH}}{1000 \text{ mL NaOH}} \right) \left( \frac{1 \text{ mol maleic acid}}{2 \text{ mol NaOH}} \right) \left( \frac{1}{5.000 \times 10^{-2} \text{ L}} \right) = 0.0100 \text{ M}$$

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

- 1. 0.027 M Maleic Acid:** Dissolve 1.566 g of maleic acid in about 500 mL of distilled water in an Erlenmeyer flask. Place a stirring bar into the solution and on a hot plate gently heat and stir the solution until all solid dissolves. Allow the solution to cool to room temperature. Transfer the solution into a 2-L volumetric flask and fill it to the mark. Mix the solution well.

Label the solution "Unknown I."

- 2. 0.027 M Fumaric Acid:** Dissolve 1.566 g of fumaric acid in about 500 mL of distilled water in an Erlenmeyer flask. Place a stirring bar into the solution and on a hot plate gently heat and stir the solution until all solid dissolves. Allow the solution to cool to room temperature. Transfer the solution into a 2-L volumetric flask and fill it to the mark. Mix the solution well.

Label the solution "Unknown II."

- 3. 0.500 M NaOH:** Dissolve 20.00 g of NaOH in about 500 mL of distilled water in an Erlenmeyer flask. Allow the solution to cool to room temperature. Transfer the solution into a 1-L volumetric flask and fill it to the mark. Mix the solution well.

**Note:** Standardize the solution and report the actual concentration to the students.

## Safety

Follow all standard laboratory procedures.

## Sequencing Challenge

The steps below are parts of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

5	2	4	3	1
Determine which is which, then determine the $pK_a$ values and the concentrations of the two solutions.	Transfer 100 mL of the first unknown (maleic or fumaric acid) into a beaker. Fill the 50-mL buret with the titrant (0.5 M NaOH).	Perform the same procedure with the second unknown.	Perform the titration until the pH curve levels off after the second equivalence point.	Set up the titration apparatus with a pH sensor and drop counter. Monitor pH versus drop counts.

## Procedure with Inquiry

After you complete a step (or answer a question), place a check mark in the box () next to that step.

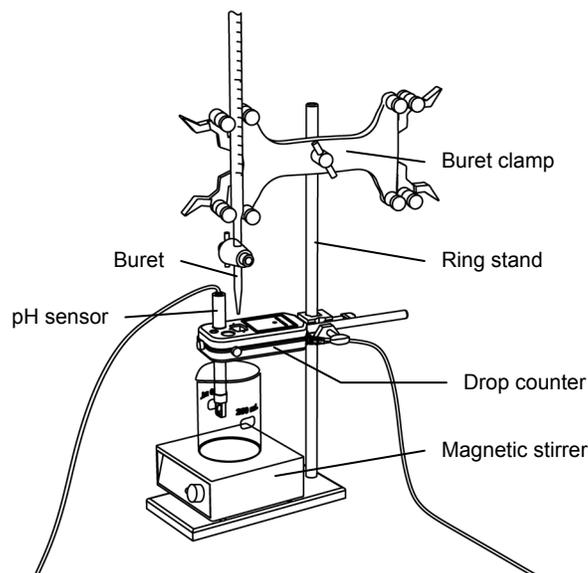
**Note:** When students see the symbol "♦" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

- Start a new experiment on the data collection system. ♦<sup>(1.2)</sup>
- Connect a pH sensor to the data collection system. ♦<sup>(2.1)</sup>
- Calibrate the pH sensor. ♦<sup>(3.6)</sup>

4.  Assemble the titration apparatus, using the steps below and the illustration as a guide.

- a. Position the magnetic stirrer on the base of the ring stand.
- b. Place a waste container (250-mL beaker) on the magnetic stirrer.
- c. Use the buret clamp to attach the buret to the ring stand.
- d. Position the drop counter over the waste container and attach it to the ring stand using the right-angle clamp.
- e. Place the pH sensor through one of the slots in the drop counter.



**Note:** Do not connect the drop counter to the data collection system yet.

5.  Rinse the buret with several milliliters of the 0.500 M NaOH solution:
- a. Ensure that the stopcock is closed and rinse the inside of the buret with several milliliters of the standardized NaOH solution.
  - b. Open the stopcock on the buret and drain the rinse NaOH into the waste container.
  - c. Repeat this process two more times.

6.  Why is it necessary to rinse the buret with the NaOH solution?

If there is any residual water or contaminant in the buret, it will dilute the NaOH and change its concentration. Rinsing eliminates any such contamination.

7.  Make sure the stopcock on the buret is in the “off” position and then use a funnel to fill the buret with about 50 mL of the 0.500 M NaOH solution (titrant).
8.  Drain a small amount of the titrant through the drop counter into the waste beaker to remove any air in the tip of the buret.
9.  Why is it important to remove air from the tip of the buret?

Any air trapped in the buret tip is counted as volume of NaOH. If this happens, the amount of titrant used will be inaccurate.

10.  Practice adjusting the stopcock on the buret so that the titrant goes through the drop counter in distinguishable drops that fall at about 1 to 2 drops per second.

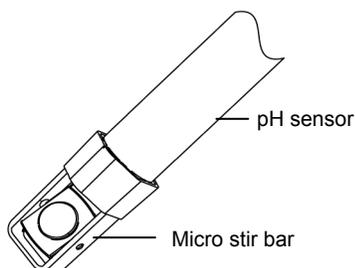
**Note:** Good control of the stopcock is important. Each drop should result in a blink of the LED on the drop counter. If the LED is continuously lit, you have opened the stopcock too far and you will have to start over.

### Lab 30: Determination of the $K_a$ Values of Two Isomeric Multi-Protic Acids

11.  Why will it be necessary to start your titration over again if you accidentally allow the titrant to stream out of the stopcock instead of emerging by drops?

The drop counter counts distinct drops. If the drops are not sufficiently distinct from one another, the drop counter will not function properly and the fluid volume will not be accurate.

12.  Add the micro stir bar to the end of the pH sensor.



13.  Why is it necessary to stir the solution during a titration?

Stirring thoroughly mixes the ions in the solution so that the recorded pH reflects the pH of the entire solution.

14.  Add additional 0.500 M NaOH to the buret so the solution is above the zero mark. Allow some of the NaOH solution to drip into the waste container until the bottom of the meniscus is lined up with or just below the zero mark and record the initial reading in Table 3.

15.  Remove the waste container.

16.  Use the graduated cylinder to pour 100.0 mL of the Unknown I solution into a 250-mL beaker and set the beaker on the magnetic stirrer.

17.  Lower the pH sensor into the solution.

18.  Turn on the magnetic stirrer at a slow and steady rate.

19.  Connect the drop counter to the data collection system.  $\diamond^{(2,2)}$

20.  Display the pH on the y-axis of a graph and Drop Count on the x-axis.  $\diamond^{(7,1.1)}$

21.  Clean the lens of the drop counter inside the opening through which the drops go with water and a cotton swab or tissue.

#### Collect Data

22.  Start recording data.  $\diamond^{(6,2)}$

23.  Turn the buret stopcock carefully, allowing the titrant to drip slowly (1 to 2 drops per second) into the solution.

24.  Continue the titration past the equivalence point until the pH curve flattens.

**Note:** Not knowing which solution is fumaric or maleic acid, determine a strategy that will ensure the titration provides the necessary information for each.

25.  Why is it important to go past the equivalence point?

It is necessary to go past the equivalence point in order to find the point where the slope is the steepest. The curve needs to continue past the steepest point to ensure you can tell when the slope begins to flatten.

26.  How do you know when to stop the titration, not knowing how many equivalence points will be there?

The pH should level off around 12 when the titration is completed.

27.  Stop recording data. ♦<sup>(6.2)</sup>

28.  In Table 3, record the final drop count and the final reading of the titrant in the buret to a precision of 0.01 mL.

29.  Calculate the volume of titrant (final reading minus initial reading) and record this value in Table 3.

Table 3: Titration data

Titration Information	Unknown I	Unknown II
Initial reading of HCl on the buret (mL)	0.00	0.00
Final reading of HCl on the buret (mL)	14.40	13.90
Volume of titrant (mL)	14.40	13.90
Final drop count	480	463

30.  Calibrate the drop counter. ♦<sup>(3.4)</sup>

31.  On the graph, set the horizontal axis to the calculated volume. ♦<sup>(7.1.9)</sup>

32.  Print the graph. ♦<sup>(11.2)</sup>

33.  Record the volume of titrant used to reach each equivalence point in Table 4.

**Note:** The equivalence point will be where the slope of the titration curve is the steepest. Find the steepest slope of the data plot to determine this point. ♦<sup>(9.3)</sup>

**Note:** For one of the unknowns, only one equivalence point will be detected, even though there are two. The one detected can be considered the second equivalence point.

## Lab 30: Determination of the $K_a$ Values of Two Isomeric Multi-Protic Acids

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34.  Refill the buret over the zero mark with the NaOH solution.
- Fill the buret above the zero mark and allow some of the NaOH solution to drip into a waste container until the bottom of the meniscus is lined up with the zero mark or just below.
  - Record the starting point in Table 3.
35.  Clean the lens of the drop counter between runs with water and a cotton swab or tissue.
36.  Rinse the pH probe tip with deionized water.
37.  Remove the beaker and dispose of its contents according to the teacher's instructions.
38.  Rinse the beaker with distilled water.
39.  Use the graduated cylinder to pour 100.0 mL of the Unknown II solution into the 250-mL beaker and set the beaker on the magnetic stirrer.
40.  Lower the pH sensor into the solution.
41.  Turn on the magnetic stirrer at a slow and steady rate.
42.  Return to the first step of the Collect Data section and repeat the titration with the Unknown II solution.
43.  Save your experiment  $\diamond^{(11.1)}$  and clean up according to your teacher's instructions.

### Data Analysis

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1.  Obtain the concentration of the NaOH solution from your teacher and record it in Table 4.

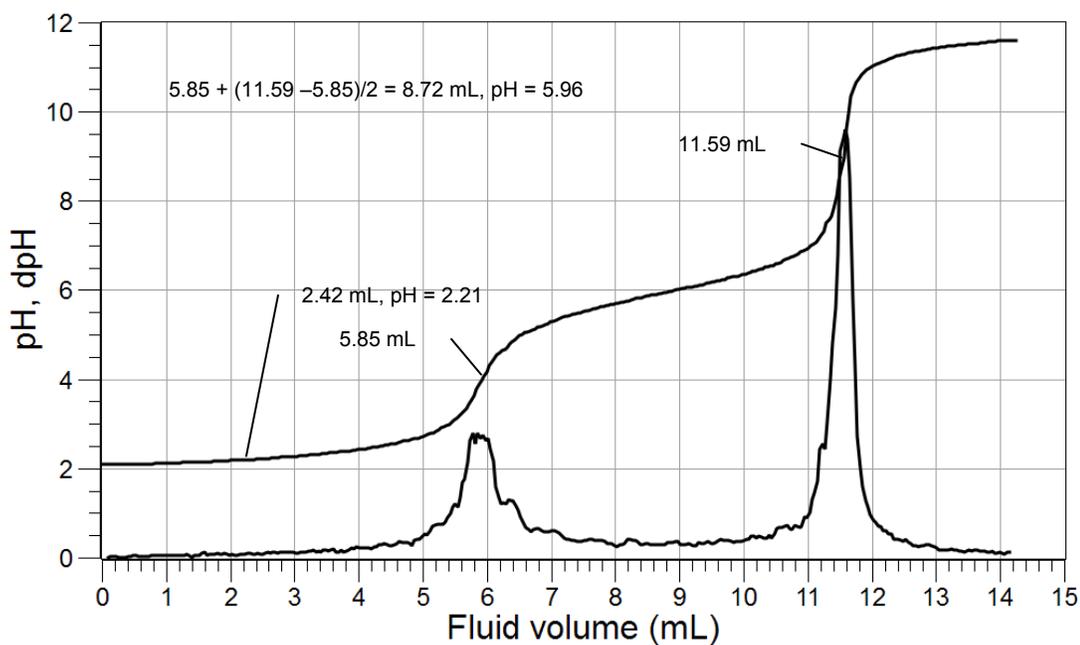
Table 4: Equivalence points of the unknown solutions

Parameter	Unknown I	Unknown II
Concentration of the NaOH solution (M)	0.5000	
Volume to the first equivalence point (mL)	5.85	
Volume to the second equivalence point (mL)	11.59	18.88

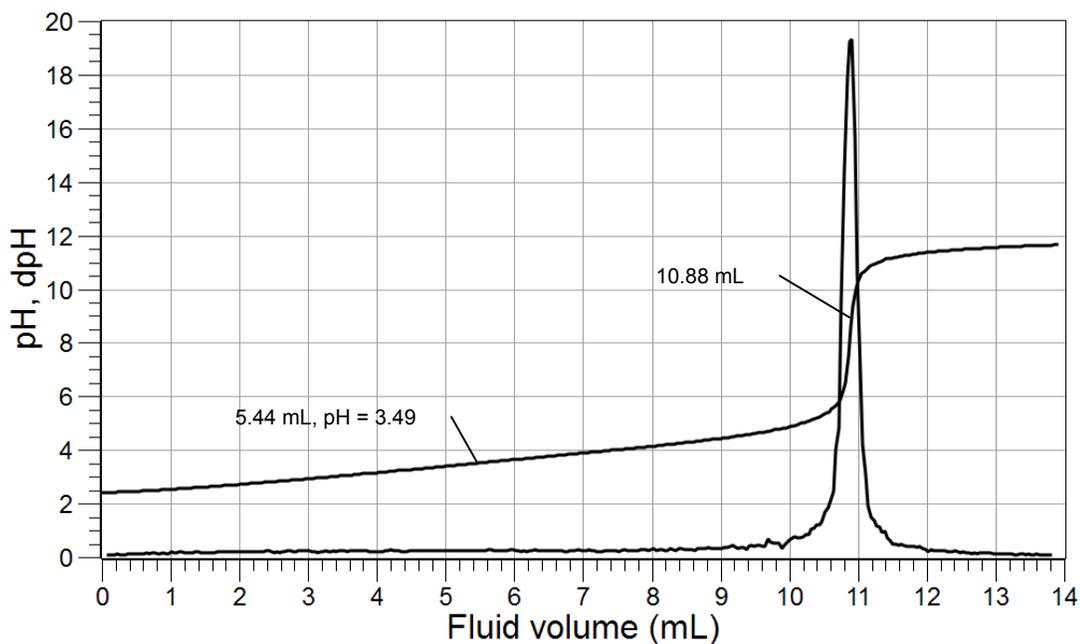
2.  Which solution is maleic acid and which is fumaric acid?

Unknown I is maleic acid, as it has two distinct equivalence points. Unknown II is fumaric acid.

3.  Copy the values in Table 4 to the appropriate column in Table 5.
4.  Sketch or attach the two titration curves below. Identify which is fumaric and which maleic acid.



Maleic acid



Fumaric acid

### Lab 30: Determination of the $K_a$ Values of Two Isomeric Multi-Protic Acids

5.  Calculate the half-titration points for each equivalence point and record them in Table 4.

For maleic acid, the first half-titration point is at half the volume of the equivalence point ( $5.85 \text{ mL}/2 = 2.42 \text{ mL}$ ). The second half-titration point is determined by adding half the volume of titrant used to the first equivalence point [ $5.85 \text{ mL} + (11.59 \text{ mL} - 5.85 \text{ mL})/2 = 8.72 \text{ mL}$ ].

For fumaric acid, the half-titration point is  $10.88 \text{ mL}/2 = 5.44 \text{ mL}$ .

6.  Use the graphs to determine the pH and  $pK_a$  at the half-titration points and record these values in Table 5. <sup>(9.1)</sup>

For maleic acid, the pH is 2.21 and 5.96. The  $pK_a$  values are 2.21 and 5.96.

For fumaric acid, the pH is 3.40 and the  $pK_a$  is 3.40.

7.  Record the published values of the  $pK_a$  for maleic and fumaric acids in Table 5.

8.  What is the percentage of error between the published values and the values you determined from the titration? Record these in Table 5.

For maleic acid and fumaric acid:

$$\text{Percent error} = \left| \frac{1.90 - 2.21}{1.9} \right| \times 100 = 16.3\%$$

$$\text{Percent error} = \left| \frac{6.07 - 5.96}{6.07} \right| \times 100 = 1.81\%$$

$$\text{Percent error} = \left| \frac{3.73 - 3.40}{3.73} \right| \times 100 = 8.85\%$$

9.  Calculate the concentrations of the two solutions and determine the  $pK_a$  values. Record these values in Table 5.

$$5.85 \text{ mL NaOH} \left( \frac{0.500 \text{ mol NaOH}}{1000 \text{ mL NaOH}} \right) \left( \frac{1 \text{ mol maleic acid}}{1 \text{ mol NaOH}} \right) \left( \frac{1}{100.0 \times 10^{-3} \text{ L}} \right) = 2.925 \times 10^{-2} \text{ M}$$

$$10.88 \text{ mL NaOH} \left( \frac{0.500 \text{ mol NaOH}}{1000 \text{ mL NaOH}} \right) \left( \frac{1 \text{ mol fumaric acid}}{2 \text{ mol NaOH}} \right) \left( \frac{1}{100.0 \times 10^{-3} \text{ L}} \right) = 2.72 \times 10^{-2} \text{ M}$$

Table 5: Determination of acidity constants and concentration

	Maleic Acid		Fumaric Acid
Equivalence point	1	2	1–2
Equivalence point (mL)	5.85	11.59	10.88
Half-titration point (mL)	2.42	8.72	5.44
pH at half-titration point	2.21	5.96	3.40
$pK_a$	2.21	5.96	3.40
$pK_a$ from literature	1.90	6.07	3.73 (avg)
Percent error (%)	16.3	1.81	8.85
Concentration (M)	$2.92 \times 10^{-2}$		$2.72 \times 10^{-2}$

### Analysis Questions

**1. Does your experimental data support the predicted values regarding the two acidity constants of maleic acid?**

Yes, the two acidity constants came out close to the predicted values.

**2. Does your experimental data support predicted value regarding the two acidity constants of fumaric acid??**

The two acidity constants were determined as one, close to the average of the two.

**3. Why was only one equivalence point detected in the fumaric acid solution?**

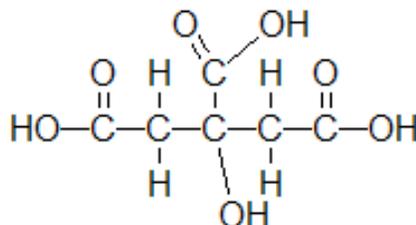
The two constants are too close to each other to be detected separately.

## Synthesis Questions

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Use available resources to help you answer the following questions.

1. Consider citric acid, which is an important ingredient in lemons:



How many acidic hydrogen atoms can you identify?

There are three: the ones attached to oxygen atoms on carboxylic groups.

2. Predict how close the acidity constants ( $K_a$ ) are to each other?

The acidity constants should be relatively close to each other, just like those of fumaric acid. Indeed, the three  $K_a$  values are:  $7.41 \times 10^{-2}$ ,  $1.74 \times 10^{-5}$ ,  $2.72 \times 10^{-7}$ .

3. Which acidity constant ( $K_a$ ) of citric acid is smallest and why?

The third one, since the third positively -charged hydrogen would have to come off of a doubly negatively-charged ion.

## Multiple Choice Questions

---

Select the best answer or completion to each of the questions or incomplete statements below.

1. Which statement is correct regarding the acidity constants of fumaric acid?
- A. They are close to each other.
  - B. The first one is much larger than the second.
  - C. The second one is much larger than the first one.
  - D. Identical.
2. Which statement is correct regarding the acidity constants ( $K_a$ ) of maleic acid?
- A. They are close to each other.
  - B. The first one is much larger than the second.
  - C. The second one is much larger than the first one.
  - D. Identical.

- 3. What is the second acidity constant of maleic acid influenced by?**
- A.** It is influenced by the pH of the solution.
  - B.** It is influenced by the formation of a stable structure after the dissociation of the first hydrogen ion.
  - C.** It is affected by the first acidity constant.
  - D.** It is influenced titrating solution.
- 4. The acidity constants of fumaric and maleic acids are different because:**
- A.** They have the same formula
  - B.** They have the same formula but different structure.
  - C.** They are both organic acids.
  - D.** They are not different.

### **Extended Inquiry Suggestions**

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Students can analyze a mixture of maleic acid and fumaric acid. There will be three equivalence points with the equivalence point for fumaric acid falling between the first and second equivalence points of maleic acid.

### **Acknowledgements**

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This activity was adapted from the following work of Dr. Frazier Nyasulu:

Nyasulu, F. W.; Macklin, J. *Journal of Chemical Education*. 2006, 83 (5), 770–773.



## Lab 31: Determining the Half-Life of an Isotope

### Objectives

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Students investigate the radioactive decay and half-life of an isotope.

### Procedural Overview

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Students gain experience conducting the following procedures:

- ◆ Measuring the radioactive decay of barium-137m
- ◆ Calculating the decay constant and half-life of an isotope

### Time Requirement

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◆ Preparation time	10 minutes
◆ Pre-lab discussion and activity	15 minutes
◆ Lab activity	25 minutes

### Materials and Equipment

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*For each student or group:*

- ◆ Data collection system
- ◆ Alpha beta gamma radiation sensor <sup>1</sup>
- ◆ Barium-137m solution<sup>2</sup>
- ◆ Aluminum plate

<sup>1</sup> The alpha beta gamma radiation sensor is also referred to as the G-M counter.

<sup>2</sup> To prepare the barium-137m solution using the isotope generator kit, refer to the Lab Preparation section.

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ First-order kinetics
- ◆ Logarithmic representation of data
- ◆ Radioactivity

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 12: Determination of the Rate of the Decomposition of Hydrogen Peroxide
- ◆ Lab 27: Order of Reaction

## Using Your Data Collection System

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting a sensor to your data collection system ◆<sup>(2.1)</sup>
- ◆ Starting and stopping data recording ◆<sup>(6.2)</sup>
- ◆ Displaying data in a graph ◆<sup>(7.1.1)</sup>
- ◆ Finding the slope and intercept of a best-fit line ◆<sup>(9.6)</sup>
- ◆ Creating calculated data set ◆<sup>(10.3)</sup>
- ◆ Save your experiment ◆<sup>(11.1)</sup>
- ◆ Printing ◆<sup>(11.2)</sup>

## Background

---

A nuclear reaction in which an unstable isotope of an element emits radiation spontaneously is called "radioactive decay." There are three basic types of radiation that can be emitted during a decay process: alpha "α" (essentially the nucleus of He atoms,  ${}^4_2\text{He}^{2+}$ ), beta "β" (made up of electrons), and gamma "γ" (high energy electromagnetic radiation).

Radioactive (or nuclear) decay is a *random* process, yet it is somewhat predictable. It is not possible to predict when a particular radioactive atom will decay, but it is safe to say that the more unstable an isotope is, the more it will decay within a period of time; that is, the number of decaying radioactive atoms within a period of time is proportional to the amount of radioactive atoms present:

$$\frac{\Delta N}{\Delta t} = -kN$$

where

$\Delta N$  = number of radioactive atoms that decays within the set time  $\Delta t$

$\Delta t$  = time of observation (s)

$k$  = decay constant (the fraction of the radioactive atoms that decays per unit time), different for each isotope ( $\text{s}^{-1}$ )

$N$  = number of radioactive atoms present

Mathematically, this equation takes the following form as a function of time:

$$N(t) = N_0 e^{-kt} \quad (1)$$

where

$N_0$  = number of radioactive atoms at time  $t = 0$

$N(t)$  = number of radioactive atoms after time  $t$

A concept that originated from the study of radioactive decay is “half-life.” The half-life  $t_{1/2}$  of a radioactive isotope is the time it takes for half of the original atoms to decay:

$$\frac{N_0}{2} = N_0 e^{-kt_{1/2}}$$

$$\ln \frac{N_0}{2} = \ln N_0 - kt_{1/2}$$

$$\ln N_0 - \ln 2 = \ln N_0 - kt_{1/2}$$

$$\ln 2 = kt_{1/2}$$

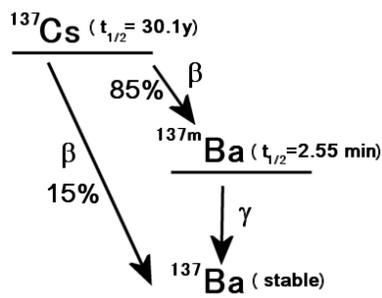
$$t_{1/2} = \frac{\ln 2}{k} = \frac{0.693}{k}$$

Half-lives can be as short as a fraction of a second or as long as a billion years, depending on the isotope.

## Pre-Lab Activity

### Setting the stage for the activity

In this activity, your teacher will use an isotope generator to provide you with a small quantity of the short-lived barium-137m isotope. The barium-137m isotope is a product of the decay of the cesium-137 isotope:



In the isotope generator, the cesium-137 radioactive atoms are bound on a special matrix. When a washing solution (eluent) is forced through the generator, the product (barium-137m) is washed off the matrix and collected in the washing solution (eluate). As the barium-137m decays to its ground state by emitting  $\gamma$  radiation, it forms the stable barium-137 isotope.

You will use a G-M counter to monitor the decay of the barium-137m in order to calculate the decay constant and half-life of the isotope. The G-M counter will provide the rate of decay data (the number of atoms that decayed during one second). To use that data, you need to substitute the equation for  $N(t)$  into the rate expression:

$$\frac{\Delta N}{\Delta t} = -kN$$

Substituting  $N_0 e^{-kt}$  for  $N$  (from Equation 1):

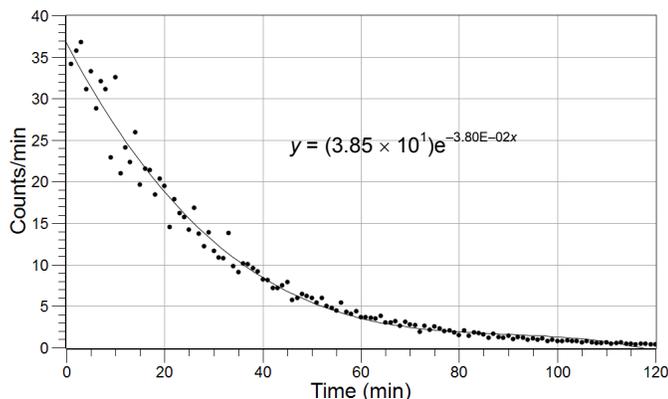
$$\frac{\Delta N}{\Delta t} = -kN_0 e^{-kt}$$

$$\ln \frac{\Delta N}{\Delta t} = -\ln(kN_0) - kt$$

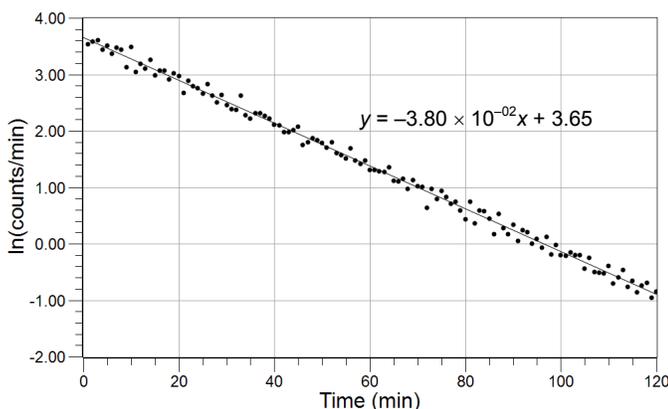
Therefore, if you plot  $\ln(\Delta N/\Delta t)$  versus  $t$ , the slope gives you the decay constant. Once you know the decay constant, you can calculate the number of radioactive atoms present at  $t = 0$  from the y-intercept  $[\ln(kN_0)]$ .

**Example calculation to try**

In an experiment, the decay of barium-141 was studied. The following graphs, showing the rate of decay, were obtained with a G-M counter:



To determine the decay constant, the logarithm of the rate as a function of time was plotted:



The value of the decay constant  $k$ , obtained from the slope, is  $3.80 \times 10^{-2} \text{ min}^{-1}$ . From that, the half-life of the barium-141 isotope was calculated:

$$t_{1/2} = \frac{0.693}{3.80 \times 10^{-2} \text{ min}^{-1}} = 18.2 \text{ min}$$

**1. Does the half-life depend on the initial number of radioactive atoms?**

No, the half-life depends only on the value of the decay constant.

**2. How would you obtain the initial number of radioactive atoms from the logarithmic graph?**

The y-intercept is  $\ln(kN_0)$ :

$$\ln(kN_0) = 3.65$$

$$kN_0 = e^{3.65}$$

$$N_0 = \frac{e^{3.65}}{k} = \frac{38.47}{3.80 \times 10^{-2} \text{ min}^{-1}} = 1.01 \times 10^3$$

## Lab Preparation

---

These are the materials and equipment to set up prior to the lab:

1. To prepare the solution with the barium-137m isotope, draw some eluting solution into the syringe, mount the isotope generator onto the tip of the syringe and squeeze the solution through the isotope generator. The solution dripping from the generator (the eluate) contains the barium-137m radioactive atoms. Collect the eluate into small aluminum plates and distribute them to students for immediate analysis.

## Safety

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Add these important safety precautions to your normal laboratory procedures:

- ◆ Gloves and lab coats should be worn when working with all liquid radioisotopes.
- ◆ As always, wash your hands thoroughly before leaving the lab, and then check for possible contamination.

## Sequencing Challenge

---

The steps below are parts of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

2	3	1	5	4
Obtain the barium-137m solution on a small aluminum plate from your instructor.	Place the plate with the solution under the radiation sensor.	Set up the radiation sensor on your data collection system. Display a graph of counts/sec versus time.	Calculate the decay constant and the half-life of barium-137m.	Start data collection. Continue data collection for about 20 min.

---

## Procedure with Inquiry

---

After you complete a step (or answer a question), place a check mark in the box (☐) next to that step.

**Note:** When students see the symbol "◆" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

### Set Up

1. ☐ Start a new experiment on the data collection system. ◆<sup>(1.2)</sup>
2. ☐ Connect an alpha beta gamma radiation sensor to the data collection system. ◆<sup>(2.1)</sup>
3. ☐ Display Counts/sec on the y-axis of a graph with Time on the x-axis. ◆<sup>(7.1.1)</sup>
4. ☐ Obtain the barium-137m solution from your instructor on a small aluminum plate.

### Collect Data

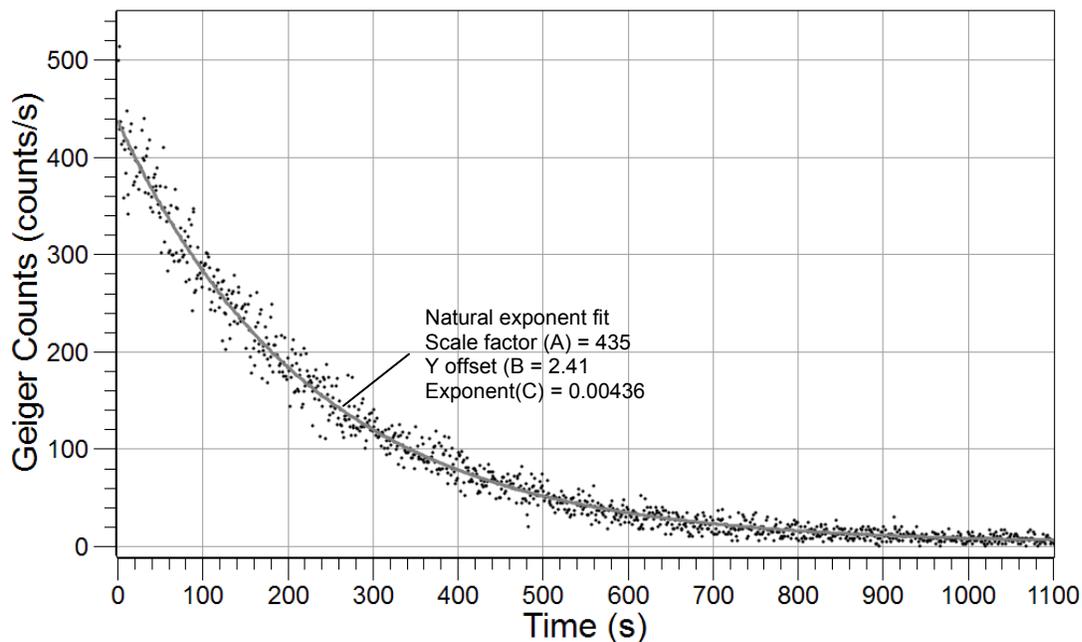
5. ☐ Start data recording. ◆<sup>(6.2)</sup>
6. ☐ Why do you have to start the data recording immediately?  
Data recording has to be started because the barium-137m isotope has a very short half life.
7. ☐ Collect data for 20 minutes.

**Note:** After 20 minutes the rate should decline to nearly zero.

8. ☐ Stop data recording. ◆<sup>(6.2)</sup>
9. ☐ Print the graph. ◆<sup>(11.2)</sup>
10. ☐ Save your experiment ◆<sup>(11.1)</sup> and clean up according to your teacher's instructions.

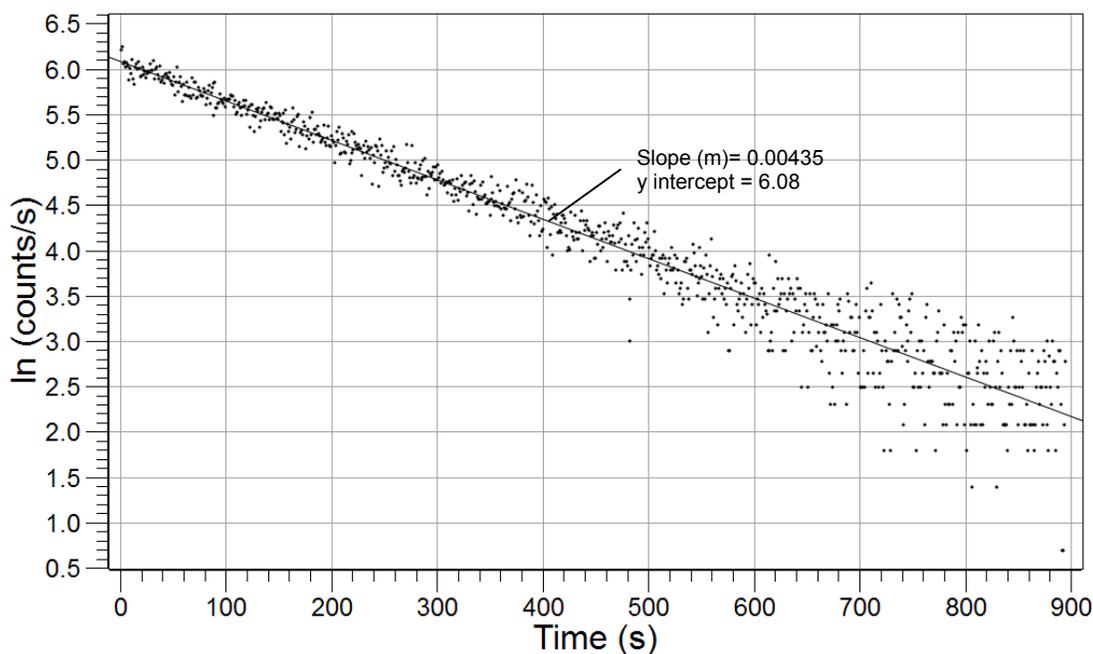
## Data Analysis

1.  Sketch or attach the Counts versus Time graph below.



2.  Create a calculated data set of  $\ln(\text{counts/s})$ .  $\diamond^{(10.3)}$
3.  Display the data set on the y-axis with Time on the x-axis.  $\diamond^{(7.1.1)}$
4.  Find the slope and y-intercept of the best-fit line.  $\diamond^{(9.6)}$
5.  Print the graph.  $\diamond^{(11.2)}$

6.  Sketch or attach the  $\ln(\text{counts/sec})$  versus Time graph below.



7.  Record the slope and y-intercept in Table 1.

The slope of the graph is  $-4.35 \times 10^{-3}$ . The y-intercept is 2.41.

8.  Convert the value of the slope from per second to per minute. Record the value in Table 1.

$$\left(-4.35 \times 10^{-3} \text{ s}^{-1}\right) \times 60 \frac{\text{s}}{\text{min}} = 0.261 \text{ min}^{-1}$$

9.  What is the decay constant? Record the value in Table 1.

The decay constant is the slope,  $0.261 \text{ min}^{-1}$ .

10.  Calculate the initial amount of barium-137m. Record the value in Table 1.

The y-intercept is  $\ln(kN_0)$ :

$$\ln(kN_0) = 6.08$$

$$kN_0 = e^{6.08}$$

$$N_0 = \frac{e^{6.08}}{k} = \frac{437}{4.35 \times 10^{-3} \text{ min}^{-1}} = 1.00 \times 10^5$$

11.  Calculate the half-life of barium-137. Record the value in Table 1.

$$t_{1/2} = \frac{0.693}{0.261 \text{ min}^{-1}} = 2.65 \text{ min}$$

## Lab 31: Determining the Half-Life of an Isotope

**12.** □ Obtain the theoretical value of the half-life of barium-137m. Record this value in Table 1.

The theoretical value is 2.55.

**13.** □ Calculate the percent error between the theoretical value and the value you calculated.

$$\text{Percent Error} = \frac{|\text{Theoretical Value} - \text{Experimental Value}|}{\text{Theoretical Value}} \times 100$$

$$\text{Percent Error} = \frac{|2.55 - 2.65|}{2.55} \times 100 = 3.92\%$$

Table 1: Determination of the half-life of barium-137m

Parameters	Values
Slope ( $\text{s}^{-1}$ )	$-4.35 \times 10^{-3}$
Slope ( $\text{min}^{-1}$ )	-0.261
y-intercept	6.08
$k$ ( $\text{min}^{-1}$ )	0.261
$N_0$	$1.00 \times 10^5$
$t_{1/2}$ (min)	2.65
Theoretical $t_{1/2}$ (min)	2.55
Error (%)	3.92

### Analysis Questions

**1.** What does it mean to say that the half-life of the barium-137m isotope is 2.65 min?

It means that it takes 2.65 min for half of the material of a radioactive isotope— in this case, barium-137m— to decay.

**2.** How would you determine  $N_0$  from the  $\ln[N(t)]$  versus  $t$  graph?

$\ln(N_0)$  is the y-intercept.

**3.** How would you determine  $N_0$  from the rate of decay versus  $t$  graph? (Hint: Each point represents the number of radioactive atoms decayed during that minute; sum those between 0 to 20 minutes.)

The area under the rate of decay curve is the sum of the number of the radioactive atoms that decayed each minute, which is the number of original radioactive atoms.

## Synthesis Questions

Use available resources to help you answer the following questions.

**1. What do you think the  $N(t)$  versus  $t$  graph looks like?**

The  $N(t)$  versus  $t$  graph should show exponential decay.

**2. Carbon dating is a method based on the radioactive dating of the carbon-14 isotope that determines the age of carbon-based matter that was once alive. It is based on the fact that as long as the matter is alive, the ratio of the radioactive carbon-14 and carbon-12 is constant. Once the material dies, the carbon-14 concentration decreases as the carbon-14 isotope decays, with the half-life of  $t_{1/2} = 5700$  year.**

The analysis of a fossil sample shows that 10% of the original carbon-14 isotope is present. How old was the sample?

$$k = \frac{\ln 2}{t_{1/2}} = \frac{0.693}{5700 \text{ years}} = 1.22 \times 10^{-4} \text{ year}^{-1}$$

$$\ln\left(\frac{N(t)}{N_0}\right) = -kt$$

$$\ln(0.1) = -(1.22 \times 10^{-4} \text{ year}^{-1})t$$

$$t = \frac{-2.302}{-1.22 \times 10^{-4} \text{ year}^{-1}} = 1.89 \times 10^4 \text{ years}$$

**3. What other process can be described formally using the same mathematical equation as  $N(t)$  versus  $t$ ? (Hint: Think kinetics.)**

The  $N(t)$  versus  $t$  relationship is formally the same as the  $[A]$  versus  $t$  in a process with first order kinetics:

$$[A] = [A]_0 e^{-kt}$$

## Multiple Choice Questions

Select the best answer or completion to each of the questions or incomplete statements below.

**1. During radioactive decay:**

- A. Unstable molecules fall apart.
- B. Unstable nuclei fly apart.
- C. There is electron transfer between atoms.
- D. Electrons are always emitted in the form of  $\beta$  radiation.

**2. The half-life of the radioactive decay of an isotope depends on:**

- A. Only the amount of the radioactive isotope present.
- B. The amount of the radioactive isotope present and the decay constant.
- C. The decay constant only.
- D. The temperature.

### Lab 31: Determining the Half-Life of an Isotope

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- 3. The barium-137m isotope has a half-life of 2.55 min, which means:**
- A.** All radioactive material of this isotope decays in 5.30 min.
  - B. Half of the radioactive material of this isotope decays after 2.55 min.**
  - C.** There will be no decay for 2.55 min.
  - D.** Half of the radioactive material of this isotope turns to cesium-137.
- 4. The original number of radioactive atoms can be obtained from:**
- A. The intercept of the  $\ln[N(t)]$  versus  $t$  graph**
  - B.** The half-life of the reaction.
  - C.** The decay constant.
  - D.** Carbon dating.

### Extended Inquiry Suggestions

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A possible extension to this activity would be the investigation of how various materials can shield radioactive radiation. Other than common materials (such as plastic, paper, and wood) some materials can be investigated that are known to be good shields, like lead. A discussion can be held on why shielding from high-energy radiation is important (for example, shielding from radiation due to a nuclear explosion or shielding from x-ray radiation with a lead apron at the dentist).

## Lab 32: The Breathalyzer™ Test for Alcohol

### Objectives

Students understand the chemical oxidation of ethanol by acidic dichromate, as used in Breathalyzer tests for alcohol, and determine the concentration of an ethanol solution.

### Procedural Overview

Students gain experience conducting the following procedures:

- ◆ Carrying out a reaction between ethanol and potassium chromate that uses a catalyst and hot water bath to increase the rate of reaction.
- ◆ Developing a calibration curve using spectroscopic measurements to use for calculating the concentration of an ethanol solution.
- ◆ Advanced preparation of solutions.

### Time Requirement

◆ Preparation time	60 minutes
◆ Pre-lab discussion and activity	30 minutes
◆ Lab activity	90 minutes

### Materials and Equipment

#### *For each student or group:*

- |                                |  |
|--------------------------------|--|
| ◆ Data collection system       | ◆ Beaker, 250-mL   |
| ◆ Colorimeter                  | ◆ Beaker, 1-L  |
| ◆ Cuvette                      | ◆ Ring stand   |
| ◆ Sensor extension cable       | ◆ Clamps (2), utility  |
| ◆ Erlenmeyer flask (7), 125-mL | ◆ Hot plate  |
| ◆ Volumetric flask, 100-mL     | ◆ 15% Sulfuric acid (H <sub>2</sub> SO <sub>4</sub> ), 800 mL <sup>1</sup>   |
| ◆ Graduated pipet, 10-mL       | ◆ Silver nitrate (AgNO <sub>3</sub> ), 15 %, 10 mL <sup>2</sup>  |
| ◆ Graduated pipet, 5-mL        | ◆ 5.10 × 10 <sup>-2</sup> M Potassium dichromate<br>(K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> ), 30 mL <sup>3</sup> |
| ◆ Pipet, plastic, 1-mL         | ◆ Ethanol solution, unknown concentration, 5 mL <sup>4</sup>   |
| ◆ Graduated cylinder, 100-mL   | ◆ Marking pen  |
| ◆ Beaker (2), 25-mL            | ◆ Wash bottle with distilled water   |
| ◆ Beaker, 100-mL               |  |
| ◆ Beaker (2), 400-mL           |  |

<sup>1-4</sup> To prepare the solutions, refer to the Lab Preparation section.

## Concepts Students Should Already Know

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Students should be familiar with the following concepts:

- ◆ Stoichiometry
- ◆ Oxidation-reduction reactions
- ◆ Beer's Law

## Related Labs in This Guide

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Labs conceptually related to this one include:

- ◆ Lab 10: Determine the Equilibrium Constant for a Chemical Reaction
- ◆ Lab 17a: Absorption Spectra
- ◆ Lab 17b: Colorimetric Analysis
- ◆ Lab 25: Order of Reactions

## Using Your Data Collection System

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Students use the following technical procedures in this activity. The instructions for them are in the appendix that corresponds to your PASCO data collection system (identified by the number following the symbol: "◆"). Please make copies of these instructions available for your students.

- ◆ Starting a new experiment on the data collection system ◆<sup>(1.2)</sup>
- ◆ Connecting sensors to the data collection system ◆<sup>(2.1)</sup>
- ◆ Calibrating the colorimeter ◆<sup>(3.2)</sup>
- ◆ Putting the data collection system into manual sampling mode with manually entered data. ◆<sup>(5.2.1)</sup>
- ◆ Monitoring live data without recording ◆<sup>(6.1)</sup>
- ◆ Starting a manually sampled new data set ◆<sup>(6.3.1)</sup>
- ◆ Recording a manually sampled data point ◆<sup>(6.3.2)</sup>
- ◆ Stopping a manually sampled data set ◆<sup>(6.3.3)</sup>
- ◆ Applying a curve fit ◆<sup>(6.3)</sup>
- ◆ Printing ◆<sup>(11.2)</sup>

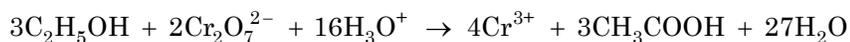
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## Background

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Modern breath analyzers rely on infrared spectroscopy or fuel cell technology (electrochemical oxidation) to measure alcohol content in exhaled air. This was not always the case. The first commercial instrument for estimating a person's blood alcohol content (BAC) by analyzing a breath sample was introduced in 1954 by Dr. Robert Borkenstein. Named the "Breathalyzer," its operation was based on oxidation-reduction (redox) chemistry and absorption photometry. This activity explores the chemical reaction used in the original breathalyzer.

Ethanol (ethyl alcohol) can be oxidized to acetic acid by dichromate as shown in the following equation:



Because the reaction is fairly slow, silver nitrate is added as a catalyst to reduce reaction time.

As ethanol is oxidized, the yellow-orange dichromate ion is reduced to the chromium(III) ion, which is green. Beer's law can be applied to find the concentration of dichromate. We will be using a colorimeter to measure the light absorbance of the solution. To calculate the quantity of ethanol in the unknown, the concentration of dichromate remaining in a solution with a known initial quantity of dichromate reacted with an unknown quantity of ethanol can be used.

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## Pre-Lab Activity

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### *Setting the stage for the activity*

In this activity, you will prepare five solutions with known  $\text{Cr}_2\text{O}_7^{2-}$  concentrations. Using a colorimeter, you will obtain a calibration curve for the five solutions. You will then prepare two solutions with unknown ethanol concentrations and allow the  $\text{Cr}_2\text{O}_7^{2-}$  ions to react with the ethanol in a hot water bath. The concentration of the remaining  $\text{Cr}_2\text{O}_7^{2-}$  ions will be determined with the colorimeter based on the calibration curve.

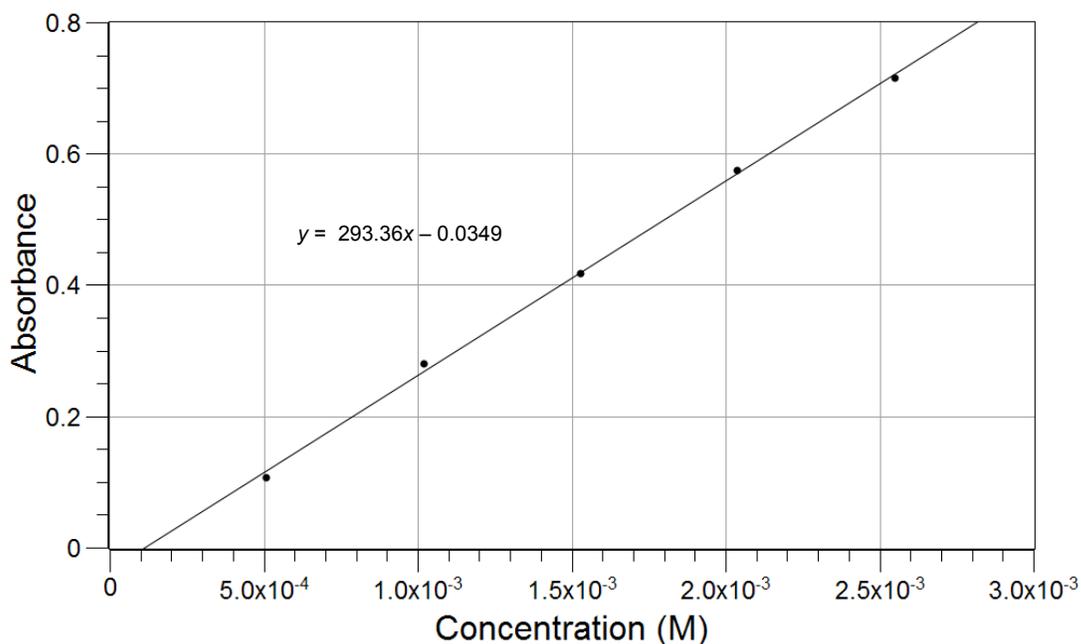
### *Example calculation to try*

In an experiment, the alcohol content of cognac, an alcohol-containing beverage, was determined. 12.00 mL of the cognac sample was diluted to 1 liter as a stock solution. From this stock solution, two unknown solutions were prepared in two 100-mL volumetric flasks as follows:

About 50 mL of 15%  $\text{H}_2\text{SO}_4$  solution were added to both flasks followed by 5.00 mL of  $5.10 \times 10^{-2}$  M  $\text{Cr}_2\text{O}_7^{2-}$  and then 1.00 mL of 150 g/L  $\text{AgNO}_3$  solution. There was some light precipitation which disappeared as the solution was mixed.

To the first unknown solution ("Unknown A"), 1.00 mL of the alcohol stock solution was added. To the second unknown solution ("Unknown B"), 2.00 mL of the alcohol stock solution was added. Both flasks were filled to the mark with 15%  $\text{H}_2\text{SO}_4$ . Both solutions were transferred into 125-mL Erlenmeyer flasks and placed in a 75 °C water bath for 45 min.

While the unknowns were in the water bath, five calibrating solutions were made. Each solution was made in a volumetric flask the same way as the unknowns, except there was no alcohol stock added and the amount of  $\text{Cr}_2\text{O}_7^{2-}$  solution varied: 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL, and 5.00 mL. The following calibration curve was obtained using a colorimeter:



The unknowns were removed from the bath, cooled, and their absorbance measured and recorded in Table 1.

Table 1: Absorbance of the unknown solutions

Solution	A
Unknown A	0.577
Unknown B	0.407

Based on the calibrating equation derived from the slope and y-intercept of the graph, the concentration of the remaining  $\text{Cr}_2\text{O}_7^{2-}$  ions is calculated for Unknown A:

$$y = 296.36x - 0.0349$$

$$x = \frac{y + 0.0349}{296.36}$$

$$\frac{(0.577 + 0.0349)}{296.36 \text{ M}^{-1}} = 2.06 \times 10^{-3} \text{ M}$$

For Unknown B,  $1.49 \times 10^{-3} \text{ M}$  was obtained the same way.

The original concentration of  $\text{Cr}_2\text{O}_7^{2-}$  ions in both solutions was:

$$\left( \frac{5.00 \text{ mL}}{100.00 \text{ mL}} \right) (5.10 \times 10^{-2} \text{ M}) = 2.55 \times 10^{-3} \text{ M}$$

The concentration of the  $\text{Cr}_2\text{O}_7^{2-}$  ions that reacted with the ethanol in Unknown A is:

$$(2.55 \times 10^{-3} \text{ M}) - (2.06 \times 10^{-3} \text{ M}) = (4.90 \times 10^{-4} \text{ M})$$

Using the same calculation, Unknown B was  $1.06 \times 10^{-3}$  M. Knowing the amount of  $\text{Cr}_2\text{O}_7^{2-}$  ions used, based on the stoichiometry, the concentration of the alcohol can be calculated:

$$\left(4.90 \times 10^{-4} \text{ M Cr}_2\text{O}_7^{2-}\right) \left(\frac{3 \text{ mol C}_2\text{H}_5\text{OH}}{2 \text{ mol Cr}_2\text{O}_7^{2-}}\right) = 7.35 \times 10^{-4} \text{ M C}_2\text{H}_5\text{OH}$$

Using the same calculation for Unknown B,  $1.59 \times 10^{-3}$  M ethanol was obtained. Since the concentrations are  $7.35 \times 10^{-4}$  M and  $1.59 \times 10^{-3}$  M, there is  $7.35 \times 10^{-3}$  mol and  $1.59 \times 10^{-2}$  mol of ethanol, respectively in the 100 mL stock solutions. The amount of ethanol in grams can be calculated from its concentration and its formula weight, 46. g/mol:

$$\left(7.35 \times 10^{-3} \text{ mol C}_2\text{H}_5\text{OH}\right) \left(\frac{46 \text{ g C}_2\text{H}_5\text{OH}}{1 \text{ mol C}_2\text{H}_5\text{OH}}\right) = 3.38 \times 10^{-3} \text{ g C}_2\text{H}_5\text{OH}$$

This means there was  $3.38 \times 10^{-3}$  g of ethanol in the 100-mL solution. Considering that this amount of ethanol was in the 1.00 mL of stock solution that was used to make the 100-mL solution, the original 1 liter stock solution had 3.38 g of ethanol. That amount of ethanol was in the 12.00 mL original alcohol. The density of ethanol is 0.800 g/mL, the ethanol concentration of the original alcohol is:

$$\frac{3.38 \text{ g}}{(12.00 \text{ mL})\left(0.800 \frac{\text{g}}{\text{mL}}\right)} \times 100 = 35.2\%$$

Using the same type of calculation for Unknown B, 38.1% ethanol was obtained.

**1. Which color do you think has to be used with the colorimeter if we know that the color of the  $\text{Cr}_2\text{O}_7^{2-}$  solution is yellow?**

According to the color chart, the yellow color comes from the mix of red and green. This means mostly blue is absorbed from the light. Monitoring the "blue" trace seems to be the most appropriate.

**2. The remaining  $\text{Cr}_2\text{O}_7^{2-}$  concentration, after the reaction, was appropriate because it fell approximately in the middle of the calibration curve. However, if you had a sample which had ten times less ethanol, the concentration of  $\text{Cr}_2\text{O}_7^{2-}$  ions would have been outside of the calibration curve. How would you change the experiment to account for such a change in the ethanol concentration?**

Ten times more of the original alcohol solution (120.0 mL instead of 12.00 mL) or ten times more stock solution (10.00 mL instead of 1.00 mL) would have had to be used.

## Lab Preparation

These are the materials and equipment to set up prior to the lab:

- 1. 15%  $\text{H}_2\text{SO}_4$ :** Dissolve 150 mL of concentrated  $\text{H}_2\text{SO}_4$  in some water in a 1-L volumetric flask and fill it to the mark. Please note each group needs about 800 mL. Scale accordingly.
- 2. 15%  $\text{AgNO}_3$ :** Dissolve 15.0 g of  $\text{AgNO}_3$  in 100 mL water.
- 3.  $5.10 \times 10^{-2}$  M  $\text{K}_2\text{CrO}_7$ :** Dissolve 15.0 g of  $\text{K}_2\text{CrO}_7$  in about 300 mL of water in a 1-L Erlenmeyer flask. Slowly add 300 mL of concentrated  $\text{H}_2\text{SO}_4$ . Allow the solution to cool to room temperature.

## Lab 32: The Breathalyzer™ Test for Alcohol

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Transfer the solution into a 1-L volumetric flask and fill it to the mark with water. Mix the solution well.

**DANGER:** Wear a NIOSH-approved respirator with proper cartridges when obtaining the mass of dry  $K_2Cr_2O_7$ .  $K_2Cr_2O_7$  is a known carcinogen and can be fatal if absorbed through the skin. Use gloves to handle this chemical.

- 4. Ethanol, unknown concentration:** Combine 5.0 mL of ethanol with some water in a 1-L volumetric flask and fill it to the mark.

### Safety

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Follow all standard laboratory procedures.

### Sequencing Challenge

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The steps below are parts of the Procedure for this lab activity. They are not in the right order. Determine the proper order and write numbers in the circles that put the steps in the correct sequence.

2	1	4	3	5
Add a catalyst and put the flasks with the unknowns in hot water baths to increase the rate of the reaction.	Create 2 samples with unknown concentrations of ethanol and known concentrations of another reactant, $K_2Cr_2O_7$ .	Measure the absorbance of the two unknown solutions.	While the reactions are in progress, create standard solutions of $Cr_2O_7^{2-}$ and obtain a calibration curve.	Use the calibration curve to calculate the concentration of the ethanol solution.

### Procedure with Inquiry

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After you complete a step (or answer a question), place a check mark in the box () next to that step.

**Note:** When students see the symbol "♦" with a superscripted number following a step, they should refer to the numbered Tech Tips listed in the Tech Tips appendix that corresponds to your PASCO data collection system. There they will find detailed technical instructions for performing that step. Please make copies of these instructions available for your students.

#### Set Up

1.  Obtain about 30 mL of  $5.10 \times 10^{-2}$  M standard potassium dichromate stock solution in a 100-mL beaker. Record the exact concentration of the dichromate solution in Table 3.

2.  Put 5 mL of the ethanol solution of unknown concentration in a 25-mL beaker. Obtain about 800 mL of 15% sulfuric acid solution in a 1-L beaker and 6 mL of silver nitrate solution in a 25-mL beaker.

Note: The use of gloves is recommended. If you get silver nitrate on your skin, wash it off immediately with plenty of water as it leaves black stain on the skin.

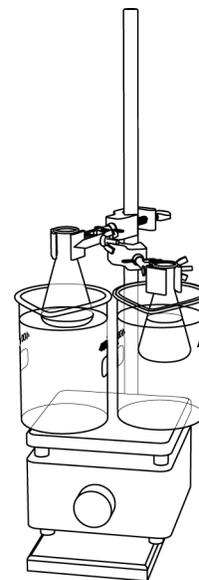
3.  Label five 125-mL Erlenmeyer flasks from “1 mL” to “5 mL”.
4.  Label the other two flasks “Unknown A” and “Unknown B”.
5.  Fill the two 400-mL beakers 3/4 full of water.
6.  Place the beakers on the hot plate and turn it on to maintain the temperature of the water baths between 75°C and 80°C.

### Prepare the ethanol test solutions

7.  Using a 10-mL graduated pipet, transfer 5.0 mL of the potassium dichromate solution to a 100-mL volumetric flask.
8.  Fill the flask about half full with 15% H<sub>2</sub>SO<sub>4</sub>.
9.  Mix the solution by swirling.
10.  Using a plastic pipet, add about 1 mL of silver nitrate solution to the flask and mix the solution again.

**Note:** If any white precipitate is visible, stopper the flask and shake the solution until the precipitate dissolves.

11.  Using a 5-mL graduated pipet, transfer 1.0 mL of the ethanol solution to the volumetric flask with the solution prepared in the previous step and swirl it to mix.
12.  Fill the flask to the calibration mark with 15% H<sub>2</sub>SO<sub>4</sub>.
13.  Stopper the flask and mix the solution by inverting the flask several times.
14.  Transfer this solution to the flask marked “Unknown A”.
15.  Using the ring stand and one of the clamps, place the flask containing the unknown into a water bath, being careful to avoid getting water from the bath in the flask.



## Lab 32: The Breathalyzer™ Test for Alcohol

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- 16.**  Rinse the volumetric flask thoroughly with distilled water and repeat the procedure using 2.0 mL of the same ethanol solution.
- 17.**  Transfer the resulting solution to the flask marked “Unknown B” and use the clamp to place it in the second hot-water bath.
- 18.**  Why do you prepare two unknown solutions?

In order to reduce experimental error, multiple measurements are taken.

- 19.**  What is the advantage of making the concentration of the unknowns different?

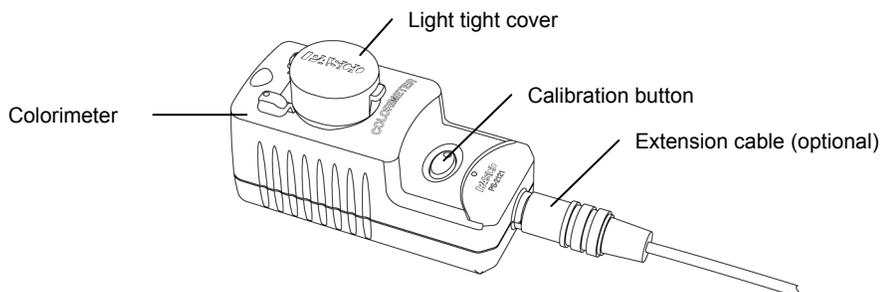
Since we do not know the actual concentration of ethanol, having two samples with significantly different concentrations help in the situation when one concentration turns out to be way off, for example, too much ethanol and not enough  $\text{Cr}_2\text{O}_7^{2-}$ .

- 20.**  Leave the unknown solutions in the water baths for 45 minutes, then remove the flasks from the water baths and allow them to cool to room temperature.

### Prepare the standardized solutions

- 21.**  While the unknown solutions are in the water baths, prepare a set of standard solutions:
- Using a 10-mL graduated pipet, transfer 1.0 mL of the dichromate solution to a rinsed, 100-mL volumetric flask.
  - Using a plastic pipet, add about 1 mL of silver nitrate solution to the flask and mix.
  - Fill the flask about half full with 15%  $\text{H}_2\text{SO}_4$  solution and mix well, being sure that any white precipitate is dissolved.
  - Fill the volumetric flask to the calibration mark with additional 15%  $\text{H}_2\text{SO}_4$  solution.
  - Mix well and transfer the solution from the volumetric flask to the flask labeled “1 mL”.
  - Rinse the volumetric flask thoroughly with distilled water and prepare a new solution following the same steps but adding 2.0 mL of dichromate solution.
  - Follow the same procedure to prepare solutions containing 3.0 mL, 4.0 mL and 5.0 mL of dichromate solution.
- 22.**  Start a new experiment on the data collection system.  $\diamond^{(1,2)}$
- 23.**  Connect the colorimeter to the data collection system using a sensor extension cable.  $\diamond^{(2,1)}$
- 24.**  Put the data collection system into manual sampling mode with manually entered data. Name the manually entered data “Absorbance”.  $\diamond^{(5,2,1)}$

25.  Calibrate the colorimeter using 15% sulfuric acid as a blank. ♦<sup>(3.2)</sup>



**Important:** Always make sure that the cuvette is clean and dry on the outside before placing it into the colorimeter.

26.  Start a new, manually sampled data set. ♦<sup>(6.3.1)</sup>

### Collect Data

#### Obtain the calibration curve

27.  Measure the absorbance of the five standardized solutions, starting with the one labeled “1 mL” and ending with the one labeled “5 mL”, following the steps below.
- Rinse the cuvette twice with a small portion of the first solution and then fill the cuvette two-thirds full. Wipe the cuvette clean and dry and place it into the colorimeter.
  - Why do you have to rinse the cell with some of the solution?

If there is any residual water in the cuvette, it will dilute the concentration of the solution and falsify the data.

- After the reading stabilizes, record a data point. ♦<sup>(6.3.2)</sup>
- Dispose of the solution appropriately and rinse the cell thoroughly with water.
- Why do you think it is important to rinse the cell thoroughly between measurements?

You need to rinse the cell to avoid contamination of the solutions.

- Stop the data set. ♦<sup>(6.3.3)</sup>

28.  Why did you have to prepare multiple calibrating solutions?

Calibration is usually done to measure the response of an instrument to the variation of an experimental parameter over a range of that parameter. In this case, you measure the response of the colorimeter over a concentration range of the  $\text{Cr}_2\text{O}_7^{2-}$ . Also, having multiple points that reflect the relationship between the signal and the varied parameter helps minimize the experimental error.

29.  Apply a linear fit to the data. ♦<sup>(9.5)</sup>

30.  Print the graph. ♦<sup>(11.2)</sup>

31.  Set the data collection system to monitor live data without recording. ♦<sup>(6.1)</sup>

**Measure the absorbance of the unknowns**

32.  Obtain the absorbance of Unknown A and then Unknown B as follows:

- a. Clean the cuvette and then rinse it with the cooled solution, then fill the cuvette with the solution.
- b. Place the cell into the colorimeter and record the value in Table 3.

33.  Save your experiment ♦<sup>(11.1)</sup> and clean up according to your teacher's instructions.

**Data Analysis**

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1.  Based on the concentration of the stock dichromate solution, calculate the concentration of the five standard solutions and record the values in Table 2.

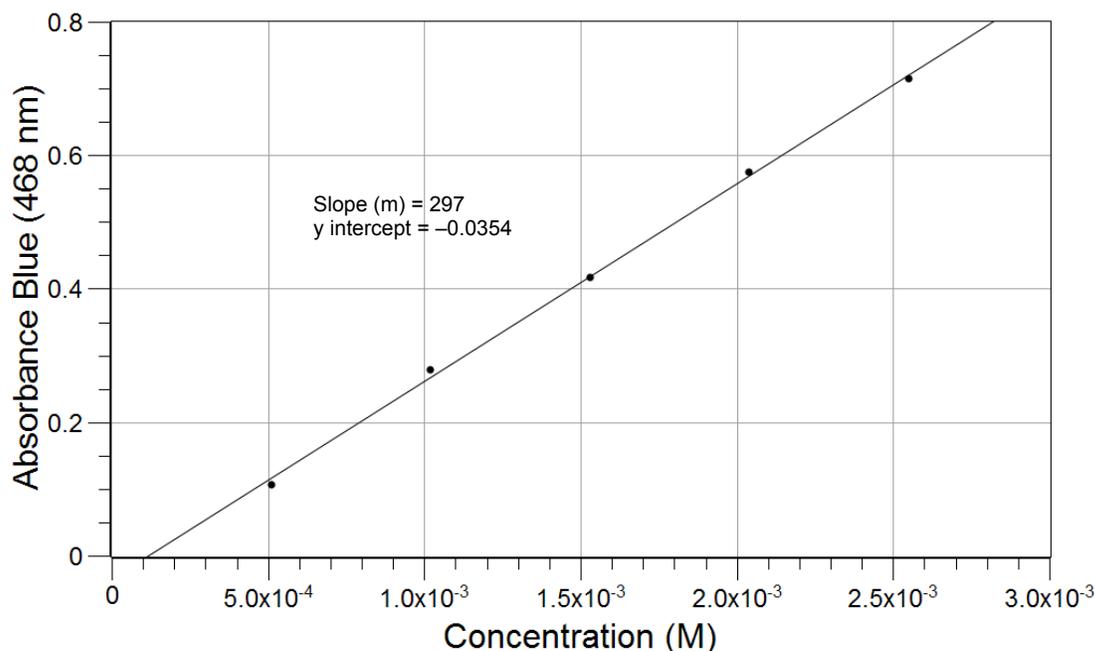
For the solution that was made with 1.0 mL standard:

$$\left(\frac{1.0 \text{ mL}}{100.00 \text{ mL}}\right)(5.1 \times 10^{-2} \text{ M}) = 5.1 \times 10^{-4} \text{ M}$$

Table 2: Potassium dichromate concentration in the standardized solutions

Volume of $\text{Cr}_2\text{O}_7^{2-}$ standard (mL)	Concentration (M)
1.0	$5.1 \times 10^{-4} \text{ M}$
2.0	$1.0 \times 10^{-3} \text{ M}$
3.0	$1.5 \times 10^{-3} \text{ M}$
4.0	$2.0 \times 10^{-3} \text{ M}$
5.0	$2.5 \times 10^{-3} \text{ M}$

2.  Sketch or paste the calibration curve below and record the slope and y-intercept on the graph.



3.  Calculate the initial concentration of  $\text{Cr}_2\text{O}_7^{2-}$  in the reaction flask. Record this value in Table 3.

$$\left(\frac{5.00 \text{ mL}}{100.00 \text{ mL}}\right)(5.10 \times 10^{-2} \text{ M}) = 2.55 \times 10^{-3} \text{ M}$$

4.  What is the calibrating equation for this calibration curve?

The calibrating equation is:  $y = 297x - 0.0354$

5.  Based on the calibrating equation, calculate the concentration of the remaining  $\text{Cr}_2\text{O}_7^{2-}$  ions for Unknown A and Unknown B. Record these values in Table 3.

For Unknown A,  $y = 0.567$ , so solving for the  $\text{Cr}_2\text{O}_7^{2-}$  ion concentration  $x$  results in

$$\frac{(0.567 + 0.0354)}{297 \text{ M}^{-1}} = 2.03 \times 10^{-3} \text{ M}$$

6.  Calculate the concentration of the  $\text{Cr}_2\text{O}_7^{2-}$  ions that reacted with the ethanol for Unknown A and Unknown B. Record these values in Table 3.

For Unknown A:

$$(2.55 \times 10^{-3} \text{ M}) - (2.03 \times 10^{-3} \text{ M}) = 5.2 \times 10^{-4} \text{ M}$$

## Lab 32: The Breathalyzer™ Test for Alcohol

7.  Knowing how much of the  $\text{Cr}_2\text{O}_7^{2-}$  ions was used, based on the stoichiometry calculate the concentration of the alcohol solutions labeled “Unknown A” and “Unknown B”.

Also determine the amount of ethanol in the 100-mL solution. Record these values in Table 3.

For Unknown A:

$$\left(5.20 \times 10^{-4} \text{ M Cr}_2\text{O}_7^{2-}\right) \left(\frac{3 \text{ mol C}_2\text{H}_5\text{OH}}{2 \text{ mol Cr}_2\text{O}_7^{2-}}\right) = 7.80 \times 10^{-4} \text{ M C}_2\text{H}_5\text{OH}$$

$$\left(7.80 \times 10^{-4} \frac{\text{mol}}{\text{L}} \text{ C}_2\text{H}_5\text{OH}\right) \times (0.100 \text{ L}) = 7.80 \times 10^{-5} \text{ mol C}_2\text{H}_5\text{OH}$$

8.  Determine the mass of ethanol added to the unknown solutions, the mass of ethanol in the 100-mL stock solution, and calculate the average of the mass of ethanol in the 100-mL stock solution for the two unknown solutions. Record your data in the table below.

For Unknown A, the mass of ethanol that had been added to the unknown solution is

$$\left(7.80 \times 10^{-5} \text{ mol C}_2\text{H}_5\text{OH}\right) \left(\frac{46.1 \text{ g C}_2\text{H}_5\text{OH}}{1 \text{ mol C}_2\text{H}_5\text{OH}}\right) = 3.60 \times 10^{-3} \text{ g C}_2\text{H}_5\text{OH}$$

This is the amount in 1 mL of the stock solution, so the mass of ethanol in the 1-L stock solution is  $(3.60 \times 10^{-3} \text{ g/mL}) \times (1000 \text{ mL/L}) = 3.60 \text{ g C}_2\text{H}_5\text{OH}$ . The average is  $(3.60 \text{ g} + 3.36 \text{ g})/2 = 3.48 \text{ g}$ .

Table 3: Determination of the amount of ethanol in the two solutions

Parameters	Unknown A	Unknown B
Initial concentration of $\text{Cr}_2\text{O}_7^{2-}$ (M) in the 1-L stock solution	$5.10 \times 10^{-2}$	
Initial concentration of $\text{Cr}_2\text{O}_7^{2-}$ (M) in the reaction flask (M)	$2.55 \times 10^{-3}$	
Absorbance	0.567	0.435
Remaining concentration of $\text{Cr}_2\text{O}_7^{2-}$ (M)	$2.03 \times 10^{-3}$	$1.58 \times 10^{-3}$
Amount of $\text{Cr}_2\text{O}_7^{2-}$ reacted with ethanol (M)	$5.20 \times 10^{-4}$	$9.70 \times 10^{-4}$
Equivalent ethanol concentration (M)	$7.80 \times 10^{-4}$	$1.46 \times 10^{-3}$
Amount of ethanol in the 100-mL solution (mol)	$7.80 \times 10^{-5}$	$1.46 \times 10^{-4}$
Mass of ethanol in the 100-mL “Unknown” solution (g)	$3.60 \times 10^{-3}$	$6.73 \times 10^{-3}$
Mass of ethanol in the 1-L stock solution (g)	3.60	3.36
Average mass of ethanol in 1-L stock solution (g)	3.48	

---

## Analysis Questions

---

- 1. How would significant evaporation of water during the heating of the unknowns influence your results?**

Less solvent would yield a higher  $\text{Cr}_2\text{O}_7^{2-}$  concentration (indicated by the absorbance). This would imply a lower amount of  $\text{Cr}_2\text{O}_7^{2-}$  was consumed, which would result in calculating less ethanol than there really was.

- 2. If the result demonstrated a lower ethanol concentration than the outcome should have been, how would you improve your results?**

Reducing evaporation during the heating would increase the amount of ethanol that was detected. Also, the reaction may not have gone to completion, being a slow reaction. Extending the reaction time of the solutions in the hot water bath could improve the results.

- 3. How many milliliters of 100% ethanol was used to make your unknown, if the density of ethanol is 0.800 g/mL?**

$$\frac{0.348 \text{ g}}{0.800 \frac{\text{g}}{\text{mL}}} = 0.435 \text{ mL}$$

---

## Synthesis Questions

---

Use available resources to help you answer the following questions.

- 1. Can you think of any other way to follow the reaction with the colorimeter? (Hint: Consider what other species present in the reaction mixture would absorb in the visible range.)**

The Cr(III) ions are green and would be suitable to follow the reaction.

- 2. How would you have to change the experiment to utilize the option above?**

The calibrating solutions would have to be Cr(III) solutions and we would measure the Cr(III) concentration, which is proportional to the amount of alcohol present in the solution, rather than the amount of unreacted reactant.

- 3. For this change, which trace would you utilize to make your measurements? (Hint: Remember the color of the species in question.)**

Since the Cr(III) is green, there should be significant absorption in the red and in the blue range. Considering that the remaining  $\text{Cr}_2\text{O}_7^{2-}$  ions also absorb in the blue range, it would interfere with the detection of Cr(III). So the red trace is a better choice.

### Multiple Choice Questions

---

Select the best answer or completion to each of the questions or incomplete statements below.

- Which statement is *not* correct?
  - Ethanol reacts with  $\text{Cr}_2\text{O}_7^{2-}$  in a redox reaction.
  - One of the products of the reaction is Cr(III).
  - The amount of the remaining  $\text{Cr}_2\text{O}_7^{2-}$  ions is measured.
  - AgNO<sub>3</sub> is used to oxidize the ethanol.**
- The blue trace is used to detect the  $\text{Cr}_2\text{O}_7^{2-}$  ions because:
  - $\text{Cr}_2\text{O}_7^{2-}$  is blue .
  - $\text{Cr}_2\text{O}_7^{2-}$  absorbs in the blue range.**
  - $\text{Cr}_2\text{O}_7^{2-}$  is yellow.
  - $\text{Cr}_2\text{O}_7^{2-}$  absorbs in the yellow range.
- Which statement is correct regarding the  $\text{Cr}_2\text{O}_7^{2-}$  ion?
  - It is used to catalyze the reaction.
  - It is employed in the stoichiometric ratio with ethanol.
  - It is added in a known quantity from which some reacts with the ethanol present and the rest is detected with the colorimeter.**
  - It is added in a known quantity from which it is detected with the colorimeter after which some is used up by the ethanol.
- Why were the unknowns kept in a 75 to 80 °C water bath?
  - To catalyze the reaction.
  - To accelerate the reaction.**
  - To help the dissolution of the reagents.
  - To help get more accurate colorimetric measurements.

## Extended Inquiry Suggestions

This method can be utilized or adapted to measure the ethanol content of a regular alcoholic beverage. In the example below, the ethanol content of cognac is determined. The bottle shows a 43% ethanol content. The same analysis was performed as before, except based on the predicted ethanol content, 12.00 mL of the beverage was used:

	Unknown A	Unknown B
Initial concentration of $\text{Cr}_2\text{O}_7^{2-}$ in the reaction flask (M)	$2.55 \times 10^{-3}$	
Absorbance	0.577	0.407
Remaining concentration of $\text{Cr}_2\text{O}_7^{2-}$ (M)	$2.06 \times 10^{-3}$	$1.49 \times 10^{-3}$
Amount of $\text{Cr}_2\text{O}_7^{2-}$ reacted with ethanol (M)	$4.90 \times 10^{-4}$	$1.05 \times 10^{-3}$
Equivalent ethanol concentration (M)	$7.35 \times 10^{-4}$	$1.59 \times 10^{-3}$
Amount of ethanol in the 100-mL solution (mol)	$7.35 \times 10^{-4}$	$1.59 \times 10^{-3}$
Mass of ethanol in the 100-mL solution (g)	$3.35 \times 10^{-3}$	$7.30 \times 10^{-3}$
Volume taken from the 1-L stock solution (mL)	1	2
Mass of ethanol in the 1-L solution (g)	3.35	3.65
Average mass of ethanol in the 1-L stock solution (g)	3.50	

The solution therefore had 3.50 g/L of ethanol. The concentration of the original beverage was:

$$\frac{3.50 \text{ g}}{(12.00 \text{ mL})(0.800 \frac{\text{g}}{\text{mL}})} \times 100 = 36.5\%$$

The error of the measurement:

$$\text{Percent Error} = \left| \frac{43.00 - 36.46}{43.00} \right| \times 100 = 15.2\%$$



# Appendix A: SPARK Science Learning System Tech Tips

## Section 1: Starting an experiment

---

### 1.1 Opening a file

1. In the Home screen, touch **OPEN**.  
*Result:* A list of folders appears.
2. Touch the folder containing the file.
3. If necessary, touch a subfolder.
4. Touch the file that you would like to open.
5. Touch **OK**.

*Task result:* The file opens.

---

### 1.2 Starting a new experiment

Experiments start at the Home screen. Do one of the following to open the Home screen.

- ◆ If the SPARK Science Learning System (SPARK) is off, press and hold the power button on the bottom to turn it on and wait for it to boot up.
- ◆ If the SPARK is already on, but the Home screen is not open, touch the **Home** button.



*Task result:* The Home screen displays live readings from any connected sensor.

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## Section 2: Setting up measurements and data collection

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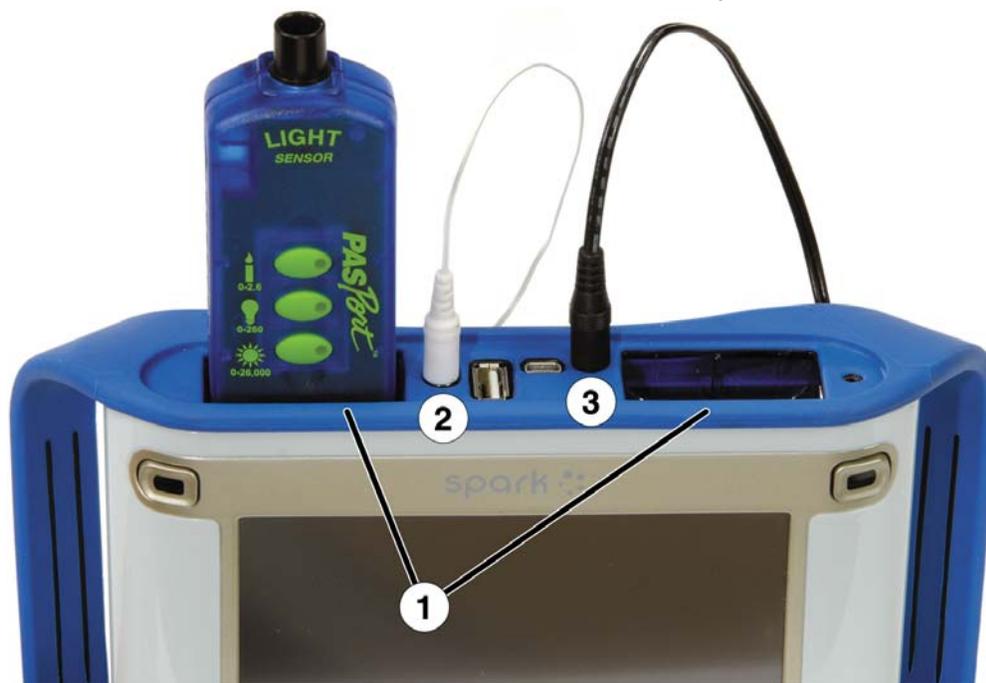
### 2.1 Connecting a sensor to the SPARK

- ◆ If you have a PASPORT sensor, plug it into either of the PASPORT ports on the SPARK.

- ◆ If you have a temperature probe (fast-response or stainless steel), plug it into the temperature port.
- ◆ If you have a voltage probe, plug it into the voltage port.

*Task result:* The SPARK detects the sensor and adds it to your experiment.

1. PASPORT ports.
2. Temperature port.
3. Voltage port.



---

## 2.2 Connecting multiple sensors to the SPARK

Complete any or all of the following steps.

- ◆ Plug PASPORT sensors into either or both of the PASPORT ports.
- ◆ Connect a temperature probe to the temperature port.
- ◆ Connect a voltage probe to the voltage port.

*Task result:* The SPARK detects all connected sensors and adds them to your experiment.

---

## 2.3 Programming the SPARK to calculate lung volume

1. Touch the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

2. Touch **CALCULATED DATA**.

*Result:* The calculator screen opens.

3. Complete the substeps below to enter this expression:

LungVolume = V0-[Total Flow(L)]

- a. Touch the **Letters** button to switch to the letters keyboard.



- b. Type: LungVolume
- c. Touch the **Numbers** button to switch back to the main calculator keyboard.



- d. Type: =
- e. Touch the **Letters** button to switch back to the letters keyboard.



- f. Type: V0-
- g. Touch the **Numbers** button to switch back to the main calculator keyboard.



- h. Touch **Measurements**.
- i. Touch **Total Flow**.

*Result:* This expression now appears in the entry box of the calculator screen: LungVolume = V0-[Total Flow(L)]

- 4. Touch **Return**.

*Result:* A second expression appears in the entry box: V0 =

- 5. Type the test subject's functional residual capacity (FRC) in liters.  
If you do not know the test subject's FRC, type: 2.5

The second line in the entry box should now be similar to: V0 = 2.5

- 6. Touch **Return**.

*Result:* The calculation is complete.



- 7. Touch **DONE** to close the calculator screen.
- 8. Touch **OK** to close the Experiment Tools screen.

*Task result:* The Lung Volume calculation is now available for viewing in a graph, table, or other display.

---

## Section 3: Calibrating sensors

---

### 3.1 Calibrating a carbon dioxide gas sensor

Once the Carbon Dioxide Gas sensor has been calibrated using this method, it retains its calibration, even after it has been unplugged.

1. Take the sensor and an empty sample bottle outdoors.  
If necessary, unplug the sensor from the SPARK.
2. Fill the sample bottle with fresh outdoor air.
3. Insert the probe of the sensor into the bottle and push the rubber stopper of the sensor into the neck of the bottle.
4. If you have disconnected the sensor, return to the SPARK and connect the sensor to it.
5. Press the **Calibrate** button on the sensor and hold it for three seconds.  
*Result:* The green light illuminates indicating that calibration is in progress.
6. Wait approximately one minute.  
When the green light flashes slowly, calibration is finished. If the light flashes rapidly (five times per second), an error has occurred and the sensor has not been calibrated.

---

### 3.2 Calibrating a colorimeter

1. Place a cuvette filled with distilled water in the colorimeter and close the lid.
2. Press the **Calibration** button on the sensor.  
*Result:* The light in the button illuminates indicating that calibration is in progress.
3. Wait for the light to turn off.

---

### 3.3 Calibrating a dissolved oxygen sensor

1. Obtain barometric pressure and temperature at your location.
2. Refer to the solubility table included with the sensor and find the standard dissolved oxygen value for the temperature and barometric pressure at your location.
3. Touch the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

4. Touch **CALIBRATE SENSOR**.  
*Result:* The Calibrate Sensor: Select Measurement screen opens.
5. Touch the **Sensor** box and select the sensor that the dissolved oxygen probe is connected to.
6. Touch the **Measurement** box and select **Dissolved Oxygen (mg/L)**.
7. Touch the **Calibration Type** box and select **1 point (Adjust Slope Only)**.
8. Touch **NEXT**.  
*Result:* The Calibrate Sensor screen opens

- Under **Calibration Point 2** touch the **Standard Value** box and enter the value that you determined from the solubility table.
- Place about 5 mL (to a height of about 1 cm) of deionized water into a clean soaker bottle. Slip the cap and O-ring of the soaker bottle over the end of the probe.
- Insert the probe into the soaker bottle and screw on the lid. Adjust the height of the probe to about 2 cm above the top of the water.
- Shake the soaker bottle vigorously for about 10 seconds. Shake off any large water drops from the membrane at the end of the probe.
- Under **Calibration Point 2** touch **Read From Sensor**.  
*Result:* The value measured by the sensor is transferred to the **Sensor Value** box.
- Touch **OK** to exit the Calibrate Sensor screen.
- Touch **OK** to exit the Experiment Tools screen.

---

### 3.4 Calibrating a drop counter

The drop counter is calibrated *after* data is taken, using the SPARK's calculator to correlate the number of drops counted to the volume of liquid dispensed from the burette. The following steps illustrate the calibration procedure. Note that in performing the experiment, these steps will be interspersed with other experiment procedure steps.

Note: Using this method, you will ignore the "Volume" measurement output directly from the sensor. Instead, volume is calculated by the SPARK. This method takes into account the fact that drop size may be different each time the burette is used. By measuring the total volume dispensed during the actual experiment, a more accurate correlation between volume and drop count is obtained.

- Set up the drop counter on a stand with a liquid-filled burette, a beaker, stir-plate, and other equipment.  
See your lab instructions for details.
- Write down the initial volume of liquid in the burette.
- Touch the **Start** button.



- Slowly turn the stopcock to start delivering liquid at about 2 drops per second.
- After the necessary amount of liquid has been dispensed from the burette, close the stopcock.
- Touch the **Stop** button.



- Read the final volume of liquid in the burette and determine the net volume that was dispensed.
- Read the final drop count (the total number of drops dispensed) in a graph or table display.

9. Touch the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

10. Touch **CALCULATED DATA**.

*Result:* The calculator screen opens.

11. Complete the substeps below to enter an expression similar to this:

volume = [Drop Count (drops)] \* 100/3050

- a. Touch the **Letters** button to switch to the letters keyboard.



- b. Type: volume

- c. Touch the **Numbers** button to switch back to the main calculator keyboard.



- d. Type: =

- e. Touch **Measurements**.

- f. Touch **Drop Count**.

- g. Type something similar to: \* 100/3050

*Note:* In this example, “100” is the total volume dispensed (in mL) and “3050” is the final drop count. When you type the expression, use your own values for these two quantities.

*Result:* An expression similar to this now appears in the entry box of the calculator screen: volume = [Drop Count (drops)] \* 100/3050

12. Touch **Return**.

*Result:* The calculation is complete.

13. Touch **DONE** to close the calculator screen.

14. Touch **OK** to close the Experiment Tools screen.

*Task result:* The calibrated Volume calculation is now available for viewing in a graph, table, or other display.

---

### 3.5 Calibrating an oxygen gas sensor

Once the Oxygen Gas sensor has been calibrated using this method, it retains its calibration, even after it has been unplugged.

1. Take the sensor and an empty sample bottle outdoors.  
If necessary, unplug the sensor from the SPARK.
2. Fill the sample bottle with fresh outdoor air.
3. Insert the probe of the sensor into the bottle and push the rubber stopper of the sensor into the neck of the bottle.

4. If you have disconnected the sensor, return to the SPARK and connect the sensor to it.
5. Press the **CAL (20.9%)** button on the sensor and hold it for three seconds.

*Task result:* The green light flashes on and off for 4 seconds, indicating that calibration is in progress. When the light stops flashing, calibration is finished. If the light flashes rapidly (5 times per second), an error has occurred and the sensor has not been calibrated.

### 3.6 Calibrating a pH sensor

You will need buffer solutions of pH 4 and pH 10 and deionized water.

1. Touch the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

2. Touch **CALIBRATE SENSOR**.

*Result:* The Calibrate Sensor: Select Measurement screen opens.

3. Touch the **Sensor** box and select the sensor that the pH probe is connected to.
4. Touch the **Measurement** box and select **pH**.
5. Touch the **Calibration Type** box and select **2 point (Adjust Slope and Offset)**.
6. Touch **NEXT**.

*Result:* The Calibrate Sensor screen opens

7. Place the pH probe into the pH 4 buffer solution and wait for about 1 minute.
8. Under **Calibration Point 1** touch the **Standard Value** box and enter 4 (the known pH of the buffer solution).
9. Under **Calibration Point 1**, touch **Read From Sensor**.

*Result:* The value measured by the sensor is transferred to the **Sensor Value** box.

10. Rinse the probe with deionized water.
11. Place the pH probe into the pH 10 buffer solution and wait about 1 minute.
12. Under **Calibration Point 2** touch the **Standard Value** box and enter 10 (the known pH of the buffer solution).
13. Under **Calibration Point 2**, touch **Read From Sensor**.

*Result:* The value measured by the sensor is transferred to the **Sensor Value** box.

14. Touch **OK** to close the Calibrate Sensor screen.
15. Touch **OK** to close the Experiment Tools screen.

### 3.7 Calibrating a turbidity sensor

1. Place a cuvette filled with distilled water in the turbidity sensor and close the lid.

2. Press the **Calibration** button on the sensor.  
*Result:* The green light in the button illuminates.
  3. When the light starts blinking, replace the cuvette with the standard 100 NTU cuvette (included with the sensor) and close the lid.
  4. Press the button again.  
*Result:* The light in the button illuminates.
- Task result:* When the light turns off, calibration is complete.
- 

### 3.8 Calibrating an ethanol sensor

You will need a solution of 1% ethanol in water. The solution should be at the same temperature as the solutions to be measured.

Once the sensor has been calibrated using this method, it retains its calibration, even after it has been unplugged.

1. Connect the sensor to the SPARK and wait about 10 minutes for the probe to warm up.
2. Place the probe about 1 cm above the 1% ethanol solution.
3. Observe the ethanol concentration reading on the SPARK and wait until the reading stabilizes.  
  
(You can observe the reading in the Home screen, or in a digits display while recording data.)
4. Press and hold the **1% CAL** button on the sensor for 4 seconds.

*Task result:* Immediately after a successful calibration, the sensor's output reads 1%, and the button is illuminated.

## Section 4: Sensor operations

---

### 4.1 Using a colorimeter with a specific color of light

The colorimeter makes measurements for four different colors; be sure to choose the measurements for the appropriate color.

The colorimeter contains four separate light sources of different colors: red (660 nm), orange (610 nm), green (565 nm), and blue (468 nm). Internally, the colorimeter uses all four light sources simultaneously and makes separate measurements for each color. However, in most experiments, you will be concerned only with one color and ignore the measurements associated with other colors. Your lab instructions may tell you which color to use, or you may need to determine the appropriate color on your own.

For each color there are two measurements: transmittance and absorbance.

As you proceed with your experiment, whenever you set up data displays or do certain other tasks, you will see a list of all eight measurements made by the colorimeter; be sure always to choose the transmittance or absorbance measurement for the appropriate color.

---

### 4.2 Setting up a conductivity sensor for a particular sensitivity

When using a conductivity sensor, it is important to select a sensitivity appropriate to the solutions that you plan to test. If the selected sensitivity is too high, the

sensor may lack the range necessary to measure your most concentrated solutions. However, if the range is larger than necessary, the measurement precision may not be high enough to detect small conductivity changes or differences.

Complete these steps to select the appropriate sensitivity and range:

1. Determine or estimate the highest concentration that you will need to measure.

Use this chart and other resources.

Solution	Conductivity ( $\mu\text{S/cm}$ )
Ultrapure water	0.05 to 0.75
Drinking water	50 to 1500
Ocean water	~53000

2. Select the range that best matches your measurement requirements.

The first column of this chart shows available ranges.

Measurement Range ( $\mu\text{S/cm}$ )	1x Probe Range Selection	10x Probe Range Selection
0 to 100		-
0 to 1000		
0 to 10000		
0 to 100000	-	

3. Select an appropriate probe (either 1x or 10x) and connect it to the sensor.
4. Use the buttons on the sensor to select an appropriate range.

Lights in the buttons indicate which range is selected.

Note: As you proceed with your experiment, whenever you set up data displays or do certain other tasks, you will see a list two different measurements made by the sensor: one for the 1x probe and one for the 10x probe; be sure always to choose the measurement that matches the connected probe.

### 4.3

Setting up a rotary motion sensor to measure linear position using the “rack” setting

The default Linear Position measurement assumes the large pulley attached to the rotary motion sensor. This measurement should be multiplied by 0.532 to give linear position data for a toothed rack inserted into the rotary motion sensor. Complete these steps to program the SPARK to make that calculation:

1. Touch the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

2. Touch **CALCULATED DATA**.

*Result:* The calculator screen opens.

3. Complete the substeps below to enter this expression:

RackLinearPosition = [Linear Position(m)]\*0.532

- a. Touch the **Letters** button to switch to the letters keyboard.



- b. Type: RackLinearPosition

- c. Touch the **Numbers** button to switch back to the main calculator keyboard.



- d. Type: =

- e. Touch **Measurements**.

- f. Touch **Linear Position**.

- g. Touch ×

- h. Type: 0.532

*Result:* This expression now appears in the entry box of the calculator screen: RackLinearPosition = [Linear Position(m)]\*0.532

4. Touch **Return**.

*Result:* The calculation is complete.

5. Touch **DONE** to close the calculator screen.

6. Touch **OK** to close the Experiment Tools screen.

*Task result:* The RackLinearPosition calculation is now available for viewing in a graph, table, or other display.

## Section 5: Data measurement setup

---

### 5.1 Changing the sampling rate

1. Touch the **Sampling Options** button.



*Result:* The Sampling Option screen opens.

2. Touch the **Sample Rate Unit** box and select **Hz, seconds, minutes, or hours**.

If you select units of Hz, the value indicates the number of samples per second. If you select units of seconds, minutes, or hours, the value indicates the time between samples.

3. Touch the **Sample Rate** box and select a value.

4. Touch **OK** to close the Sampling Options screen.

## 5.2 Putting the SPARK into manual sampling mode

1. Touch the **Sampling Options** button.



*Result:* The Sampling Option screen opens.

2. Touch **Manual**.
3. Touch **OK** to close the Sampling Options screen.

*Task result:* The SPARK is now ready to record manually sampled data. ♦(6.3)

### 5.2.1 Putting the SPARK into manual sampling mode with manually entered data

1. Do one of the following to open the page-build screen.
  - ♦ If a SPARKlab is open, touch the **New Page** button.



- ♦ If the Home screen is open, touch **BUILD**.

*Result:* The page-build screen opens.

**Page-build screen:** 1. Measurements list. 2. Display buttons. 3. Preview section.



2. Create an empty user-entered data set:
  - a. In the measurements list under **User-entered Number Data** or under **User-entered Text Data**, touch **Create Data Set**.
 

*Note:* You may need to scroll the list to see these options.

*Result:* The Define the Data Set screen opens.
  - b. Touch the **Measurement Name** box, type a name that describes the data or text that you plan to enter manually, and touch **OK**.
  - c. Optionally, if you are creating a numerical data set, touch the **Unit Name** box, type the units of the manually entered data, and touch **OK**.
  - d. Touch **OK** to return to the page-build screen.
 

*Result:* The user-entered data set that you created now appears in the measurements list on the page-build screen.
3. In the measurements list, touch the data set that you just created to select it.
 

*Result:* The selected data set is highlighted.

4. In the measurements list, touch a sensor measurement to select it.  
Data from this sensor measurement will be recorded alongside your user-entered data.

*Result:* There are now two highlighted items in the measurements list: the user-entered data set and a sensor measurement.

5. Touch the **Table** button.



6. Touch **OK**.

*Result:* A table prepared to display the manually entered data and sensor data appears.

7. Touch the **Sampling Options** button.



*Result:* The Sampling Option screen opens.

8. Touch **Manual**.
9. Touch **OK** to close the Sampling Options screen.

*Task result:* The SPARK is now ready to record manually sampled data with manually entered data. <sup>◆(6.3)</sup>

---

## 5.2.2

Putting the SPARK into manual sampling mode without manually entered data

1. Touch the **Sampling Options** button.



*Result:* The Sampling Option screen opens.

2. Touch **Manual**.
3. Touch **OK** to close the Sampling Options screen.

*Task result:* The SPARK is now ready to record manually sampled data. <sup>◆(6.3)</sup>

---

## 5.3

Changing the units of a measurement

Complete these steps to change the units of a measurement displayed in an existing graph, digits display, table, or meter:

1. Touch the **Tools** button of a graph, digits display, table, or meter to open the tools palette.



2. Touch the **Properties** button to open the properties screen.



3. If you are changing the units of a measurement in a table, touch the **Column** box and select the column containing the measurement.

Columns are numbered 1, 2, 3, and so on from left to right.

4. Touch the **Units** box and select a unit of measure.
5. Touch **OK**.

*Task result:* The display shows the measurement with the selected units.

## 5.4 Changing the number of digits with which a variable is displayed

1. Touch the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

2. Touch **DATA PROPERTIES**.

*Result:* The Data Properties screen opens.

3. Touch the **Measurement** box and select a measurement or other variable.
4. Touch the **Number Format** arrow.

*Result:* The number format options appear.

5. Touch the **Number Style** box and select **Fixed Precision**.
6. Use the **Digits** arrows to select the number of digits to be displayed after the decimal point.
7. Touch **OK** to close the Data Properties screen.
8. Touch **OK** to close the Experiment Tools screen.

## Section 6: Collecting and working with data

### 6.1 Monitoring live data

1. Display the measurement that you would like to monitor in a digits display.<sup>(7.3.1)</sup>
2. Touch the **Sampling Options** button.



*Result:* The Sampling Option screen opens.

3. Touch **Manual**.
4. Touch **OK** to close the Sampling Options screen.
5. Touch the **Start** button.



*Result:* The live readings are displayed.

6. To stop monitoring, touch the **Stop** button.



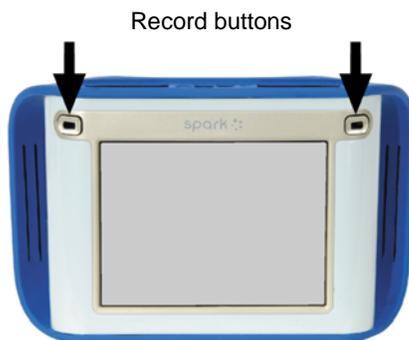
## 6.2 Recording a run of data

Complete these steps to record a data run in periodic sampling mode:

1. Touch the **Start** button.



Alternatively, you can press one of the record buttons instead of touching the on-screen **Start** button.



*Result:* The SPARK creates a new data run and starts recording data points into it.

2. To stop recording data, touch the **Stop** button.



Alternatively, you can press one of the record buttons instead of touching the on-screen **Stop** button.

*Result:* The SPARK stops recording data.

Repeat these steps to record another data run.

---

## 6.3 Recording a set of manually sampled data

If the SPARK is in manual sampling mode <sup>(5.2)</sup>, complete these steps to record a data set:

1. If you plan to type in user-entered data while you manually sample data from a sensor, turn to a page in your SPARKlab where you will be able to see the user-entered data and sensor data in a table.
2. Touch the **Start** button.



Alternatively, you can press one of the record buttons instead of touching the on-screen **Start** button.

*Result:* The SPARK creates a new data set. Live data appear in the data displays. The record buttons start flashing to indicate that the SPARK is ready to be triggered.

3. When you are ready to trigger the recording of a data point, touch the **Keep** button.



Alternatively, you can press one of the record buttons instead of touching the on-screen **Keep** button.



*Result:* The SPARK records a single value from each measurement.

4. Optionally, if you have set up the SPARK to accept manually entered data along with the sensor data <sup>(5.2.1)</sup>, complete these substeps to enter a number or text:
  - a. If the table tool palette is not already open, touch the **Table Tools** button.



- b. If it is not already highlighted in the tool palette, touch the Select button.



*Result:* The **Select** button turns orange.

- c. Touch the table cell where you would like to enter data.

*Result:* A yellow box appears around the cell.

- d. Touch the **Data Entry** button.



*Result:* The on-screen keyboard opens.

- e. Type a number or text and touch **OK**.

*Result:* The data that you entered appears in the selected table cell.

5. Repeat step 3 (and optionally step 4) as many times as necessary to record all of the data that you want in the data set.

6. When the entire set has been recorded, touch the **Stop** button.



*Result:* The data set closes.

Note: If you accidentally stop the data collection early (by touching the **Stop** button instead of the **Keep** button), you will need to start over again from the beginning. If desired, delete the incomplete data set before starting again. <sup>(8.1)</sup>

---

### 6.3.1 Starting a manually sampled data set

- ◆ Touch the **Start** button.



Alternatively, you can press one of the record buttons instead of touching the on-screen **Start** button.

*Result:* The SPARK creates a new data set. Live data appear in the data displays. The record buttons start flashing to indicate that the SPARK is ready to be triggered.

---

### 6.3.2 Recording a manually sampled data point

1. When you are ready to trigger the recording of a data point, touch the **Keep** button.



Alternatively, you can press one of the record buttons instead of touching the on-screen **Keep** button.



*Result:* The SPARK records a single value from each measurement.

2. Optionally, if you have set up the SPARK to accept manually entered data along with the sensor data <sup>(5.2.1)</sup>, complete these substeps to enter a number or text:
  - a. If the table tool palette is not already open, touch the **Table Tools** button.



- b. If it is not already highlighted in the tool palette, touch the **Select** button.



*Result:* The **Select** button turns orange.

- c. Touch the table cell where you would like to enter data.

*Result:* A yellow box appears around the cell.

- d. Touch the **Data Entry** button.



*Result:* The on-screen keyboard opens.

- e. Type a number or text and touch **OK**.

*Result:* The data that you entered appears in the selected table cell.

Note: If you accidentally stop the data collection early (by touching the **Stop** button instead of the **Keep** button), you will need to start over again from the beginning. If desired, delete the incomplete data set before starting again. <sup>(8.1)</sup>

### 6.3.3 Stopping a manually sampled data set

- ◆ When the entire set has been recorded, touch the **Stop** button.



*Result:* The data set closes.

## Section 7: Data display

### 7.1 Graph

#### 7.1.1 Displaying data in a graph

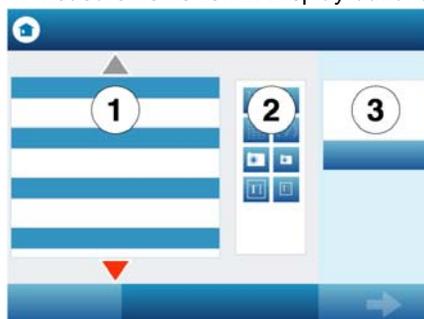
- Do one of the following to open the page-build screen.
  - If a SPARKlab is open, touch the **New Page** button.



- If the Home screen is open, touch **BUILD**.

*Result:* The page-build screen opens.

**Page-build screen:** 1. Measurements list. 2. Display buttons. 3. Preview section.



2. Touch a measurement (or two measurements) to select them.

Note: Selected measurements are highlighted. If you select one measurement it will be plotted on the y-axis with time on the x-axis. If you select two measurements, the first selected will be plotted on the y-axis and the second will be plotted on the x-axis.

3. Touch the **Graph** button.



4. Touch **OK**.

*Task result:* A new page is created with a graph.

### 7.1.2 Adjusting the scale of a graph

To adjust the x- and y-scales, do one or more of the following:

- ◆ Scale the graph to fit all data:
  - a. If the graph tool palette is not already open, touch the **Graph Tools** button.



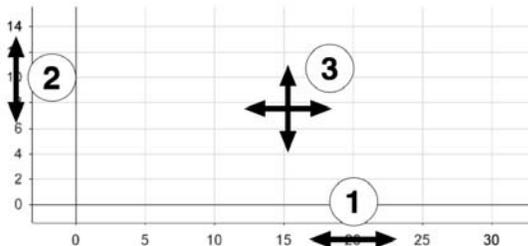
- b. Touch the **Scale-to-fit** button.



*Result:* The graph adjusts to fit all data.

- ◆ Manually scale the graph:
  - ◆ Touch one of the numbers labeling the x-scale of the graph and drag it left or right. The graph expands or contracts horizontally.
  - ◆ Touch one of the numbers labeling the y-scale of the graph and drag it up or down. The graph expands or contracts vertically.
  - ◆ Touch the middle of the graph and drag it in any direction. The graph moves.

1. Expanding and contracting horizontally. 2. Expanding and contracting vertically. 3. Moving.



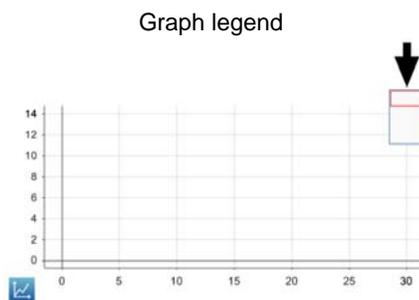
### 7.1.3 Displaying multiple data runs on a graph

Complete these steps to display up to four data runs in a graph.

1. Touch the graph legend in the upper right corner of the graph.

*Result:* The legend enlarges to show available data runs.

2. Select or clear the check box next to each data run that you want to show or hide.
3. Optionally, touch outside the legend to reduce the size of the legend.



### 7.1.4 Selecting data points in a graph

After you have selected data points in a graph, they will be highlighted. Scale-to-fit, statistics, graph tools, and curve fits will be applied only to the selected data points. Complete these steps to select part of a data run:

1. If there is more than one data run on the graph, first select the run from which you will select data points:
  - a. Touch the graph legend.
 

*Result:* The legend enlarges.
  - b. In the legend, touch the symbol of the run that you want to select.
 

*Result:* The red outline moves to the selected run.
2. Touch the **Graph Tools** button to open the tool palette.



3. Touch the **Select** button.

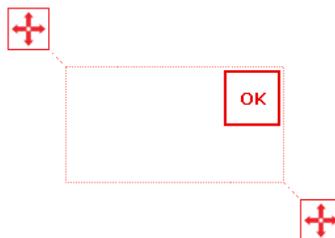


*Result:* The button turns orange.

4. Touch a point on the graph; then, within one second, touch another point on the graph.

The two locations that you touch define the corners of a selection box.

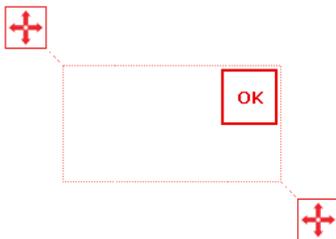
*Result:* A selection box appears. Data points inside the box are highlighted.



5. Optionally, adjust the size and position of the selection box by dragging the handles at the corners of the box.



6. When the desired data points are highlighted, touch **OK**.



*Result:* The selection box disappears, but the points remain selected.

To clear the selection, touch the **Select** button again.

### 7.1.5 Adding a note to a graph

1. If there is more than one data run on the graph, first select the run to which the note will be attached:
  - a. Touch the graph legend.

*Result:* The legend enlarges.

- b. In the legend, touch the symbol of the run that you want to select.

*Result:* The red outline moves to the selected run.

2. Touch the **Graph Tools** button to open the tool palette.



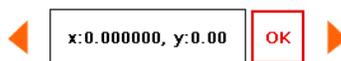
3. Touch the **Select** button.



*Result:* The button turns orange.

4. Touch a point on the graph.

5. Touch **OK**.



6. Touch the **Annotation** button.



*Result:* The on-screen keyboard appears.

7. Enter a note and touch **OK**.

*Result:* An annotation appears on the graph.

8. Touch the **Select** button.

*Result:* The button turns blue.

### 7.1.6 Removing a note from a graph

1. If necessary, touch the **Graph Tools** button to open the tool palette.



2. Touch the annotation that you want to edit or delete.  
*Result:* The annotation is highlighted.
3. Touch the **Annotation** button.

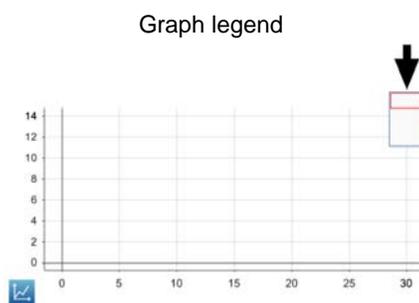


*Result:* The on-screen keyboard appears.

4. Backspace over the annotation and touch **OK**.

### 7.1.7 Showing and hiding data runs in a graph

1. Touch the graph legend in the upper right corner of the graph.  
*Result:* The legend enlarges to show available data runs.
2. Select or clear the check box next to each data run that you want to show or hide.
3. Optionally, touch outside the legend to reduce the size of the legend.



### 7.1.8 Showing and hiding connecting lines between data points

Note: This feature is not currently supported. Please visit [www.pasco.com](http://www.pasco.com) for the latest updates to SPARK firmware and documentation.

### 7.1.9 Changing the variable on the x- or y-axis

1. Touch the **Graph Tools** button to open the tool palette.



2. Touch the **Properties** button to open the properties screen.



3. For each axis, touch the **Measurement** box and select a measurement or other variable.

### 7.1.10 Displaying multiple variables on the y-axis

Complete these steps to create a page containing two graphs with a different variable on the y-axis of each graph and time on both x-axes.

1. Do one of the following to open the page-build screen.
  - ◆ If a SPARKlab is open, touch the **New Page** button.



- ◆ If the Home screen is open, touch **BUILD**.

*Result:* The page-build screen opens.

**Page-build screen:** 1. Measurements list. 2. Display buttons. 3. Preview section.



2. In the measurements list, touch one of the measurements or variables that you would like to appear on the y-axis.

The selected measurement is highlighted.

3. Touch the **Graph** button.



*Result:* The first graph appears in the preview section of the page-build screen.

4. In the measurements list, touch the second measurement or variable that you would like to appear on the y-axis.

The selected measurement is highlighted.

5. Touch the **Graph** button.



*Result:* The second graph appears in the preview section of the page-build screen.

6. Touch **OK**.

*Task result:* A new page is created with two graphs displaying both variables on the y-axes.

### 7.1.11 Displaying multiple graphs

- ◆ To display multiple graphs, each on its own page, repeat the steps in Tech Tip 7.1.1 for each graph.
- ◆ To display two graphs on one page, complete the steps in Tech Tip 7.1.10.

### 7.1.12 Drawing a prediction

1. Touch the **Graph Tools** button to open the tool palette.



2. Touch the **Prediction** button.



3. Do one of the following:
  - ◆ Trace a continuous curve on the graph.
  - ◆ Touch several locations on the graph to draw a series of connected points.
4. Touch **OK**.

## 7.2 Table

### 7.2.1 Displaying data in a table

1. Do one of the following to open the page-build screen.
  - ◆ If a SPARKlab is open, touch the **New Page** button.



- ◆ If the Home screen is open, touch **BUILD**.

*Result:* The page-build screen opens.

**Page-build screen:** 1. Measurements list. 2. Display buttons. 3. Preview section.



2. Touch a measurement (or up to six measurements) to select them. Selected measurements are highlighted.
3. Touch the **Table** button.



4. Touch **OK**.

*Result:* A new page is created containing a table with a column for time and a column for each measurement that you selected. (Time is not included if you selected six measurements.)

5. Optionally, remove unwanted columns:
  - a. Touch the **Table Tools** button to open the tool palette.



- b. Touch the **Select** button.



*Result:* The button turns orange.

- c. Touch the column that you want to remove.
  - d. Touch the **Remove Column** button.



6. Optionally, add additional columns<sup>(7.2.2)</sup>.

---

### 7.2.2 Adding a measurement to a table

1. Touch the **Table Tools** button to open the tool palette.



2. Touch the **Add Column** button.



*Result:* A new column is added to the table.

3. Select a measurement for the new column:
  - a. Touch the **Properties** button to open the properties screen.



- b. Touch the **Measurement** box and select the measurement or other variable that you want to see in the new column.
  - c. Touch **OK**.

### 7.2.3 Manually entering data into a table

1. Do one of the following to open the page-build screen.
  - ◆ If a SPARKlab is open, touch the **New Page** button.



- ◆ If the Home screen is open, touch **BUILD**.

*Result:* The page-build screen opens.

**Page-build screen:** 1. Measurements list. 2. Display buttons. 3. Preview section.



2. Create an empty user-entered data set:
  - a. In the measurements list under **User-entered Number Data** or under **User-entered Text Data**, touch **Create Data Set**.  
*Result:* The Define the Data Set screen opens.
  - b. Touch the **Measurement Name** box, type a name that describes the data or text that you plan to enter manually, and touch **OK**.
  - c. Touch **OK** to return to the page-build screen.  
*Result:* The user-entered data set that you created now appears in the measurements list on the page-build screen.
3. In the measurements list, touch the data set that you just created to select it.  
*Result:* The selected data set is highlighted.
4. Touch the **Table** button.



5. Touch **OK**.  
*Result:* A table prepared to accept the manually entered data appears.
6. Enter data:
  - a. If the table tool palette is not already open, touch the **Table Tools** button.



- b. If it is not already highlighted in the tool palette, touch the **Select** button.



*Result:* The **Select** button turns orange.

- c. Touch the table cell where you would like to enter data.

*Result:* A yellow box appears around the cell.

- d. Touch the **Data Entry** button.



*Result:* The on-screen keyboard opens.

- e. Type a number or text and touch **OK**.

7. Repeat step 6 to enter data in other cells of the table.
- 

#### 7.2.4 Showing and hiding data runs in a table

1. Touch the run number at the top of the column.

*Result:* A list of available runs appears.

2. Touch the run that you want to see.
- 

#### 7.2.5 Changing the variable displayed in a column

1. Touch the **Table Tools** button to open the tool palette.



2. Touch the **Properties** button to open the properties screen.



3. Touch the **Column** box and select the column that you want to change.

Columns are number 1, 2, 3, and so on from left to right.

4. Touch the **Measurement** box and select the measurement or other variable that you want to see in the column.
5. Touch **OK**.

---

## 7.3 Digits display

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### 7.3.1 Displaying data in a digits display

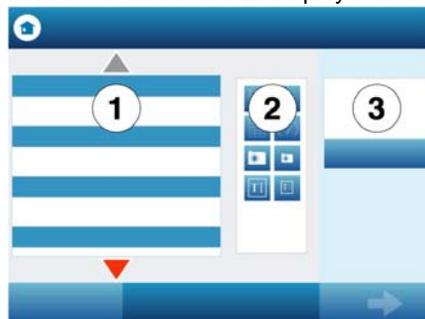
1. Do one of the following to open the page-build screen.
  - ◆ If a SPARKlab is open, touch the **New Page** button.



- ◆ If the Home screen is open, touch **BUILD**.

*Result:* The page-build screen opens.

**Page-build screen:** 1. Measurements list. 2. Display buttons. 3. Preview section.



2. Touch one measurement to select it.  
The selected measurement is highlighted.
3. Touch the **Digits Display** button.



4. Touch **OK**.

*Task result:* A new page is created with a digits display.

### 7.3.2 Adding a measurement to a digits display

Complete these steps to create a page displaying more than one measurement in digits displays.

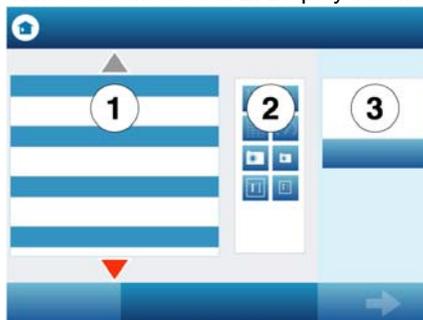
1. Do one of the following to open the page-build screen.
  - ◆ If a SPARKlab is open, touch the **New Page** button.



- ◆ If the Home screen is open, touch **BUILD**.

*Result:* The page-build screen opens.

**Page-build screen:** 1. Measurements list. 2. Display buttons. 3. Preview section.



2. Touch one measurement to select it.  
The selected measurement is highlighted.
3. Touch the **Digits Display** button.



*Result:* The digits display appears in the preview section of the page-build screen.

4. Repeat steps 2 and 3 to add other measurements to the same page.
5. Touch **OK**.

*Task result:* A new page is created with multiple digits displays.

## Section 8: Data run operations

---

### 8.1 Deleting a data run

1. Touch the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

2. Touch **MANAGE RUNS**.

*Result:* The Manage Runs screen opens.

3. Do one of the following:
  - ◆ Touch **Delete Last Run**.
  - ◆ Touch **Delete All Runs**.
  - ◆ Touch **Delete Run** and select the run that you want to delete.
4. Touch **DONE** to close the Manage Runs screen.
5. Touch **OK** to close the Experiment Tools screen.

## 8.2 Naming a data run

Complete these steps to name a data run on a graph by attaching a note to it.

1. If there is more than one data run on the graph, first select the run that the note will be attached to:
  - a. Touch the graph legend.
 

*Result:* The legend enlarges.
  - b. In the legend, touch the symbol of the run that you want to select.
 

*Result:* The red outline moves to the selected run.
2. Touch the **Graph Tools** button to open the tool palette.

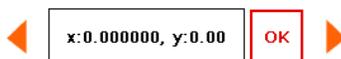


3. Touch the **Select** button.



*Result:* The button turns orange.

4. Touch a point on the graph.
5. Touch **OK**.



6. Touch the **Annotation** button.



*Result:* The on-screen keyboard appears.

7. Enter a note and touch **OK**.
 

*Result:* An annotation appears on the graph.
8. Touch the **Select** button.
 

*Result:* The button turns blue.

## Section 9: Analyzing data

### 9.1 Finding the coordinates of a point in a graph

Complete these steps to select a point on a graph and display its coordinates:

1. If more than one data run is displayed, first select a run:
  - a. Touch the graph legend.  
*Result:* The legend enlarges.
  - b. In the legend, touch the symbol of the run that you want to select.  
*Result:* The red outline moves to the selected run.
2. Touch the **Graph Tools** button to open the tool palette.

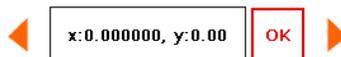


3. Touch the **Select** button.



*Result:* The button turns orange.

4. Touch a point on the graph.  
*Result:* The x- and y-values of the selected point are displayed.
5. Optionally, use the point selector to change which point is selected.



6. Touch **OK**.
7. Optionally, touch the **Coordinates** button.



*Result:* An annotation displaying the x- and y-values appears on the graph.

To clear the annotation, touch the **Coordinates** button again. To clear the selection, touch the **Select** button again.

### 9.2 Measuring the difference between two points in a graph

Complete these steps to select a range of points and display the change-in-x and change-in-y between the first and last points in the selected range:

1. If more than one data run is displayed, first select a run:
  - a. Touch the graph legend.  
*Result:* The legend enlarges.
  - b. In the legend, touch the symbol of the run that you want to select.  
*Result:* The red outline moves to the selected run.
2. Touch the **Graph Tools** button to open the tool palette.



3. Touch the **Select** button.

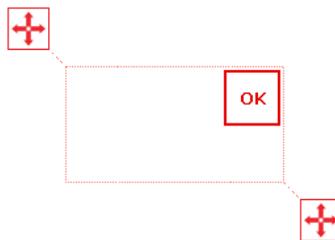


*Result:* The button turns orange.

4. Touch a point on the graph; then, within one second, touch another point on the graph.

The two locations that you touch define the corners of a selection box.

*Result:* A selection box appears. Data points inside the box are highlighted.



5. Optionally, adjust the size and position of the selection box by dragging the handles at the corners of the box.



6. When the desired data points are highlighted, touch **OK**.

*Result:* The selection box disappears, but the points remain selected.

7. Touch the **Coordinates** button.



*Task result:* An annotation with the following information appears on the graph:

- ◆ The x- and y-values of the first point in the selected range ( $x_1$  and  $y_1$ ),
- ◆ The x- and y-values of the last point in the selected range ( $x_2$  and  $y_2$ ), and
- ◆ The x- and y-differences between those two points ( $dx$  and  $dy$ ).

Note: To clear the annotation, touch the **Coordinates** button again. To clear the selection, touch the **Select** button again.

### 9.3

#### Finding the slope at a point on a data plot

Complete these steps to display the slope at a selected point.

1. If more than one data run is displayed, first select a run:

- a. Touch the graph legend.

*Result:* The legend enlarges.

- b. In the legend, touch the symbol of the run that you want to select.

*Result:* The red outline moves to the selected run.

2. Touch the **Graph Tools** button to open the tool palette.

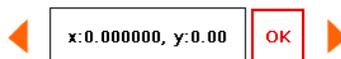


3. Touch the **Slope Tool** button.



*Result:* A slope tool appears on the graph displaying the slope at one point. If no data points have been selected, the slope tool appears in the middle of the data run. If data points have been selected <sup>◆(7.1.4)</sup>, the slope tool appears in the middle of the selected range. If a single data point has been selected, the slope tool appears on that point.

4. Use the point selector to move the slope tool to nearby points.



To hide the slope tool, touch the **Slope Tool** button again.

## 9.4 Viewing statistics of data

Complete these steps to see the minimum, maximum, mean, standard deviation, count, and area-under-the-curve of a data run displayed in a graph:

1. If more than one data run is displayed, first select a run:
  - a. Touch the graph legend.  
*Result:* The legend enlarges.
  - b. In the legend, touch the symbol of the run that you want to select.  
*Result:* The red outline moves to the selected run.
2. Touch the **Graph Tools** button to open the tool palette.



3. Touch the **Statistics** button to open the Statistics screen.



4. Touch one or more of the statistics.  
Selected statistics are highlighted.
5. Touch **OK**.  
*Result:* Statistics appear on the graph.
6. Optionally, select part of the data set for statistics to be applied to. <sup>◆(7.1.4)</sup>

Note: To remove the statistics, touch the **Statistics** button again.

## 9.5 Applying a curve fit

Complete these steps to apply a linear, quadratic, power, inverse, inverse square, or sine fit to a data run:

1. If more than one data run is displayed, first select a run:
  - a. Touch the graph legend.  
*Result:* The legend enlarges.
  - b. In the legend, touch the symbol of the run that you want to select.  
*Result:* The red outline moves to the selected run.

2. Touch the **Graph Tools** button to open the tool palette.



3. Touch the **Curve Fit** button to open the Curve Fit screen.



4. Touch one curve fit to select it.
5. Touch **OK**.

*Result:* The curve and parameters of the curve appear on the graph.

6. Optionally, select part of the data set for the curve fit to be applied to. <sup>◆(7.1.4)</sup>

## 9.6

### Finding the slope and intercept of a best-fit line

Complete these steps to apply a linear fit to a data run:

1. If more than one data run is displayed, first select a run:
  - a. Touch the graph legend.

*Result:* The legend enlarges.

- b. In the legend, touch the symbol of the run that you want to select.

*Result:* The red outline moves to the selected run.

2. Touch the **Graph Tools** button to open the tool palette.



3. Touch the **Curve Fit** button to open the Curve Fit screen.



4. Touch **Linear Fit** to select it.
5. Touch **OK**.

*Result:* The best-fit with its slope ( $m$ ) and y-intercept ( $b$ ) appear on the graph.

6. Optionally, select part of the data set for the linear fit to be applied to. <sup>◆(7.1.4)</sup>

## 9.7

### Finding the area under a curve

Complete these steps to see the area-under-the-curve of a data run:

1. If more than one data run is displayed, first select a run:
  - a. Touch the graph legend.

*Result:* The legend enlarges.

- b. In the legend, touch the symbol of the run that you want to select.

*Result:* The red outline moves to the selected run.

2. Touch the **Graph Tools** button to open the tool palette.



3. Touch the **Statistics** button to open the Statistics screen.



4. Touch **Area** to select it.
5. Touch **OK**.

*Result:* The area under the curve is shaded and the value appears on the graph.

6. Optionally, select part of the data set for the area operation to be applied to. <sup>(7.1.4)</sup>

Note: To remove the area, touch the **Statistics** button again.

## Section 10: Data operations

---

### 10.1 Editing data manually

1. Touch the **Table Tools** button to open the tool palette.



2. Touch the **Select** button.



*Result:* The button turns orange.

3. Touch the table cell where you would like to edit data.

*Result:* A yellow box appears around the cell.

4. Touch the **Data Entry** button.



*Result:* The on-screen keyboard opens.

5. Type a number or text and touch **OK**.

Repeat this task to edit data in other cells.

---

### 10.2 Deleting data points

Complete these steps to delete manually entered data from a table cell.

1. If the table tool palette is not already open, touch the **Table Tools** button.



2. If it is not already highlighted in the tool palette, touch the Select button.



*Result:* The **Select** button turns orange.

3. Touch the table cell where you would like to delete data.  
*Result:* A yellow box appears around the cell.
4. Touch the **Data Entry** button.



*Result:* The on-screen keyboard opens.

5. Backspace over the data and touch **OK**.

---

## 10.3 Creating calculated data

1. Touch the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

2. Touch **CALCULATED DATA**.

*Result:* The calculator screen opens.

3. Use the on-screen keyboard to type an expression.

If you have been given an expression in your lab instructions, enter it into the calculator exactly as it appears in the instructions; however, if you change the text to the left of the equals sign, do not use spaces between words.

- ◆ To insert a measurement into the expression, touch **Measurements**.
- ◆ To cycle through the various functions available for use in the expression, touch **Functions**.
- ◆ To enter words or letters into the expression, touch the **Letters** button.



4. Touch **Return** to complete the calculation.
5. Touch **DONE** to close the calculator screen.
6. Touch **OK** to close the Experiment Tools screen.

*Task result:* The calculation is now available for viewing in a graph, table, or other display.

---

## Section 11: Saving and sharing your experiment

### 11.1 Saving your experiment

Complete these steps to save your work on the SPARK or on a USB flash drive (or other USB storage device).

1. If you plan to save the lab on a USB flash drive, connect the flash drive to the SPARK.
2. Touch the **Sharing** button to open the Sharing screen.



3. Touch **SAVE FILE AS**.  
*Result:* The save file screen opens.
4. Touch the **Name** box and enter a name for your experiment.
5. If you plan to save the lab on a USB flash drive, touch **USB**.
6. Optionally, touch a folder to select it.  
The experiment will be saved in the selected folder.
7. Optionally, touch an existing file to select it.  
The selected file will be overwritten.
8. Touch **SAVE**.  
*Result:* The experiment is saved, and the SPARK returns to the Sharing screen.
9. Touch **DONE** to close the Sharing screen.

Note: After you have saved a file once using the procedure above, you can quickly save the file again by touching **SAVE FILE** instead of **SAVE FILE AS** in the Sharing screen.

---

## 11.2 Printing

To print one or more pages from a SPARKlab, complete these steps to capture pages in the journal and print the journal. You will need a compatible USB printer. Most USB inkjet printers manufactured by HP are compatible with SPARK.

1. On each SPARKlab page that you wish to print, touch the **Snapshot** button.



*Result:* Each time you touch the **Snapshot** button, the Snapshot Quick View appears briefly and an image of the page is added to the journal.

2. Connect a printer to the USB port of the SPARK.
3. Touch the **Sharing** button to open the Sharing screen.



4. Touch the **JOURNAL** tab.
5. Touch **PRINT JOURNAL** to open the journal print page.
6. Touch **OK**.

*Result:* The journal is printed, and the SPARK returns to the Sharing screen.

7. Touch **DONE** to close the Sharing screen.

---

## 11.3 Exporting data for use in mapping software

To export data, you will need a USB flash drive (or other USB storage device). All data in the experiment that you have open on the SPARK will be saved on the flash drive in a file (or files) that can be opened by the mapping software on a computer.

Complete these steps to export data:

1. Connect a flash drive to the USB port of the SPARK.

2. Touch the **Sharing** button to open the Sharing screen.



3. Touch **EXPORT DATA** to open the export data page.
4. Touch the **Name** box and enter a name for the file (or files) that will be saved on the flash drive.
5. Touch **EXPORT**.

*Result:* The SPARK returns to the Sharing screen and a data file is saved on the USB flash drive. If there were multiple data runs on the SPARK, a separate, numbered file is saved for each data run.

6. Touch **DONE** to close the Sharing screen.
7. Disconnect the flash drive.



# Appendix B: SPARKvue Tech Tips

## Section 1: Starting an experiment

---

### 1.1 Opening a file

1. In the Home screen, click **OPEN**.  
*Result:* The Open window appears.
2. Navigate to the folder where the file is located.
3. Select the file that you would like to open.
4. Click **Open**.

*Task result:* The file opens.

---

### 1.2 Starting a new experiment

- ◆ Click the **Home** button.



*Task result:* The Home screen displays live readings from any connected sensor.

---

## Section 2: Setting up measurements and data collection

---

### 2.1 Adding a sensor to your SPARKvue experiment

1. Connect an interface such as a SPARKlink or a SPARK Science Learning System to your computer.
2. Connect a sensor to the interface.

*Task result:* SPARKvue detects the sensor.

---

### 2.2 Adding multiple sensors to your SPARKvue experiment

1. Connect an interface such as a SPARKlink or a SPARK Science Learning System to your computer.
2. Connect sensors to any available ports on the interface.

*Task result:* SPARKvue detects all connected sensors.

---

### 2.3 Programming SPARKvue to calculate lung volume

1. Click the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

2. Click **CALCULATED DATA**.

*Result:* The calculator screen opens.

---

3. Complete the substeps below to enter this expression:

LungVolume = V0-[Total Flow(L)]

- a. Type: LungVolume = V0-
- b. Click **Measurements**.
- c. Click **Total Flow**.

*Result:* This expression now appears in the entry box of the calculator screen: LungVolume = V0-[Total Flow(L)]

4. Click **Return**.

*Result:* A second expression appears in the entry box: V0 =

5. Type the test subject's functional residual capacity (FRC) in liters.

If you do not know the test subject's FRC, type: 2.5

The second line in the entry box should now be similar to: V0 = 2.5

6. Click **Return**.

*Result:* The calculation is complete.



7. Click **DONE** to close the calculator screen.
8. Click **OK** to close the Experiment Tools screen.

*Task result:* The Lung Volume calculation is now available for viewing in a graph, table, or other display.

## Section 3: Calibrating sensors

### 3.1 Calibrating a carbon dioxide gas sensor

Once the carbon dioxide gas sensor has been calibrated using this method, it retains its calibration, even after it has been unplugged.

1. Take the sensor and an empty sample bottle outdoors.  
If necessary, unplug the sensor from the interface.
2. Fill the sample bottle with fresh outdoor air.
3. Insert the probe of the sensor into the bottle and push the rubber stopper of the sensor into the neck of the bottle.

4. If you have disconnected the sensor, return to the interface and connect the sensor to it.
5. Press the **Calibrate** button on the sensor and hold it for three seconds.  
*Result:* The green light illuminates indicating that calibration is in progress.
6. Wait approximately one minute.  
When the green light flashes slowly, calibration is finished. If the light flashes rapidly (five times per second), an error has occurred and the sensor has not been calibrated.

---

### 3.2 Calibrating a colorimeter

1. Place a cuvette filled with distilled water in the colorimeter and close the lid.
2. Press the **Calibration** button on the sensor.  
*Result:* The light in the button illuminates indicating that calibration is in progress.
3. Wait for the light to turn off.

---

### 3.3 Calibrating a dissolved oxygen sensor

1. Obtain barometric pressure and temperature at your location.
2. Refer to the solubility table included with the sensor and find the standard dissolved oxygen value for the temperature and barometric pressure at your location.
3. Click the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

4. Click **CALIBRATE SENSOR**.  
*Result:* The Calibrate Sensor: Select Measurement screen opens.
5. Click the **Sensor** box and select the sensor that the dissolved oxygen probe is connected to.
6. Click the **Measurement** box and select **Dissolved Oxygen (mg/L)**.
7. Click the **Calibration Type** box and select **1 point (Adjust Slope Only)**.
8. Click **NEXT**.  
*Result:* The Calibrate Sensor screen opens
9. Under **Calibration Point 2** click the **Standard Value** box and enter the value that you determined from the solubility table.
10. Place about 5 mL (to a height of about 1 cm) of deionized water into a clean soaker bottle. Slip the cap and O-ring of the soaker bottle over the end of the probe.
11. Insert the probe into the soaker bottle and screw on the lid. Adjust the height of the probe to about 2 cm above the top of the water.
12. Shake the soaker bottle vigorously for about 10 seconds. Shake off any large water drops from the membrane at the end of the probe.

13. Under **Calibration Point 2** click **Read From Sensor**.  
*Result:* The value measured by the sensor is transferred to the **Sensor Value** box.
14. Click **OK** to exit the Calibrate Sensor screen.
15. Click **OK** to exit the Experiment Tools screen.

---

### 3.4 Calibrating a drop counter

The drop counter is calibrated *after* data is taken, using SPARKvue’s calculator to correlate the number of drops counted to the volume of liquid dispensed from the burette. The following steps illustrate the calibration procedure. Note that in performing the experiment, these steps will be interspersed with other experiment procedure steps.

Note: Using this method, you will ignore the “Volume” measurement output directly from the sensor. Instead, volume is calculated by SPARKvue. This method takes into account the fact that drop size may be different each time the burette is used. By measuring the total volume dispensed during the actual experiment, a more accurate correlation between volume and drop count is obtained.

1. Set up the drop counter on a stand with a liquid-filled burette, a beaker, stir-plate, and other equipment.  
 See your lab instructions for details.
2. Write down the initial volume of liquid in the burette.
3. Click the **Start** button.



4. Slowly turn the stopcock to start delivering liquid at about 2 drops per second.
5. After the necessary amount of liquid has been dispensed from the burette, close the stopcock.
6. Click the **Stop** button.



7. Read the final volume of liquid in the burette and determine the net volume that was dispensed.
8. Read the final drop count (the total number of drops dispensed) in a graph or table display.
9. Click the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

10. Click **CALCULATED DATA**.

*Result:* The calculator screen opens.

11. Complete the substeps below to enter an expression similar to this:  

$$\text{volume} = [\text{Drop Count (drops)}] * 100/3050$$

- a. Type: volume =
- b. Click **Measurements**.
- c. Click **Drop Count**.
- d. Type something similar to: \* 100/3050

Note: In this example, “100” is the total volume dispensed (in mL) and “3050” is the final drop count. When you type the expression, use your own values for these two quantities.

*Result:* An expression similar to this appears in the entry box of the calculator screen: volume = [Drop Count (drops)] \* 100/3050

12. Click **Return**.

*Result:* The calculation is complete.

13. Click **DONE** to close the calculator screen.
14. Click **OK** to close the Experiment Tools screen.

*Task result:* The calibrated Volume calculation is now available for viewing in a graph, table, or other display.

### 3.5 Calibrating an oxygen gas sensor

Once the oxygen gas sensor has been calibrated using this method, it retains its calibration, even after it has been unplugged.

1. Take the sensor and an empty sample bottle outdoors.  
If necessary, unplug the sensor from the interface.
2. Fill the sample bottle with fresh outdoor air.
3. Insert the probe of the sensor into the bottle and push the rubber stopper of the sensor into the neck of the bottle.
4. If you have disconnected the sensor, return to the interface and connect the sensor to it.
5. Press the **CAL (20.9%)** button on the sensor and hold it for three seconds.

*Task result:* The green light flashes on and off for 4 seconds, indicating that calibration is in progress. When the light stops flashing, calibration is finished. If the light flashes rapidly (5 times per second), an error has occurred and the sensor has not been calibrated.

### 3.6 Calibrating a pH sensor

You will need buffer solutions of pH 4 and pH 10 and deionized water.

1. Click the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

2. Click **CALIBRATE SENSOR**.

*Result:* The Calibrate Sensor: Select Measurement screen opens.

3. Click the **Sensor** box and select the sensor that the pH probe is connected to.
4. Click the **Measurement** box and select **pH**.

5. Click the **Calibration Type** box and select **2 point (Adjust Slope and Offset)**.
6. Click **NEXT**.  
*Result:* The Calibrate Sensor screen opens
7. Place the pH probe into the pH 4 buffer solution and wait for about 1 minute.
8. Under **Calibration Point 1** click the **Standard Value** box and enter 4 (the known pH of the buffer solution).
9. Under **Calibration Point 1**, click **Read From Sensor**.  
*Result:* The value measured by the sensor is transferred to the **Sensor Value** box.
10. Rinse the probe with deionized water.
11. Place the pH probe into the pH 10 buffer solution and wait for about 1 minute.
12. Under **Calibration Point 2** click the **Standard Value** box and enter 10 (the known pH of the buffer solution).
13. Under **Calibration Point 2**, click **Read From Sensor**.  
*Result:* The value measured by the sensor is transferred to the **Sensor Value** box.
14. Click **OK** to close the Calibrate Sensor screen.
15. Click **OK** to close the Experiment Tools screen.

---

### 3.7 Calibrating a turbidity sensor

1. Place a cuvette filled with distilled water in the turbidity sensor and close the lid.
2. Press the **Calibration** button on the sensor.  
*Result:* The green light in the button illuminates.
3. When the light starts blinking, replace the cuvette with the standard 100 NTU cuvette (included with the sensor) and close the lid.
4. Press the button again.  
*Result:* The light in the button illuminates.

*Task result:* When the light turns off, calibration is complete.

---

### 3.8 Calibrating an ethanol sensor

You will need a solution of 1% ethanol in water. The solution should be at the same temperature as the solutions to be measured.

Once the sensor has been calibrated using this method, it retains its calibration, even after it has been unplugged.

1. Connect the sensor to the interface and wait about 10 minutes for the probe to warm up.
2. Place the probe about 1 cm above the 1% ethanol solution.

3. Observe the ethanol concentration reading in SPARKvue and wait until the reading stabilizes.  
(You can observe the reading in the Home screen, or in a digits display while recording data.)
4. Press and hold the **1% CAL** button on the sensor for 4 seconds.

*Task result:* Immediately after a successful calibration, the sensor's output reads 1%, and the button is illuminated.

## Section 4: Sensor operations

### 4.1 Using a colorimeter with a specific color of light

The colorimeter makes measurements for four different colors; be sure to choose the measurements for the appropriate color.

The colorimeter contains four separate light sources of different colors: red (660 nm), orange (610 nm), green (565 nm), and blue (468 nm). Internally, the colorimeter uses all four light sources simultaneously and makes separate measurements for each color. However, in most experiments, you will be concerned only with one color and ignore the measurements associated with other colors. Your lab instructions may tell you which color to use, or you may need to determine the appropriate color on your own.

For each color there are two measurements: transmittance and absorbance.

As you proceed with your experiment, whenever you set up data displays or do certain other tasks, you will see a list of all eight measurements made by the colorimeter; be sure always to choose the transmittance or absorbance measurement for the appropriate color.

### 4.2 Setting up a conductivity sensor for a particular sensitivity

When using a conductivity sensor, it is important to select a sensitivity appropriate to the solutions that you plan to test. If the selected sensitivity is too high, the sensor may lack the range necessary to measure your most concentrated solutions. However, if the range is larger than necessary, the measurement precision may not be high enough to detect small conductivity changes or differences.

Complete these steps to select the appropriate sensitivity and range:

1. Determine or estimate the highest concentration that you will need to measure.

Use this chart and other resources.

<b>Solution</b>	<b>Conductivity (<math>\mu\text{S}/\text{cm}</math>)</b>
Ultrapure water	0.05 to 0.75
Drinking water	50 to 1500
Ocean water	~53000

- Select the range that best matches your measurement requirements.

The first column of this chart shows available ranges.

Measurement Range ( $\mu\text{S/cm}$ )	1x Probe Range Selection	10x Probe Range Selection
0 to 100		-
0 to 1000		
0 to 10000		
0 to 100000	-	

- Select an appropriate probe (either 1x or 10x) and connect it to the sensor.
- Use the buttons on the sensor to select an appropriate range.

Lights in the buttons indicate which range is selected.

Note: As you proceed with your experiment, whenever you set up data displays or do certain other tasks, you will see a list two different measurements made by the sensor: one for the 1x probe and one for the 10x probe; be sure always to choose the measurement that matches the connected probe.

### 4.3

Setting up a rotary motion sensor to measure linear position using the “rack” setting

The default Linear Position measurement assumes the large pulley attached to the rotary motion sensor. This measurement should be multiplied by 0.532 to give linear position data for a toothed rack inserted into the rotary motion sensor. Complete these steps to program the SPARK to make that calculation:

- Click the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

- Click **CALCULATED DATA**.

*Result:* The calculator screen opens.

- Complete the substeps below to enter this expression:

$\text{RackLinearPosition} = [\text{Linear Position(m)}] * 0.532$

- Type: RackLinearPosition =
- Click **Measurements**.
- Click **Linear Position**.
- Type: \*0.532

*Result:* This expression now appears in the entry box of the calculator screen:  $\text{RackLinearPosition} = [\text{Linear Position(m)}] * 0.532$

- Click **Return**.

*Result:* The calculation is complete.

5. Click **DONE** to close the calculator screen.
6. Click **OK** to close the Experiment Tools screen.

*Task result:* The RackLinearPosition calculation is now available for viewing in a graph, table, or other display.

## Section 5: Data measurement setup

---

### 5.1 Changing the sampling rate

1. Click the **Sampling Options** button.



*Result:* The Sampling Option screen opens.

2. Click the **Sample Rate Unit** box and select **Hz**, **seconds**, **minutes**, or **hours**.

If you select units of Hz, the value indicates the number of samples per second. If you select units of seconds, minutes, or hours, the value indicates the time between samples.

3. Click the **Sample Rate** box and select a value.
4. Click **OK** to close the Sampling Options screen.

---

### 5.2 Putting SPARKvue into manual sampling mode

1. Click the **Sampling Options** button.



*Result:* The Sampling Option screen opens.

2. Click **Manual**.
3. Click **OK** to close the Sampling Options screen.

*Task result:* SPARKvue is now ready to record manually sampled data. <sup>(6.3)</sup>

## 5.2.1 Putting SPARKvue into manual sampling mode with manually entered data

1. Do one of the following to open the page-build screen.
  - ◆ If a SPARKlab is open, click the **New Page** button.



- ◆ If the Home screen is open, click **BUILD**.

*Result:* The page-build screen opens.

**Page-build screen:** 1. Measurements list. 2. Display buttons. 3. Preview section.



2. Create an empty user-entered data set:
  - a. In the measurements list under **User-entered Number Data** or under **User-entered Text Data**, click **Create Data Set**.
 

*Note:* You may need to scroll the list to see these options.

*Result:* The Define the Data Set screen opens.
  - b. Click the **Measurement Name** box, type a name that describes the data or text that you plan to enter manually, and click **OK**.
  - c. Optionally, if you are creating a numerical data set, click the **Unit Name** box, type the units of the manually entered data, and click **OK**.
  - d. Click **OK** to return to the page-build screen.

*Result:* The user-entered data set that you created now appears in the measurements list on the page-build screen.

3. In the measurements list, click the data set that you just created to select it.

*Result:* The selected data set is highlighted.

4. In the measurements list, click a sensor measurement to select it.

Data from this sensor measurement will be recorded alongside your user-entered data.

*Result:* There are now two highlighted items in the measurements list: the user-entered data set and a sensor measurement.

5. In the measurements list, click a sensor measurement to select it.

Data from this sensor measurement will be recorded alongside your user-entered data.

*Result:* There are now two highlighted items in the measurements list: the user-entered data set and a sensor measurement.

- Click the **Table** button.



- Click **OK**.

*Result:* A table prepared to display the manually entered data and sensor data appears.

- Click the **Sampling Options** button.



*Result:* The Sampling Option screen opens.

- Click **Manual**.
- Click **OK** to close the Sampling Options screen.

*Task result:* SPARKvue is now ready to record manually sampled data with manually entered data. <sup>◆(6.3)</sup>

## 5.2.2

Putting SPARKvue into manual sampling mode without manually entered data

- Click the **Sampling Options** button.



*Result:* The Sampling Option screen opens.

- Click **Manual**.
- Click **OK** to close the Sampling Options screen.

*Task result:* SPARKvue is now ready to record manually sampled data. <sup>◆(6.3)</sup>

## 5.3

Changing the units of a measurement

Complete these steps to change the units of a measurement displayed in an existing graph, digits display, table, or meter:

- Click the **Tools** button of a graph, digits display, table, or meter to open the tools palette.



- Click the **Properties** button to open the properties screen.



- If you are changing the units of a measurement in a table, click the **Column** box and select the column containing the measurement.

Columns are numbered 1, 2, 3, and so on from left to right.

- Click the **Units** box and select a unit of measure.
- Click **OK**.

*Task result:* The display shows the measurement with the selected units.

**5.4** Changing the number of digits with which a variable is displayed

1. Click the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

2. Click **DATA PROPERTIES**.

*Result:* The Data Properties screen opens.

3. Click the **Measurement** box and select a measurement or other variable.
4. Click the **Number Format** arrow.

*Result:* The number format options appear.

5. Click the **Number Style** box and select **Fixed Precision**.
6. Use the **Digits** arrows to select the number of digits to be displayed after the decimal point.
7. Click **OK** to close the Data Properties screen.
8. Click **OK** to close the Experiment Tools screen.

---

**Section 6: Collecting and working with data**

---

**6.1** Monitoring live data

1. Display the measurement that you would like to monitor in a digits display. <sup>◆(7.3.1)</sup>
2. Click the **Sampling Options** button.



*Result:* The Sampling Option screen opens.

3. Click **Manual**.
4. Click **OK** to close the Sampling Options screen.
5. Click the **Start** button.



*Result:* The live readings are displayed.

6. To stop monitoring, click the **Stop** button.



---

**6.2** Recording a run of data

Complete these steps to record a data run in periodic sampling mode:

1. Click the **Start** button.

*Result:* SPARKvue creates a new data run and starts recording data points into it.

- To stop recording data, click the **Stop** button.



*Result:* SPARKvue stops recording data.

Repeat these steps to record another data run.

### 6.3 Recording a set of manually sampled data

If SPARKvue is in manual sampling mode, <sup>(5.2)</sup> complete these steps to record a data set:

- If you plan to type in user-entered data while you manually sample data from a sensor, turn to a page in your SPARKlab where you will be able to see the user-entered data and sensor data in a table.
- Click the **Start** button.



*Result:* SPARKvue creates a new data set. Live data appear in the data displays.

- When you are ready to trigger the recording of a data point, click the **Keep** button.



*Result:* SPARKvue records a single value from each measurement.

- Optionally, if you have set up SPARKvue to accept manually entered data along with the sensor data <sup>(5.2.1)</sup>, complete these substeps to enter a number or text:
  - If the table tool palette is not already open, click the **Table Tools** button.



- If it is not already highlighted in the tool palette, click the **Select** button.



*Result:* The **Select** button turns orange.

- Click the table cell where you would like to enter data.

*Result:* A yellow box appears around the cell.

- Click the **Data Entry** button.



*Result:* The on-screen keyboard opens.

- Type a number or text and click **OK**.

*Result:* The data that you entered appears in the selected table cell.

- Repeat step 3 (and optionally step 4) as many times as necessary to record all of the data that you want in the data set.

6. When the entire set has been recorded, click the **Stop** button.



*Result:* The data set closes.

Note: If you accidentally stop the data collection early (by clicking the **Stop** button instead of the **Keep** button), you will need to start over again from the beginning. If desired, delete the incomplete data set before starting again. <sup>(8.1)</sup>

---

### 6.3.1 Starting a manually sampled data set

- ◆ Click the **Start** button.



*Result:* SPARKvue creates a new data set. Live data appear in the data displays.

---

### 6.3.2 Recording a manually sampled data point

1. When you are ready to trigger the recording of a data point, click the **Keep** button.



*Result:* SPARKvue records a single value from each measurement.

2. Optionally, if you have set up SPARKvue to accept manually entered data along with the sensor data <sup>(5.2.1)</sup>, complete these substeps to enter a number or text:
  - a. If the table tool palette is not already open, click the **Table Tools** button.



- b. If it is not already highlighted in the tool palette, click the **Select** button.



*Result:* The **Select** button turns orange.

- c. Click the table cell where you would like to enter data.

*Result:* A yellow box appears around the cell.

- d. Click the **Data Entry** button.



*Result:* The on-screen keyboard opens.

- e. Type a number or text and click **OK**.

*Result:* The data that you entered appears in the selected table cell.

Note: If you accidentally stop the data collection early (by clicking the **Stop** button instead of the **Keep** button), you will need to start over again from the beginning. If desired, delete the incomplete data set before starting again. <sup>(8.1)</sup>

### 6.3.3 Stopping a manually sampled data set

- ◆ When the entire set has been recorded, click the **Stop** button.



*Result:* The data set closes.

## Section 7: Data display

### 7.1 Graph

#### 7.1.1 Displaying data in a graph

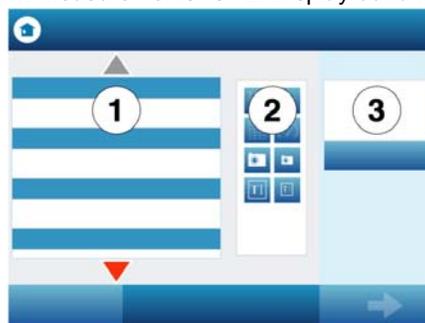
1. Do one of the following to open the page-build screen.
  - ◆ If a SPARKlab is open, click the **New Page** button.



- ◆ If the Home screen is open, click **BUILD**.

*Result:* The page-build screen opens.

**Page-build screen:** 1. Measurements list. 2. Display buttons. 3. Preview section.



2. Click a measurement (or two measurements) to select them.

*Note:* Selected measurements are highlighted. If you select one measurement it will be plotted on the y-axis with time on the x-axis. If you select two measurements, the first selected will be plotted on the y-axis and the second will be plotted on the x-axis.

3. Click the **Graph** button.



4. Click **OK**.

*Task result:* A new page is created with a graph.

### 7.1.2 Adjusting the scale of a graph

To adjust the x- and y-scales, do one or more of the following:

- ◆ Scale the graph to fit all data:
  - a. If the graph tool palette is not already open, click the **Graph Tools** button.



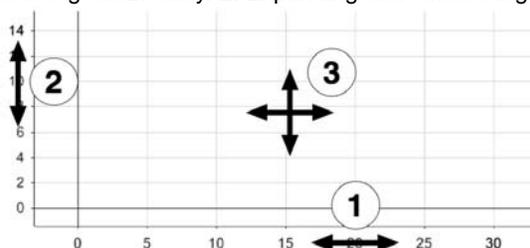
- b. Click the **Scale-to-fit** button.



*Result:* The graph adjusts to fit all data.

- ◆ Manually scale the graph:
  - ◆ Click one of the numbers labeling the x-scale of the graph and drag it left or right. The graph expands or contracts horizontally.
  - ◆ Click one of the numbers labeling the y-scale of the graph and drag it up or down. The graph expands or contracts vertically.
  - ◆ Click the middle of the graph and drag it in any direction. The graph moves.

1. Expanding and contracting horizontally. 2. Expanding and contracting vertically. 3. Moving.



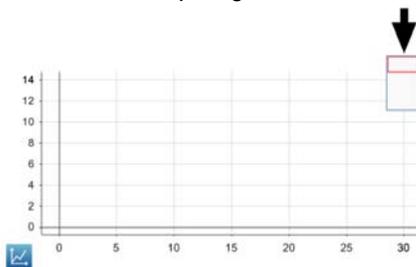
### 7.1.3 Displaying multiple data runs on a graph

Complete these steps to display up to four data runs in a graph.

1. Click the graph legend in the upper right corner of the graph.
 

*Result:* The legend enlarges to show available data runs.
2. Select or clear the check box next to each data run that you want to show or hide.
3. Optionally, click outside the legend to reduce the size of the legend.

Graph legend



### 7.1.4 Selecting data points in a graph

After you have selected data points in a graph, they will be highlighted. Scale-to-fit, statistics, graph tools, and curve fits will be applied only to the selected data points. Complete these steps to select part of a data run:

1. If there is more than one data run on the graph, first select the run from which you will select data points:

- a. Click the graph legend.

*Result:* The legend enlarges.

- b. In the legend, click the symbol of the run that you want to select.

*Result:* The red outline moves to the selected run.

2. Click the **Graph Tools** button to open the tool palette.



3. Click the **Select** button.

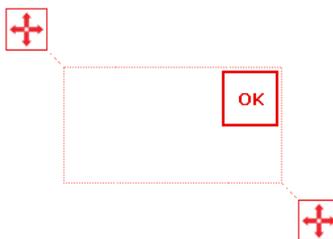


*Result:* The button turns orange.

4. Click a point on the graph; then, within one second, click another point on the graph.

The two locations that you click define the corners of a selection box.

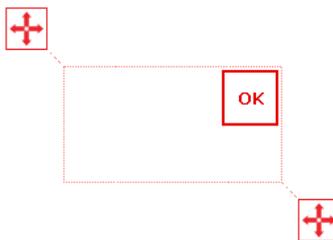
*Result:* A selection box appears. Data points inside the box are highlighted.



5. Optionally, adjust the size and position of the selection box by dragging the handles at the corners of the box.



6. When the desired data points are highlighted, click **OK**.



*Result:* The selection box disappears, but the points remain selected.

To clear the selection, click the **Select** button again.

### 7.1.5 Adding a note to a graph

1. If there is more than one data run on the graph, first select the run to which the note will be attached:
  - a. Click the graph legend.  
*Result:* The legend enlarges.
  - b. In the legend, click the symbol of the run that you want to select.  
*Result:* The red outline moves to the selected run.
2. Click the **Graph Tools** button to open the tool palette.

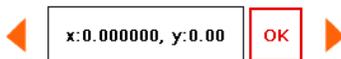


3. Click the **Select** button.



*Result:* The button turns orange.

4. Click a point on the graph.
5. Click **OK**.



6. Click the **Annotation** button.



*Result:* The on-screen keyboard appears.

7. Enter a note and click **OK**.  
*Result:* An annotation appears on the graph.
8. Click the **Select** button.  
*Result:* The button turns blue.

### 7.1.6 Removing a note from a graph

1. If necessary, click the **Graph Tools** button to open the tool palette.



2. Click the annotation that you want to edit or delete.  
*Result:* The annotation is highlighted.

3. Click the **Annotation** button.



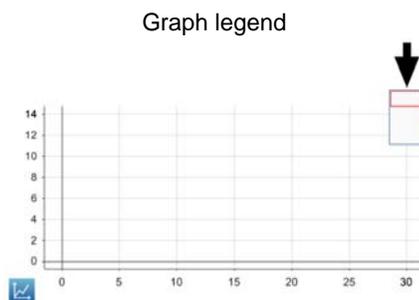
*Result:* The on-screen keyboard appears.

4. Backspace over the annotation and click **OK**.

---

### 7.1.7 Showing and hiding data runs in a graph

1. Click the graph legend in the upper right corner of the graph.  
*Result:* The legend enlarges to show available data runs.
2. Select or clear the check box next to each data run that you want to show or hide.
3. Optionally, click outside the legend to reduce the size of the legend.



---

### 7.1.8 Showing and hiding connecting lines between data points

Note: This feature is not currently supported. Please visit [www.pasco.com](http://www.pasco.com) for the latest updates to SPARKvue and documentation.

---

### 7.1.9 Changing the variable on the x- or y-axis

1. Click the **Graph Tools** button to open the tool palette.



2. Click the **Properties** button to open the properties screen.



3. For each axis, click the **Measurement** box and select a measurement or other variable.

### 7.1.10 Displaying multiple variables on the y-axis

Complete these steps to create a page containing two graphs with a different variable on the y-axis of each graph and time on both x-axes.

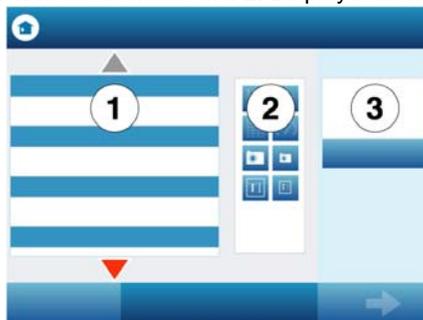
1. Do one of the following to open the page-build screen.
  - ◆ If a SPARKlab is open, click the **New Page** button.



- ◆ If the Home screen is open, click **BUILD**.

*Result:* The page-build screen opens.

**Page-build screen:** 1. Measurements list. 2. Display buttons. 3. Preview section.



2. In the measurements list, click one of the measurements or variables that you would like to appear on the y-axis.

The selected measurement is highlighted.

3. Click the **Graph** button.



*Result:* The first graph appears in the preview section of the page-build screen.

4. In the measurements list, click the second measurement or variable that you would like to appear on the y-axis.

The selected measurement is highlighted.

5. Click the **Graph** button.



*Result:* The second graph appears in the preview section of the page-build screen.

6. Click **OK**.

*Task result:* A new page is created with two graphs displaying both variables on the y-axes.

### 7.1.11 Displaying multiple graphs

- ◆ To display multiple graphs, each on its own page, repeat the steps in Tech Tip 7.1.1 for each graph.
- ◆ To display two graphs on one page, complete the steps in Tech Tip 7.1.10.

**7.1.12** Drawing a prediction

1. Click the **Graph Tools** button to open the tool palette.



2. Click the **Prediction** button.



3. Do one of the following:
  - ◆ Trace a continuous curve on the graph.
  - ◆ Click several locations on the graph to draw a series of connected points.
4. Click **OK**.

**7.2** Table**7.2.1** Displaying data in a table

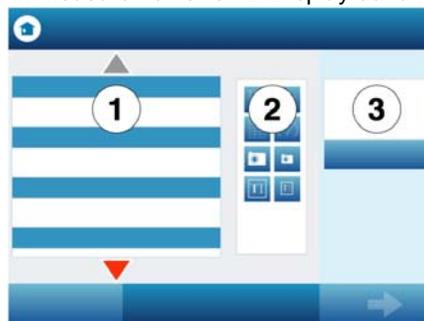
1. Do one of the following to open the page-build screen.
  - ◆ If a SPARKlab is open, click the **New Page** button.



- ◆ If the Home screen is open, click **BUILD**.

*Result:* The page-build screen opens.

**Page-build screen:** 1. Measurements list. 2. Display buttons. 3. Preview section.



2. Click a measurement (or up to six measurements) to select them.  
Selected measurements are highlighted.
3. Click the **Table** button.



4. Click **OK**.

*Result:* A new page is created containing a table with a column for time and a column for each measurement that you selected. (Time is not included if you selected six measurements.)

5. Optionally, remove unwanted columns:
  - a. Click the **Table Tools** button to open the tool palette.



- b. Click the **Select** button.



*Result:* The button turns orange.

- c. Click the column that you want to remove.
  - d. Click the **Remove Column** button.



6. Optionally, add additional columns <sup>◆(7.2.2)</sup>.

---

### 7.2.2 Adding a measurement to a table

1. Click the **Table Tools** button to open the tool palette.



2. Click the **Add Column** button.



*Result:* A new column is added to the table.

3. Select a measurement for the new column:
  - a. Click the **Properties** button to open the properties screen.



- b. Click the **Measurement** box and select the measurement or other variable that you want to see in the new column.
  - c. Click **OK**.

### 7.2.3 Manually entering data into a table

1. Do one of the following to open the page-build screen.
  - ◆ If a SPARKlab is open, click the **New Page** button.



- ◆ If the Home screen is open, click **BUILD**.

*Result:* The page-build screen opens.

**Page-build screen:** 1. Measurements list. 2. Display buttons. 3. Preview section.



2. Create an empty user-entered data set:
  - a. In the measurements list under **User-entered Number Data** or under **User-entered Text Data**, click **Create Data Set**.
 

*Result:* The Define the Data Set screen opens.
  - b. Click the **Measurement Name** box, type a name that describes the data or text that you plan to enter manually, and click **OK**.
  - c. Click **OK** to return to the page-build screen.

*Result:* The user-entered data set that you created now appears in the measurements list on the page-build screen.
3. In the measurements list, click the data set that you just created to select it.
 

*Result:* The selected data set is highlighted.
4. Click the **Table** button.



5. Click **OK**.
 

*Result:* A table prepared to accept the manually entered data appears.
6. Enter data:
  - a. If the table tool palette is not already open, click the **Table Tools** button.



- b. If it is not already highlighted in the tool palette, click the **Select** button.



*Result:* The **Select** button turns orange.

- c. Click the table cell where you would like to enter data.

*Result:* A yellow box appears around the cell.

- d. Click the **Data Entry** button.



*Result:* The on-screen keyboard opens.

- e. Type a number or text and click **OK**.
7. Repeat step 6 to enter data in other cells of the table.

---

### 7.2.4 Showing and hiding data runs in a table

1. Click the run number at the top of the column.

*Result:* A list of available runs appears.

2. Click the run that you want to see.

---

### 7.2.5 Changing the variable displayed in a column

1. Click the **Table Tools** button to open the tool palette.



2. Click the **Properties** button to open the properties screen.



3. Click the **Column** box and select the column that you want to change.  
Columns are number 1, 2, 3, and so on from left to right.
4. Click the **Measurement** box and select the measurement or other variable that you want to see in the column.
5. Click **OK**.

---

## 7.3 Digits display

---

### 7.3.1 Displaying data in a digits display

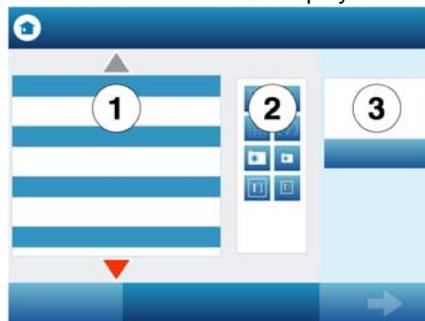
1. Do one of the following to open the page-build screen.
  - ◆ If a SPARKlab is open, click the **New Page** button.



- ◆ If the Home screen is open, click **BUILD**.

*Result:* The page-build screen opens.

**Page-build screen:** 1. Measurements list. 2. Display buttons. 3. Preview section.



2. Click one measurement to select it.  
The selected measurement is highlighted.
3. Click the **Digits Display** button.



4. Click **OK**.

*Task result:* A new page is created with a digits display.

### 7.3.2 Adding a measurement to a digits display

Complete these steps to create a page displaying more than one measurement in digits displays.

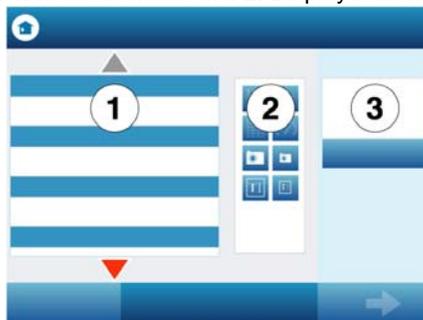
- Do one of the following to open the page-build screen.
  - If a SPARKlab is open, click the **New Page** button.



- If the Home screen is open, click **BUILD**.

*Result:* The page-build screen opens.

**Page-build screen:** 1. Measurements list. 2. Display buttons. 3. Preview section.



- Click one measurement to select it.  
The selected measurement is highlighted.
- Click the **Digits Display** button.



*Result:* The digits display appears in the preview section of the page-build screen.

- Repeat steps 2 and 3 to add other measurements to the same page.
- Click **OK**.

*Task result:* A new page is created with multiple digits displays.

## Section 8: Data run operations

### 8.1 Deleting a data run

- Click the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

- Click **MANAGE RUNS**.

*Result:* The Manage Runs screen opens.

3. Do one of the following:
  - ◆ Click **Delete Last Run**.
  - ◆ Click **Delete All Runs**.
  - ◆ Click **Delete Run** and select the run that you want to delete.
4. Click **DONE** to close the Manage Runs screen.
5. Click **OK** to close the Experiment Tools screen.

## 8.2 Naming a data run

Complete these steps to name a data run on a graph by attaching a note to it.

1. If there is more than one data run on the graph, first select the run that the note will be attached to:
  - a. Click the graph legend.  
*Result:* The legend enlarges.
  - b. In the legend, click the symbol of the run that you want to select.  
*Result:* The red outline moves to the selected run.
2. Click the **Graph Tools** button to open the tool palette.

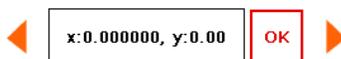


3. Click the **Select** button.



*Result:* The button turns orange.

4. Click a point on the graph.
5. Click **OK**.



6. Click the **Annotation** button.



*Result:* The on-screen keyboard appears.

7. Enter a note and click **OK**.  
*Result:* An annotation appears on the graph.
8. Click the **Select** button.  
*Result:* The button turns blue.

## Section 9: Analyzing data

### 9.1 Finding the coordinates of a point in a graph

Complete these steps to select a point on a graph and display its coordinates:

1. If more than one data run is displayed, first select a run:
  - a. Click the graph legend.  
*Result:* The legend enlarges.
  - b. In the legend, click the symbol of the run that you want to select.  
*Result:* The red outline moves to the selected run.
2. Click the **Graph Tools** button to open the tool palette.

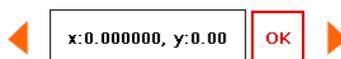


3. Click the **Select** button.



*Result:* The button turns orange.

4. Click a point on the graph.  
*Result:* The x- and y-values of the selected point are displayed.
5. Optionally, click the arrows on the point selector to change which point is selected.



6. Click **OK**.
7. Optionally, click the **Coordinates** button.



*Result:* An annotation displaying the x- and y-values appears on the graph.

To clear the annotation, click the **Coordinates** button again. To clear the selection, click the **Select** button again.

### 9.2 Measuring the difference between two points in a graph

Complete these steps to select a range of points and display the change-in-x and change-in-y between the first and last points in the selected range:

1. If more than one data run is displayed, first select a run:
  - a. Click the graph legend.  
*Result:* The legend enlarges.
  - b. In the legend, click the symbol of the run that you want to select.  
*Result:* The red outline moves to the selected run.
2. Click the **Graph Tools** button to open the tool palette.



- Click the **Select** button.

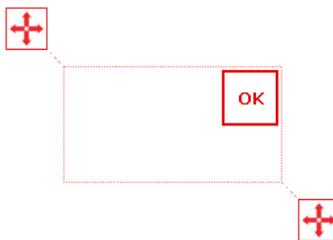


*Result:* The button turns orange.

- Click a point on the graph; then, within one second, click another point on the graph.

The two locations that you click define the corners of a selection box.

*Result:* A selection box appears. Data points inside the box are highlighted.



- Optionally, adjust the size and position of the selection box by dragging the handles at the corners of the box.



- When the desired data points are highlighted, click **OK**.

*Result:* The selection box disappears, but the points remain selected.

- Click the **Coordinates** button.



*Task result:* An annotation with the following information appears on the graph:

- ◆ The x- and y-values of the first point in the selected range ( $x_1$  and  $y_1$ ),
- ◆ The x- and y-values of the last point in the selected range ( $x_2$  and  $y_2$ ), and
- ◆ The x- and y-differences between those two points ( $dx$  and  $dy$ ).

Note: To clear the annotation, click the **Coordinates** button again. To clear the selection, click the **Select** button again.

### 9.3

#### Finding the slope at a point on a data plot

Complete these steps to display the slope at a selected point.

- If more than one data run is displayed, first select a run:

- Click the graph legend.

*Result:* The legend enlarges.

- In the legend, click the symbol of the run that you want to select.

*Result:* The red outline moves to the selected run.

- Click the **Graph Tools** button to open the tool palette.

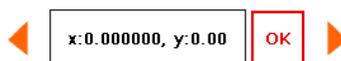


3. Click the **Slope Tool** button.



*Result:* A slope tool appears on the graph displaying the slope at one point. If no data points have been selected, the slope tool appears in the middle of the data run. If data points have been selected <sup>◆(7.1.4)</sup>, the slope tool appears in the middle of the selected range. If a single data point has been selected, the slope tool appears on that point.

4. Click the arrows on the point selector to move the slope tool to nearby points.



To hide the slope tool, click the **Slope Tool** button again.

## 9.4 Viewing statistics of data

Complete these steps to see the minimum, maximum, mean, standard deviation, count, and area-under-the-curve of a data run displayed in a graph:

1. If more than one data run is displayed, first select a run:
  - a. Click the graph legend.  
*Result:* The legend enlarges.
  - b. In the legend, click the symbol of the run that you want to select.  
*Result:* The red outline moves to the selected run.
2. Click the **Graph Tools** button to open the tool palette.



3. Click the **Statistics** button to open the Statistics screen.



4. Click one or more of the statistics.  
Selected statistics are highlighted.
5. Click **OK**.  
*Result:* Statistics appear on the graph.
6. Optionally, select part of the data set for statistics to be applied to. <sup>◆(7.1.4)</sup>

Note: To remove the statistics, click the **Statistics** button again.

## 9.5 Applying a curve fit

Complete these steps to apply a linear, quadratic, power, inverse, inverse square, or sine fit to a data run:

1. If more than one data run is displayed, first select a run:
  - a. Click the graph legend.  
*Result:* The legend enlarges.
  - b. In the legend, click the symbol of the run that you want to select.  
*Result:* The red outline moves to the selected run.

- Click the **Graph Tools** button to open the tool palette.



- Click the **Curve Fit** button to open the Curve Fit screen.



- Click one curve fit to select it.
- Click **OK**.

*Result:* The curve and parameters of the curve appear on the graph.

- Optionally, select part of the data set for the curve fit to be applied to. <sup>◆(7.1.4)</sup>

## 9.6

### Finding the slope and intercept of a best-fit line

Complete these steps to apply a linear fit to a data run:

- If more than one data run is displayed, first select a run:
  - Click the graph legend.

*Result:* The legend enlarges.

- In the legend, click the symbol of the run that you want to select.

*Result:* The red outline moves to the selected run.

- Click the **Graph Tools** button to open the tool palette.



- Click the **Curve Fit** button to open the Curve Fit screen.



- Click **Linear Fit** to select it.
- Click **OK**.

*Result:* The best-fit with its slope ( $m$ ) and y-intercept ( $b$ ) appear on the graph.

- Optionally, select part of the data set for the linear fit to be applied to. <sup>◆(7.1.4)</sup>

## 9.7

### Finding the area under a curve

Complete these steps to see the area-under-the-curve of a data run:

- If more than one data run is displayed, first select a run:
  - Click the graph legend.

*Result:* The legend enlarges.

- In the legend, click the symbol of the run that you want to select.

*Result:* The red outline moves to the selected run.

- Click the **Graph Tools** button to open the tool palette.



3. Click the **Statistics** button to open the Statistics screen.



4. Click **Area** to select it.
5. Click **OK**.

*Result:* The area under the curve is shaded and the value appears on the graph.

6. Optionally, select part of the data set for the area operation to be applied to. <sup>(7.1.4)</sup>

Note: To remove the area, click the **Statistics** button again.

---

## Section 10: Data operations

---

### 10.1 Editing data manually

1. Click the **Table Tools** button to open the tool palette.



2. Click the **Select** button.



*Result:* The button turns orange.

3. Click the table cell where you would like to edit data.

*Result:* A yellow box appears around the cell.

4. Click the **Data Entry** button.



*Result:* The on-screen keyboard opens.

5. Type a number or text and click **OK**.

Repeat this task to edit data in other cells.

---

### 10.2 Deleting data points

Complete these steps to delete manually entered data from a table cell.

1. If the table tool palette is not already open, click the **Table Tools** button.



2. If it is not already highlighted in the tool palette, click the **Select** button.



*Result:* The **Select** button turns orange.

- Click the table cell where you would like to delete data.  
*Result:* A yellow box appears around the cell.
- Click the **Data Entry** button.



*Result:* The on-screen keyboard opens.

- Backspace over the data and click **OK**.

## 10.3 Creating calculated data

- Click the **Experiment Tools** button.



*Result:* The Experiment Tools screen opens.

- Click **CALCULATED DATA**.

*Result:* The calculator screen opens.

- Type an expression.

If you have been given an expression in your lab instructions, enter it into the calculator exactly as it appears in the instructions; however, if you change the text to the left of the equals sign, do not use spaces between words.

- ◆ To insert a measurement into the expression, click **Measurements**.
- ◆ To cycle through the various functions available for use in the expression, click **Functions**.
- ◆ To enter words or letters into the expression, click the **Letters** button.



- Click **Return** to complete the calculation.
- Click **DONE** to close the calculator screen.
- Click **OK** to close the Experiment Tools screen.

*Task result:* The calculation is now available for viewing in a graph, table, or other display.

## Section 11: Saving and sharing your experiment

### 11.1 Saving your experiment

Complete these steps to save your work.

- Click the **Sharing** button to open the Sharing screen.



- Click **SAVE FILE AS**.

*Result:* The Save As window opens.

- Navigate to a folder where you would like to save the file.

4. Enter a filename.
5. Click **Save**.
6. Click **DONE** to close the Sharing screen.

Note: After you have saved a file once using the procedure above, you can quickly save the file again by clicking **SAVE FILE** instead of **SAVE FILE AS** in the Sharing screen.

---

### 11.2 Printing

To print one or more pages from a SPARKlab, complete these steps to capture pages in the journal and print the journal.

1. On each SPARKlab page that you wish to print, click the **Snapshot** button.



*Result:* Each time you click the **Snapshot** button, the Snapshot Quick View appears briefly and an image of the page is added to the journal.

2. Click the **Sharing** button to open the Sharing screen.



3. Click the **JOURNAL** tab.
4. Click **PRINT JOURNAL** to open the Print window.
5. Select a printer and click **Print**.

*Result:* The journal is printed, and the Print window closes.

6. Click **DONE** to close the Sharing screen.

---

### 11.3 Exporting data for use in mapping software

When you export data, all data in the experiment that you have open in SPARKvue will be saved in a file (or files) that can be opened by the mapping software.

Complete these steps to export data:

1. Click the **Sharing** button to open the Sharing screen.



2. Click **EXPORT DATA** to open the Export Data window.
3. Navigate to a folder where you would like to save the file.
4. Enter a filename.
5. Click **Save**.

*Result:* The Export Data window closes and a data file is saved. If there were multiple data runs in the SPARKvue, a separate, numbered file is saved for each data run.

6. Click **DONE** to close the Sharing screen.

# Appendix C: Xplorer GLX Tech Tips

## Section 1: Starting an experiment

---

### 1.1 Opening a file

1. Press  to open the Home screen.
2. Use the arrow keys to highlight the **Data Files** icon and press .



*Result:* The Data Files screen opens.

3. In the Data Files screen, use the arrow keys to highlight the desired file and press  (**Open**).

*Result:* The file opens. The name of the open file appears near the top of the screen.

4. Press  to return to the Home screen.

---

### 1.2 Starting a new experiment

1. Press  to open the Home screen.
2. Use the arrow keys to highlight the **Data Files** icon and press .



*Result:* The Data Files screen opens.

3. Press  to open the **Files** menu.
4. In the **Files** menu, use the arrow keys to highlight **New** and press .

*Result:* A new experiment is opened on the GLX.

5. Press  to return to the Home screen.

## Section 2: Setting up measurements and data collection

---

### 2.1 Connecting a sensor to the GLX

- ◆ If you have a PASPORT sensor, plug it into one of the PASPORT ports on the GLX.
- ◆ If you have a temperature probe (fast-response or stainless steel), plug it into one of the temperature ports on the side of the GLX.

The temperature ports are labeled with the icons  and .

- ◆ If you have a voltage probe, plug it into the voltage port on the side of the GLX.

The voltage port is labeled with the icon .

*Task result:* The GLX detects the sensor and adds it to your experiment.

**2.2** Connecting multiple sensors to the GLX

Complete any or all of the following steps.

- ◆ Connect up to four PASPORT sensors into the PASPORT ports.
- ◆ Connect one or two temperature probes to the temperature ports.
- ◆ Connect a voltage probe to the voltage port.

*Task result:* The GLX detects all connected sensors and adds them to your experiment.

---

**2.3** Programming the GLX to calculate lung volume

After connecting a spirometer to the GLX<sup>♦(2.1)</sup>, complete these steps:

1. Press  $\text{Ⓜ} + \text{F3}$  together to open the Calculator screen.
2. If there are previous calculations in the Calculator screen, use the arrow keys to move the cursor to the first blank line.
3. Complete the substeps below to enter this expression:

lung volume = v0-[Total Flow (liters)]

- a. Press  $\text{F4}$  to open the **Edit** menu, use the arrow keys to select **Num Lock** and press  $\text{✓}$ .

*Result:* Num lock is turned off, making it possible to type letters on the keypad.

- b. Type: lung volume = v0-

- c. Press  $\text{F2}$  to open the **[Data]** menu, use the arrow keys to select **Total Flow**, and press  $\text{✓}$ .

*Result:* The units menu opens.

- d. In the units menu, use the arrow keys to select **liters** and press  $\text{✓}$ .

*Result:* This expression now appears:

lung volume = v0-[Total Flow (liters)]

4. Press  $\text{✓}$ .

*Result:* A second expression appears on the next line: v0 =

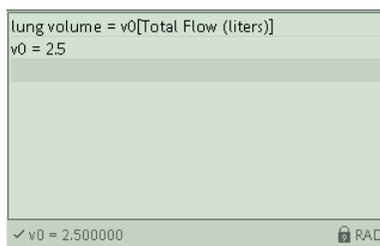
5. Type the test subject's functional residual capacity (FRC) in liters.

If you do not know the test subject's FRC, type 2.5.

*Result:* The second line should now be similar to: v0 = 2.5

6. Press  again.

*Result:* The calculation is complete.



7. Press  to return to the Home screen.

*Task result:* The lung volume calculation is now available for viewing in a graph, table, or other display.

## Section 3: Calibrating sensors

### 3.1 Calibrating a carbon dioxide gas sensor

Once the Carbon Dioxide Gas sensor has been calibrated using this method, it retains its calibration, even after it has been unplugged.

1. Take the sensor and an empty sample bottle outdoors.  
If necessary, unplug the sensor from the GLX.
2. Fill the sample bottle with fresh outdoor air.
3. Insert the probe of the sensor into the bottle and push the rubber stopper of the sensor into the neck of the bottle.
4. If you have disconnected the sensor, return to the GLX and connect the sensor to it.
5. Press the **Calibrate** button on the sensor and hold it for three seconds.

*Result:* The green light illuminates indicating that calibration is in progress.

6. Wait approximately one minute.

When the green light flashes slowly, calibration is finished. If the light flashes rapidly (five times per second), an error has occurred and the sensor has not been calibrated.

### 3.2 Calibrating a colorimeter

1. Place a cuvette filled with distilled water in the colorimeter and close the lid.
2. Press the **Calibration** button on the sensor.

*Result:* The light in the button illuminates indicating that calibration is in progress.

3. Wait for the light to turn off.

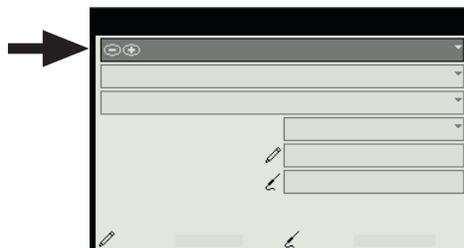
### 3.3 Calibrating a dissolved oxygen sensor

1. Obtain barometric pressure and temperature at your location.

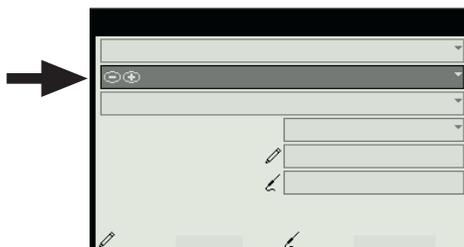
2. Refer to the solubility table included with the sensor and find the standard dissolved oxygen value for the temperature and barometric pressure at your location.
3. Press  $\text{⏏}$  +  $\text{F4}$  together to open the Sensors screen.
4. Press  $\text{F4}$  to open the **Sensors** menu, use the arrow keys to select **Calibrate**, and press  $\text{✓}$ .

*Result:* The Calibrate Sensors screen opens.

5. In the Calibrate Sensors screen, the first box should read **Dissolved Oxygen Sensor**. If it does not, press  $\text{+}$  or  $\text{-}$  to select **Dissolved Oxygen Sensor**.



6. The second box should read, **Dissolved Oxygen (mg/L)**. If it does not, press  $\text{⏏}$  to highlight the second box, and press  $\text{+}$  or  $\text{-}$  to select **Dissolved Oxygen (mg/L)**.



7. Press  $\text{⏏}$  a few times to highlight the **Pt 2 (mg/L)** box (the first box labeled **Pt 2**).



8. Press  $\text{✓}$ , type the value that you determined from the solubility table, and press  $\text{✓}$  again.
9. Place about 5 mL (to a height of about 1 cm) of deionized water into a clean soaker bottle. Slip the cap and O-ring of the soaker bottle over the end of the probe.
10. Insert the probe into the soaker bottle and screw on the lid. Adjust the height of the probe to about 2 cm above the top of the water.
11. Shake the soaker bottle vigorously for about 10 seconds. Shake off any large water drops from the membrane at the end of the probe.

12. Press **F4** (**Read Pt 2**).  
*Result:* The output from the sensor is transferred to the **Pt 2 (units)** box (the second box labeled **Pt 2**).
13. Press **F1** (**OK**) to close the Calibrate Sensors screen.
14. Press **Home** to return to the Home screen.

### 3.4 Calibrating a drop counter

The drop counter is calibrated *after* data is taken, using the GLX's calculator to correlate the number of drops counted to the volume of liquid dispensed from the burette. The following steps illustrate the calibration procedure. Note that in performing the experiment, these steps will be interspersed with other experiment procedure steps.

Note: Using this method, you will ignore the "Volume" measurement output directly from the sensor. Instead, volume is calculated by the GLX. This method takes into account the fact that drop size may be different each time the burette is used. By measuring the total volume dispensed during the actual experiment, a more accurate correlation between volume and drop count is obtained.

1. Set up the drop counter on a stand with a liquid-filled burette, a beaker, stir-plate, and other equipment.  
See your lab instructions for details.
2. Write down the initial volume of liquid in the burette.
3. Press **Play** to start recording.
4. Slowly turn the stopcock to start delivering liquid at about 2 drops per second.
5. After the necessary amount of liquid has been dispensed from the burette, close the stopcock.
6. Press **Play** to stop recording.
7. Read the final volume of liquid in the burette and determine the net volume that was dispensed.
8. Read the final drop count (the total number of drops dispensed) in a graph or table display.
9. Press **Home** + **F3** together to open the Calculator screen.
10. If there are previous calculations in the Calculator screen, use the arrow keys to move the cursor to the first blank line.
11. Complete the substeps below to enter this expression:  

$$\text{volume} = [\text{Drop Count (drops)}] * 100 / 3050$$
  - a. Press **F4** to open the **Edit** menu, use the arrow keys to select **Num Lock** and press **Checkmark**.  
*Result:* Num lock is turned off, making it possible to type letters on the keypad.
  - b. Type: volume =
  - c. Press **F2** to open the **[Data]** menu, use the arrow keys to select **Drop Count**, and press **Checkmark**.  
*Result:* This expression now appears: volume = [Drop Count (drops)]

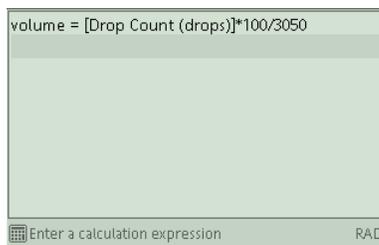
- d. Type something similar to: \*100/3050

In this example, “100” is the total volume dispensed (in mL) and “3050” is the final drop count. When you type the expression, use your own values for these two quantities.

*Result:* This expression now appears similar to:  
 volume = [Drop Count (drops)]\*100/3050

12. Press  $\checkmark$ .

*Result:* The calculation is complete.



13. Press  $\leftarrow$  to return to the Home screen.

*Task result:* The calibrated volume calculation is now available for viewing in a graph, table, or other display.

### 3.5 Calibrating an oxygen gas sensor

Once the Oxygen Gas sensor has been calibrated using this method, it retains its calibration, even after it has been unplugged.

1. Take the sensor and an empty sample bottle outdoors.  
 If necessary, unplug the sensor from the GLX.
2. Fill the sample bottle with fresh outdoor air.
3. Insert the probe of the sensor into the bottle and push the rubber stopper of the sensor into the neck of the bottle.
4. If you have disconnected the sensor, return to the GLX and connect the sensor to it.
5. Press the **CAL (20.9%)** button on the sensor and hold it for three seconds.

*Task result:* The green light flashes on and off for 4 seconds, indicating that calibration is in progress. When the light turns off, calibration is finished. If the light flashes rapidly (5 times per second), an error has occurred and the sensor has not been calibrated.

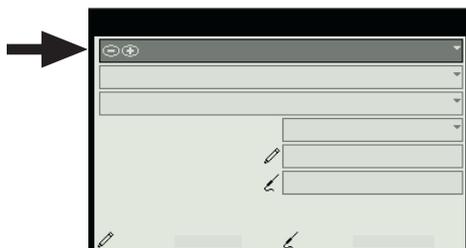
### 3.6 Calibrating a pH sensor

You will need buffer solutions of pH 4 and pH 10 and deionized water.

1. Press  $\leftarrow$  +  $F4$  together to open the Sensors screen.
2. Press  $F4$  to open the **Sensors** menu, use the arrow keys to select **Calibrate**, and press  $\checkmark$ .

*Result:* The Calibrate Sensors screen opens.

- In the Calibrate Sensors screen, the first box should read **pH Sensor**. If it does not, press  $\oplus$  or  $\ominus$  to select **pH Sensor**.



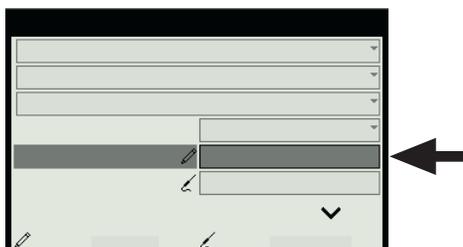
- The second box should read, **pH**. If it does not, press  $\checkmark$  to highlight the second box, and press  $\oplus$  or  $\ominus$  to select **pH**.



- Place the pH probe into the pH 4 buffer solution and wait for the reading to stabilize.

The reading is indicated near the bottom of the screen.

- Press  $\checkmark$  to highlight the **Pt 1 (pH)** box (the first box labeled **Pt 1**).



- Press  $\checkmark$ , type 4 (the known pH of the buffer solution), and press  $\checkmark$  again.

- Press  $F3$  (**Read Pt 1**).

*Result:* The output from the sensor is transferred to the **Pt 1 (units)** box (the second box labeled **Pt 1**).

- Rinse the probe with deionized water.
- Place the pH probe into the pH 10 buffer solution and wait for the reading to stabilize.

- Press  to highlight the **Pt 2 (pH)** box (the first box labeled **Pt 2**).



Note: As you press , the screen will scroll to make this box visible.

- Press , type 10 (the known pH of the buffer solution), and press  again.
- Press  (**Read Pt 2**).  
*Result:* The output from the sensor is transferred to the **Pt 2 (units)** box (the second box labeled **Pt 2**).
- Press  (**OK**) to close the Calibrate Sensors screen.
- Press  to return to the Home screen.

---

### 3.7 Calibrating a turbidity sensor

- Place a cuvette filled with distilled water in the turbidity sensor and close the lid.
  - Press the **Calibration** button on the sensor.  
*Result:* The green light in the button illuminates.
  - When the light starts blinking, replace the cuvette with the standard 100 NTU cuvette (included with the sensor) and close the lid.
  - Press the button again.  
*Result:* The light in the button illuminates.
- Task result:* When the light turns off, calibration is complete.

---

### 3.8 Calibrating an ethanol sensor

You will need a solution of 1% ethanol in water. The solution should be at the same temperature as the solutions to be measured.

Once the sensor has been calibrated using this method, it retains its calibration, even after it has been unplugged.

- Connect the sensor to the GLX and wait about 10 minutes for the probe to warm up.
- Place the probe about 1 cm above the 1% ethanol solution.
- Observe the ethanol concentration reading on the GLX and wait until the reading stabilizes.
- Press and hold the **1% CAL** button on the sensor for 4 seconds.

*Task result:* Immediately after a successful calibration, the sensor's output reads 1%, and the button is illuminated.

---

## Section 4: Sensor operations

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### 4.1 Using a colorimeter with a specific color of light

The colorimeter makes measurements for four different colors; be sure to choose the measurements for the appropriate color.

The colorimeter contains four separate light sources of different colors: red (660 nm), orange (610 nm), green (565 nm), and blue (468 nm). Internally, the colorimeter uses all four light sources simultaneously and makes separate measurements for each color. However, in most experiments, you will be concerned only with one color and ignore the measurements associated with other colors. Your lab instructions may tell you which color to use, or you may need to determine the appropriate color on your own.

For each color there are two measurements: transmittance and absorbance.

As you proceed with your experiment, whenever you set up data displays or do certain other tasks, you will see a list of all eight measurements made by the colorimeter; be sure always to choose the transmittance or absorbance measurement for the appropriate color.

---

### 4.2 Setting up a conductivity sensor for a particular sensitivity

When using a conductivity sensor, it is important to select a sensitivity appropriate to the solutions that you plan to test. If the selected sensitivity is too high, the sensor may lack the range necessary to measure your most concentrated solutions. However, if the range is larger than necessary, the measurement precision may not be high enough to detect small conductivity changes or differences.

Complete these steps to select the appropriate sensitivity and range:

1. Determine or estimate the highest concentration that you will need to measure.

Use this chart and other resources.

<b>Solution</b>	<b>Conductivity (<math>\mu\text{S}/\text{cm}</math>)</b>
Ultrapure water	0.05 to 0.75
Drinking water	50 to 1500
Ocean water	$\sim 53000$

- Select the range that best matches your measurement requirements.

The first column of this chart shows available ranges.

Measurement Range ( $\mu\text{S}/\text{cm}$ )	1x Probe Range Selection	10x Probe Range Selection
0 to 100		-
0 to 1000		
0 to 10000		
0 to 100000	-	

- Select an appropriate probe (either 1x or 10x) and connect it to the sensor.
- Use the buttons on the sensor to select an appropriate range.

Lights in the buttons indicate which range is selected.

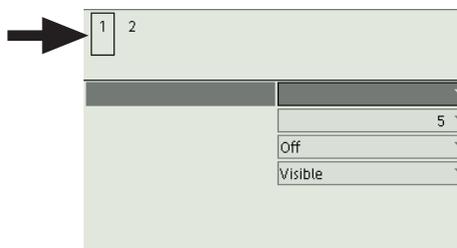
Note: As you proceed with your experiment, whenever you set up data displays or do certain other tasks, you will see two measurements made by the conductivity sensor: one for the 1x probe and one for the 10x probe; be sure always to choose the measurement that matches the attached probe.

### 4.3

Setting up a rotary motion sensor to measure linear position using the “rack” setting

- Press  +  together to open the Sensors screen.
- If there are multiple sensors connected to the GLX, press  or  to select the rotary motion sensor.

The icon of the selected sensor (near the top of the screen) has a box around it.



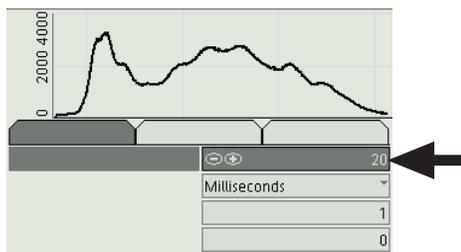
- Press  to highlight **Linear Position Scale** and press .
- Press  to highlight **Rack** and press .
- Set the **Linear Position** measurement to **Visible**, and set the other measurements to **Not Visible**:
  - Press  to highlight **Angular Position**.
  - Press  to set **Angular Position** to **Not Visible**.
  - Press  to highlight **Linear Position**.

Note: As you press , screen will scroll.

- d. Press  $\checkmark$  to set **Linear Position** to **Visible**.
6. Press  $\text{Home}$  to return to the Home screen.

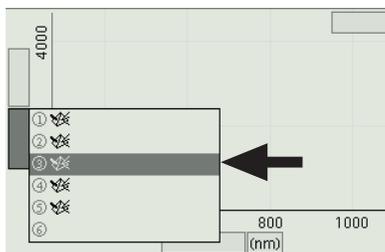
#### 4.4 Setting up the spectrometer

1. Turn on the GLX first. Make sure that the GLX is not connected to a computer. Plug the spectrometer into the GLX. If the Amadeus spectrometer is used, connect the front port of the cell holder with the spectrometer using the fiber optic cable.
2. Plug the spectrometer into the GLX.  
*Result:* The GLX will go through the initialization process.
3. Press  $\checkmark$  at the default selection (**Integration Time**) and enter 20. Press  $\checkmark$  to accept.



4. Turn off the light in the spectrometer:
  - ◆ If the Amadeus spectrometer is used, disconnect the power supply from the cell holder.
  - ◆ If the Red Tide spectrometer is used, with the  $\text{Lamp}$  key select the **Lamp** tab and press  $\checkmark$  to turn the lamp off.
5. Place the black block into the cell holder. Press  $F1$  to save the dark reference.
6. Turn on the light in the spectrometer:
  - ◆ If the Amadeus spectrometer is used, connect the power supply to the cell holder.
  - ◆ If the Red Tide spectrometer is used, with the  $\text{Lamp}$  key select the **Lamp** tab (if it is not already selected) and press  $\checkmark$  to turn the lamp on.
7. Remove the black block from the cell holder.
8. Fill a square spectroscopic cell two-thirds full with the blank solution (usually distilled water). Wipe the cell clean and dry. Place the cell into the cell holder so that the light passes through the transparent faces of the cell.
9. Press  $F2$  to save the blank reference.
10. Press  $F4$  to close the configuration screen.
11. Press  $\checkmark$  to highlight the vertical axis label. Press  $\checkmark$  a second time to open the axis label menu.

- With the  $\checkmark$  key select **Absorbance**. Press  $\checkmark$  to accept your selection.



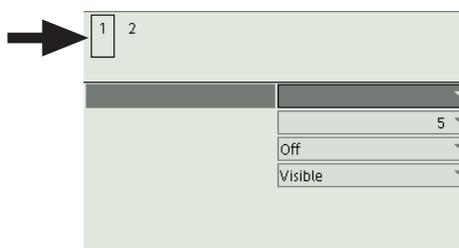
*Result:* The vertical axis changed to **Absorbance**.

## Section 5: Data measurement setup

### 5.1 Changing the sampling rate

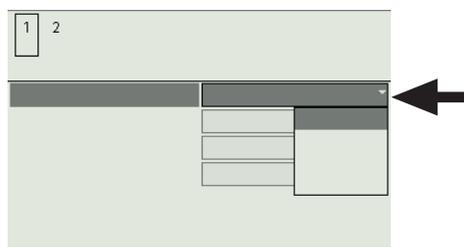
- Press  $\text{Home}$  +  $F4$  together to open the Sensors screen.
- If there are multiple sensors connected to the GLX, press  $\leftarrow$  or  $\rightarrow$  to select the sensor that you wish to change.

The icon of the selected sensor (near the top of the screen) has a box around it.

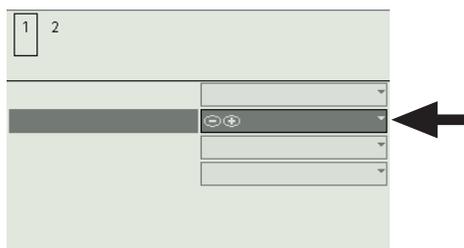


- With **Sample Rate Unit** highlighted, press  $\checkmark$ .

*Result:* A menu of units opens.



- In the menu, use the arrows keys to highlight the desired units and press  $\checkmark$ .
- Press  $\checkmark$  to highlight **Sample Rate**.



6. Press  $\oplus$  or  $\ominus$  to select a value.  
If you select units of samples/s, the value indicates the number of samples per second. If you select units of seconds, minutes, or hours, the value indicates the time between samples.
  7. Press  $\text{Home}$  to return to the Home screen.
- Repeat this task for all other connected sensors.

## 5.2

### Putting the GLX into manual sampling mode

1. Press  $\text{Home}$  +  $F4$  together to open the Sensors screen.
2. Press  $F1$  to open the **Mode** menu, use the arrow keys to highlight **Manual**, and press  $\checkmark$ .  
*Result:* The Data Properties screen opens.
3. For more instructions see Tech Tip 5.2.1 (sampling with manually entered data) and Tech Tip 5.2.2 (sampling without manually entered data).

### 5.2.1

#### Putting the GLX into manual sampling mode with manually entered data

1. Press  $\text{Home}$  +  $F4$  together to open the Sensors screen.
2. Press  $F1$  to open the **Mode** menu, use the arrow keys to highlight **Manual**, and press  $\checkmark$ .  
*Result:* The Data Properties screen opens.
3. In the Data Properties screen, with **Measurement Name** highlighted, press  $\checkmark$ , type a name that describes the data or text that you plan to enter manually, and press  $\checkmark$ .
4. Press  $F1$  (**OK**).  
*Result:* The Data Properties screen closes. The GLX is now in manual sampling mode.
5. Press  $\text{Home}$  +  $F2$  together to open the Table screen.  
*Result:* A two-column table appears prepared to display sensor data in the first column and your manually entered data in the second column.
6. If desired, select a different measurement for display in the first column.  $\blacklozenge^{(7.2.5)}$

### 5.2.2

#### Putting the GLX into manual sampling mode without manually entered data

1. Press  $\text{Home}$  +  $F4$  together to open the Sensors screen.
2. Press  $F1$  to open the **Mode** menu, use the arrow keys to highlight **Manual**, and press  $\checkmark$ .  
*Result:* The Data Properties screen opens.
3. Press  $F2$  (**No Data**).  
*Result:* The Data Properties screen closes. The GLX is now in manual sampling mode.
4. Press  $\text{Home}$  to return to the Home screen.

### 5.3 Changing the units of a measurement

1. Display the measurement in a graph <sup>◆(7.1.1)</sup>, table <sup>◆(7.2.1)</sup>, or digits display <sup>◆(7.3.1)</sup>.
  2. Press  to activate the fields in the display.
  3. Use the arrow keys to move the highlight to the measurement units and press .
- Result:* A menu of available units opens.
4. In the menu, use the arrow keys to highlight the desired units and press .

### 5.4 Changing the number of digits with which a variable is displayed

1. Display the measurement in a graph <sup>◆(7.1.1)</sup>, table <sup>◆(7.2.1)</sup>, or digits display <sup>◆(7.3.1)</sup>.
  2. Press  to activate the fields in the display.
  3. Use the arrow keys to move the highlight to the measurement name and press .
- Result:* A menu opens.
4. In the menu, use the arrow keys to highlight **Data Properties** and press .
- Result:* The Data Properties screen opens.
5. In the Data Properties screen, press  to highlight **Number of Digits**.
  6. Press  or  to increase or decrease the number of digits to be displayed to the right of the decimal point.
  7. Press  (**OK**) to close the Data Properties screen.

### 5.5 Setting up digital timers

#### 5.5.1 Setting up a photogate to measure velocity

1. Connect a Digital Adapter to the GLX.
  2. Connect a photogate to the Digital Adapter.
- Result:* A menu opens.
3. In the menu, use the arrow keys to highlight **Photogate Timing** and press .
- Result:* The Timing screen opens.
4. In the Timing screen, with **Flag Length** highlighted, press , type the length (in meters) of the flag that will pass through the photogate, and press  again.
  5. Set the **Velocity In Gate** measurement to **Visible**, and set the other measurements to **Not Visible**:
    - a. Use the arrow keys to highlight **Time in Gate**.
    - b. Press  to set **Time in Gate** to **Not Visible**.
    - c. Use the arrow keys to highlight **Velocity in Gate**.
    - d. Press  to set **Velocity in Gate** to **Visible**.

- e. Use the arrow keys to highlight **Time Between Gates**.
  - f. Press  $\checkmark$  to set **Time Between Gates** to **Not Visible**.
6. Press  $\text{Home}$  to return to the Home screen.

*Task result:* The GLX is now set up to measure and display the magnitude of velocity of an object passing through the photogate.

### 5.5.2 Setting up a photogate to measure the period of a pendulum

1. Connect a Digital Adapter to the GLX.
2. Connect a photogate to the Digital Adapter.  
*Result:* A menu opens.
3. In the menu, use the arrow keys to highlight **Photogate and Pendulum** and press  $\checkmark$ .  
*Result:* The Timing screen opens.
4. Set the **Velocity** measurement to **Not Visible**:
  - a. Use the arrow keys to highlight **Velocity**.
  - b. Press  $\checkmark$  to set **Velocity** to **Not Visible**.
5. Press  $\text{Home}$  to return to the Home screen.

*Task result:* The GLX is now set up to measure and display the period of a pendulum passing through the photogate.

### 5.5.3 Setting up a Free Fall Adapter to measure time of fall

1. Connect a Digital Adapter to the GLX.
2. Connect a Free Fall Adapter to the Digital Adapter.  
*Result:* A menu opens.
3. In the menu, use the arrow keys to highlight **Free Fall Adapter** and press  $\checkmark$ .  
*Result:* The Timing screen opens.
4. Set the **Acceleration** measurement to **Not Visible**:
  - a. Use the arrow keys to highlight **Acceleration**.
  - b. Press  $\checkmark$  to set **Acceleration** to **Not Visible**.
5. Press  $\text{Home}$  to return to the Home screen.

*Task result:* The GLX is now set up to measure and display the time for an object to fall from the clip to the pad of the Free Fall Adapter.

### 5.5.4 Setting up photogates to measure time between gates

1. Connect a Digital Adapter to the GLX.
2. Connect a photogate to the Digital Adapter.  
*Result:* A menu opens.
3. Connect a second photogate to the Digital Adapter.

4. In the menu, use the arrow keys to highlight **Photogate Timing** and press .
 

*Result:* The Timing screen opens.
5. Set the **Time Between Gates** measurement to **Visible**, and set the other measurements to **Not Visible**:
  - a. Use the arrow keys to highlight **Time in Gate**.
  - b. Press  to set **Time in Gate** to **Not Visible**.
6. Press  to return to the Home screen.

*Task result:* The GLX is now set up to measure and display the time for an object to pass from one photogate to the other.

## Section 6: Collecting and working with data

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### 6.1 Monitoring live data

- ◆ Create a digits display to show the measurement (or measurements) that you wish to monitor. ◆(7.3.1, 7.3.2)

*Task result:* The digits display shows live readings.

---

### 6.2 Recording a run of data

Complete these steps to record a data run in normal sampling mode:

1. Press  to start data recording.
2. Press  again to stop data recording.

Repeat this task to collect another data run.

---

### 6.3 Recording a set of manually sampled data

If the GLX is in manual sampling mode ◆(5.2), complete these steps to record a data set:

1. Press .

*Result:* A new data set is started. Live data appear in the data displays. A flag icon starts blinking in the upper right corner of the screen indicating that the GLX is ready to be triggered.

2. When you are ready to record a point, press .
3. If the GLX prompts you to enter keyboard data, enter a number or text and press  (**OK**).

*Note:* To enter text, you may first need to press  to turn off Num Lock.

4. Each time you wish to record another data point, press .
5. When you have recorded the *entire* data set, press .

*Result:* The data set is stopped.

*Note:* If you accidentally stop data collection early (by pressing  instead of ) , you will need to start over again from the beginning. If desired, delete the incomplete data set before starting again. ◆(8.1)

---

**6.3.1** Starting a manually sampled data set

- ◆ Press **▶**.

*Result:* A new data set is started. Live data appear in the data displays. A flag icon starts blinking in the upper right corner of the screen indicating that the GLX is ready to be triggered.

---

**6.3.2** Recording a manually sampled data point

1. When you are ready to record a point, press **Ⓟ**.
2. If the GLX prompts you to enter keyboard data, enter a number or text and press **F1** (**OK**).

Note: To enter text, you may first need to press **F3** to turn off Num Lock.

Note: If you accidentally stop data collection early (by pressing **▶** instead of **Ⓟ**), you will need to start over again from the beginning. If desired, delete the incomplete data set before starting again. <sup>◆(8.1)</sup>

---

**6.3.3** Stopping a manually sampled data set

- ◆ When you have recorded the *entire* data set, press **▶**.

*Result:* The data set is stopped.

---

**Section 7: Data display**

---

**7.1** Graph

---

**7.1.1** Displaying data in a graph

1. Press **Ⓢ** + **F1** together to open the Graph screen.
2. Optionally, change the variables on the x- and y-axes of the graph <sup>◆(7.1.9)</sup>.

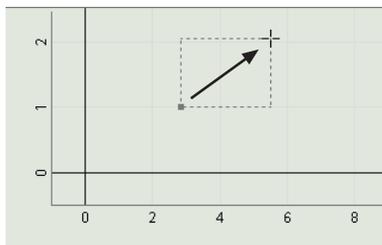
---

**7.1.2** Adjusting the scale of a graph

To adjust the x- and y-scales, do one or more of the following:

- ◆ Press **F1** (**Scale to fit**).  
*Result:* The graph adjusts to fit all data.
- ◆ Compress or stretch the graph:
  - a. Press **F2** once to put the graph into **Scale** mode.
  - b. Use the arrow keys to compress or stretch the graph vertically or horizontally.
- ◆ Move the graph:
  - a. Press **F2** twice to put the graph into **Move** mode.
  - b. Use the arrow keys to move the graph left, right, up, or down.

- ◆ Zoom in on a selected area:
  - a. Press **F3** to open the **Tools** menu, use the arrow keys to highlight **Zoom** and press **✓**.  
*Result:* A cursor appears on the graph.
  - b. Use the arrow keys to move the cursor to a position on the graph.
  - c. Press **✓**.  
*Result:* A small box appears on the graph.
  - d. Use the arrow keys to stretch out the box, defining the area that you would like to zoom in on.

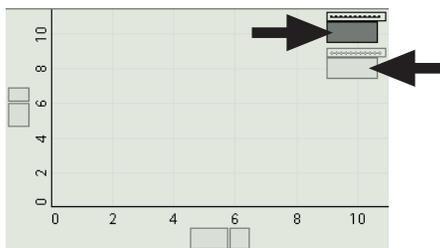


- e. Press **✓**.  
*Result:* The graph adjusts to fit the selected area.

### 7.1.3 Displaying multiple data runs on a graph

Complete these steps to display two runs on a graph.

1. In the Graph screen, press **F4** to open the **Graphs** menu.
2. In the **Graphs** menu, use the arrow keys to select **Two Runs** and press **✓**.  
*Result:* A second run is displayed in the graph.
3. Optionally, select which runs are displayed:
  - a. Press **✓** to activate the fields of the graph.
  - b. Use the arrow keys to move the highlight to one of the run numbers (or run names).



- c. Press **✓** to open a menu of available runs.
- d. In the menu, use the arrow keys to highlight the desired run and press **✓**.

### 7.1.4 Selecting data points in a graph

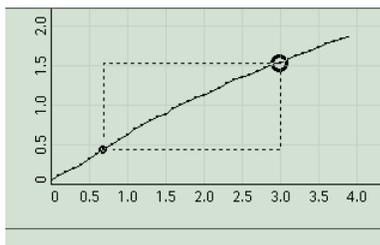
When a function such as statistics <sup>◆(9.4)</sup>, a linear fit <sup>◆(9.6)</sup>, or area <sup>◆(9.7)</sup> is shown on a graph, a box appears indicating the part of the data run that the function is

applied to. By default the box surrounds the entire data set. Complete these steps to change the range of selected points.

1. Use the arrow keys to adjust the position of the right side of the box.
2. Press  $\text{Esc} + \text{X}$  together.

*Result:* The control cursor is swapped to the opposite corner of the box.

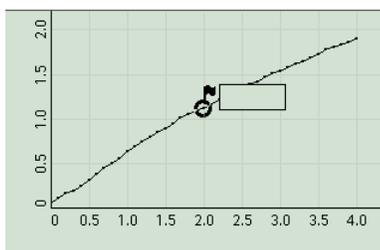
3. Use the arrow keys to adjust the position of the left side of the box.



### 7.1.5 Adding a note to a graph

1. In the Graph screen, use the arrow keys to move the data cursor to a data point where you would like to attach the note.
2. Press  $\text{F}$ .

*Result:* An empty note is added to the graph.



3. Press  $\text{F}$  again.

*Result:* A Menu Opens.

4. In the menu, use the arrow keys to highlight **Edit Note** and press  $\checkmark$ .

*Result:* The Note Editor screen opens.

5. Type the text of the note.

6. Press  $\text{F1}$  (**OK**).

*Result:* The Note Editor screen closes and the note appears on the graph.

### 7.1.6 Removing a note from a graph

1. Hold down  $\text{Esc}$  and press  $\text{B}$  or  $\text{b}$  to move the data cursor to the note.
2. Press  $\text{F}$ .

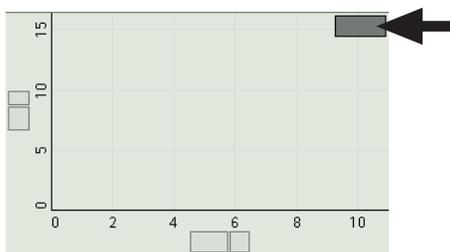
*Result:* A menu opens.

3. In the menu, use the arrow keys to highlight **Delete Note** and press  $\checkmark$ .

### 7.1.7 Showing and hiding data runs in a graph

1. In the Graph screen, press  $\checkmark$  to activate the fields.

- Use the arrow keys to move the highlight to the run number (or run name).



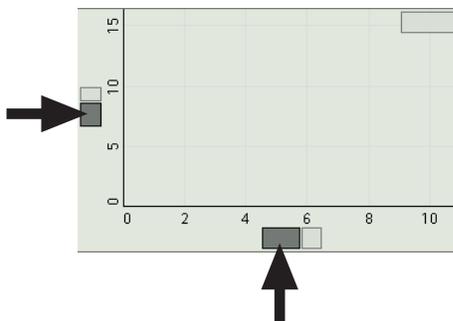
- Press  $\checkmark$ .  
*Result:* A menu of available runs opens.
- In the menu, use the arrow keys to highlight the run that you want to show (or highlight **No Data** to hide all runs) and press  $\checkmark$ .

### 7.1.8 Showing and hiding connecting lines between data points

- In the Graph screen, press  $F4$  to open the **Graphs** menu.
- In the **Graphs** menu, use the arrow keys to highlight **Connected Lines** and press  $\checkmark$ .

### 7.1.9 Changing the variable on the x- or y-axis

- In the Graph screen, press  $\checkmark$  to activate the fields.
- In the menu, use the arrow keys to move the highlight to the variable name on the x- or y-axis.



- Press  $\checkmark$ .  
*Result:* A menu of available variables opens.
- Use the arrow keys to highlight the desired variable and press  $\checkmark$ .  
If you do not see the desired variable, highlight **More** and press  $\checkmark$ .

### 7.1.10 Displaying multiple variables on the y-axis

Complete these steps display two variables on the y-axis.

- In the Graph screen, press  $F4$  to open the **Graphs** menu.
- In the **Graphs** menu, use the arrow keys to select **Two Measurements** and press  $\checkmark$ .  
*Result:* A second variable is displayed on the y-axis.
- Optionally, change one or both of the variables displayed on the y-axis  $\blacklozenge$ (7.1.9).

---

**7.1.11** Displaying multiple graphs

Do one of the following:

- ◆ Add another graph on a separate page:
    - a. In the Graph screen, press  $\text{F4}$  to open the **Graphs** menu.
    - b. In the **Graphs** menu, use the arrow keys to highlight **New Graph Page** and press  $\checkmark$ .  
*Result:* A new page is created.
    - c. Optionally, change the variables on the x- and y-axes  $\diamond(7.1.9)$ .
  - ◆ Add a second graph to an existing graph page:
    - a. In the Graph screen, press  $\text{F4}$  to open the **Graphs** menu.
    - b. In the **Graphs** menu, use the arrow keys to highlight **Two Graphs** and press  $\checkmark$ .  
*Result:* A second graph is added to the Graph screen.
    - c. Optionally, change the variables on the x- and y-axes  $\diamond(7.1.9)$ .
- 

**7.1.12** Drawing a prediction

1. On paper, sketch your graph. Label the axes and indicate the scale.
  2. Draw your prediction on the sketch.
- 

**7.2** Table**7.2.1** Displaying data in a table

1. Press  $\text{F1} + \text{F2}$  together to open the Table screen.
  2. Optionally, change the variables displayed in the columns  $\diamond(7.2.5)$ .
  3. Optionally, add additional columns  $\diamond(7.2.2)$ .
- 

**7.2.2** Adding a measurement to a table

1. In the Table screen, press  $\text{F4}$  to open the **Tables** menu.
  2. In the **Tables** menu, use the arrow keys to select the number of columns you would like to display and press  $\checkmark$ .
  3. Select variables to display in the new columns  $\diamond(7.2.5)$ .
- 

**7.2.3** Manually entering data into a table

1. Press  $\text{F1} + \text{F2}$  together to open the Table screen.
2. Press  $\text{F3}$  to open the **Edit** menu.
3. In the **Edit** menu, use the arrow keys to highlight **New Data Column** and press  $\checkmark$ .

*Result:* An empty editable data set is created and added to the table. A box surrounding the first cell indicates that data can be entered into it.

4. Type a number or text and press  $\checkmark$ .

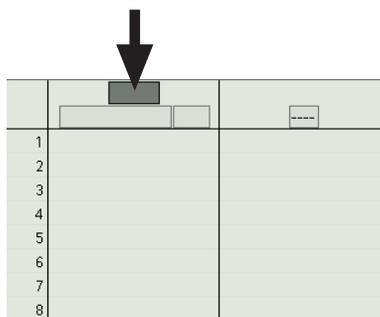
*Result:* The data you entered appears in the table, and the box moves down to the next cell.

5. Repeat step 4 to enter data into other cells of the table.

To skip cells or move back to a previous cell, press  $\text{Esc}$ , use the arrow keys to select a cell, and press  $\text{F2}$  (**Edit Cell**).

### 7.2.4 Showing and hiding data runs in a table

1. In the Table screen, press  $\checkmark$  to activate the fields.
2. Use the arrow keys to move the highlight to the run number (or run name) at the top of a column.

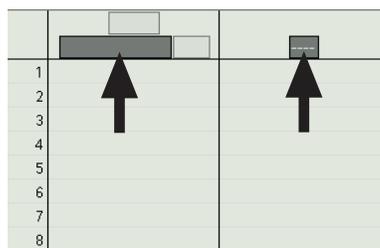


1		
2		
3		
4		
5		
6		
7		
8		

3. Press  $\checkmark$ .  
*Result:* A menu of available runs opens.
4. In the menu, use the arrow keys to highlight the run that you want to show (or highlight **No Data** to hide all runs) and press  $\checkmark$ .

### 7.2.5 Changing the variable displayed in a column

1. In the Table screen, press  $\checkmark$  to activate the fields.
2. Use the arrow keys to move the highlight to the variable name at the top of a column.



1		
2		
3		
4		
5		
6		
7		
8		

If no variable is shown, move the highlight to the series of dashes (---).

3. Press  $\checkmark$ .  
*Result:* A menu of available variables opens.
4. In the menu, use the arrow keys to highlight the desired variable and press  $\checkmark$ .  
If you do not see the desired variable, highlight **More** and press  $\checkmark$ .

## 7.3 Digits display

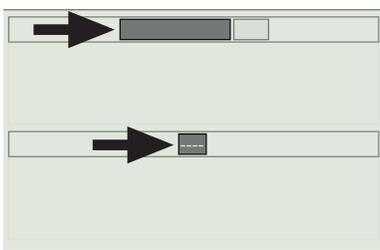
### 7.3.1 Displaying data in a digits display

1. Press  $\leftarrow$  to open the Home screen.
2. Use the arrow keys to highlight the Digits icon and press  $\rightarrow$ .



*Result:* The Digits Display screen opens.

3. Optionally, change the variable displayed:
  - a. Press  $\rightarrow$  to activate the fields in the digits display.
  - b. Use the arrow keys to move the highlight to the variable name.



If no variable is shown, move the highlight to the series of dashes (----).

- c. Press  $\rightarrow$ .

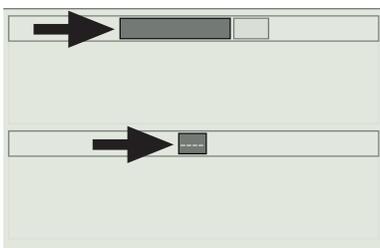
*Result:* A menu of available variables opens.

  - d. In the menu, use the arrow keys to highlight the desired variable and press  $\rightarrow$ .

If you do not see the desired variable, highlight **More** and press  $\rightarrow$ .
4. Optionally, add additional digits displays  $\blacklozenge$ (7.3.2).

### 7.3.2 Adding a measurement to a digits display

1. In the Digits Display screen, press  $F1$ ,  $F2$ ,  $F3$ , or  $F4$  to select the number of digits displays shown on the screen.
2. Optionally, change the variable shown in each digits display:
  - a. Press  $\rightarrow$  to activate the fields.
  - b. Use the arrow keys to move the highlight to the variable name.



If no variable is shown, move the highlight to the series of dashes (----).

- c. Press  $\rightarrow$ .

*Result:* A menu of available variables opens.

- d. In the menu, use the arrow keys to highlight the desired variable and press  $\checkmark$ .

If you do not see the desired variable, highlight **More** and press  $\checkmark$ .

## Section 8: Data run operations

### 8.1 Deleting a data run

1. View the run that you want to delete in a graph or table  $\blacklozenge$ (7.1.1, 7.1.7, 7.2.1, 7.2.4)
2. In the Graph screen or the Table screen, press  $\checkmark$  to activate the fields.
3. Use the arrow keys to move the highlight to the run number (or run name).
4. Press  $\checkmark$ .

*Result:* A menu opens.

5. In the menu, use the arrow keys to highlight **Delete Run** and press  $\checkmark$ .

### 8.2 Naming a data run

1. View the run that you want to name in a graph or table  $\blacklozenge$ (7.1.1, 7.1.7, 7.2.1, 7.2.4)
2. In the Graph screen or the Table screen, press  $\checkmark$  to activate the fields.
3. Use the arrow keys to move the highlight to the run number (or run name).
4. Press  $\checkmark$ .

*Result:* A menu opens.

5. In the menu, use the arrow keys to highlight **Rename Run** and press  $\checkmark$ .

*Result:* A text box appears.

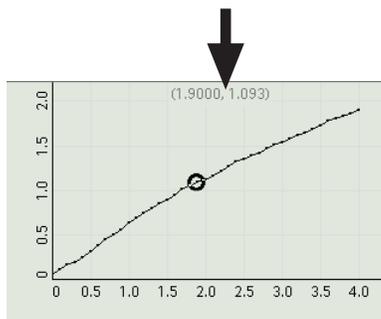
6. Type the new name of the run and press  $F1$  (OK).

## Section 9: Analyzing data

### 9.1 Finding the coordinates of a point in a graph

- $\blacklozenge$  In the Graph screen, use the arrow keys to move the cursor to a data point.

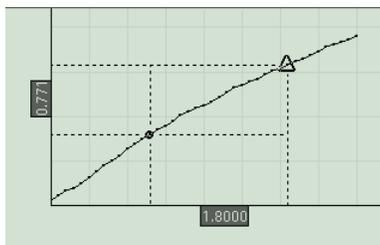
*Task result:* The coordinates of the point are displayed near the top of the graph.



### 9.2 Measuring the difference between two points in a graph

1. In the Graph screen, press  $F3$  to open the **Tools** menu.

- In the menu, use the arrow keys to highlight **Delta Tool** and press  $\checkmark$ .  
*Result:* The Delta Tool appears on the graph.
- Use the arrow keys to move one corner of the Delta Tool to a point on the graph.
- Press  $\text{Esc} + \text{X}$  together.  
*Result:* The control cursor is swapped to the opposite corner of the Delta Tool.
- Use the arrow keys to move to the other corner of the Delta Tool.

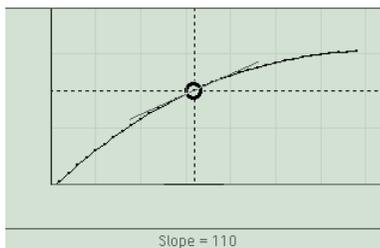


*Task result:* The x- and y-differences between the corners of the Delta Tool are displayed on the graph.

### 9.3

#### Finding the slope at a point on a data plot

- In the Graph screen, press  $F3$  to open the **Tools** menu.
- In the menu, use the arrow keys to highlight **Slope Tool** and press  $\checkmark$ .  
*Result:* The Slope Tool appears on the graph.
- Use the arrow keys to move Slope Tool to a point on the graph.



*Task result:* The the slope of the data plot at the location of the Slope Tool is displayed below the graph.

### 9.4

#### Viewing statistics of data

- In the Graph screen, press  $F3$  to open the **Tools** menu.
- In the menu, use the arrow keys to highlight **Statistics** and press  $\checkmark$ .  
*Result:* The minimum, maximum, average, and standard deviation are displayed below the graph.  
A dashed box indicates the data points included in the statistics.
- Optionally, adjust the box to select a part of the data run to be included in the statistics.  $\blacklozenge(7.1.4)$

**9.5** Applying a curve fit

The curve fit available on the GLX is a linear fit.

1. In the Graph screen, press **F3** to open the **Tools** menu.
2. In the menu, use the arrow keys to highlight **Linear Fit** and press **✓**.

*Result:* The best-fit line is displayed on the graph. The slope, and y-intercept are displayed below the graph.

A dashed box indicates the data points included in the linear fit.

3. Optionally, adjust the box to select a part of the data run to be included in the linear fit. ◆(7.1.4)

---

**9.6** Finding the slope and intercept of a best-fit line

1. In the Graph screen, press **F3** to open the **Tools** menu.
2. In the menu, use the arrow keys to highlight **Linear Fit** and press **✓**.

*Result:* The best-fit line is displayed on the graph. The slope, and y-intercept are displayed below the graph.

A dashed box indicates the data points included in the linear fit.

3. Optionally, adjust the box to select a part of the data run to be included in the linear fit. ◆(7.1.4)

---

**9.7** Finding the area under a curve

1. In the Graph screen, press **F3** to open the **Tools** menu.
2. In the menu, use the arrow keys to highlight **Area Tool** and press **✓**.

*Result:* The area under the curve is shaded and the value is displayed below the graph.

Two cursors indicate the range of data points included in the area.

3. Optionally, adjust the cursors to select a part of the data run to be included in the area. ◆(7.1.4)

---

**Section 10: Data operations****10.1** Editing manually entered data

1. With the manually entered data displayed in a table, press **Esc**.

*Result:* A dashed box appears around one of the editable cells.

2. Use the arrow keys to move the dashed box to the desired cell.
3. Press **F2** (**Edit Cell**).

*Result:* The dashed box is replaced by a solid-line box.

4. Type new data into the cell and press **✓**.

Repeat this task to edit data in other cells.

**10.2** Deleting data points

1. With the manually entered data displayed in a table, press **Esc**.  
*Result:* A dashed box appears around one of the editable cells.
  2. Use the arrow keys to move the dashed box to the desired cell.
  3. Press **F2** (**Edit Cell**).  
*Result:* The dashed box is replaced by a solid-line box.
  4. Press **X** to delete the data in the cell.
- Repeat this task to delete data in other cells.

**10.3** Creating calculated data

1. Press **Home** + **F3** together to open the Calculator screen.
  2. If there are previous calculations in the Calculator screen, use the arrow keys to move the cursor to the first blank line.
  3. Enter an expression.  
If you have been given an expression in your lab instructions, enter it into the calculator exactly as it appears in the instructions. Entering the expression exactly as given will allow the GLX to automatically associate variables in the expression with sensor measurements.
  4. Press **Enter**.  
*Result:* The calculation is complete.
  5. Press **Home** to return to the Home screen.
- Task result:* The calculation is now available for viewing in a graph, table, or other display.

**Section 11: Saving and sharing your experiment****11.1** Saving your experiment

1. Press **Home** to open the Home screen.
2. Use the arrow keys to highlight the **Data Files** icon and press **Enter**.



*Result:* The Data Files screen opens.

3. Press **F4** to open the **Files** menu, use the arrow keys to highlight **Save As** and press **Enter**.
4. Type a filename and press **Enter**.
5. Press **Home** to return to the Home screen.

**11.2** Printing

1. Connect a compatible printer to the larger USB port on the right side of the GLX.  
Use the USB cable that came with the printer.

2. Display the data that you would like to print in a graph <sup>◆(7.1.1)</sup> or table <sup>◆(7.2.1)</sup>.
3. Press **F4**.  
*Result:* A menu opens.
4. In the menu, use the arrow keys to highlight **Print** and press **✓**.

---

### 11.3 Exporting data for use in mapping software

1. Connect a USB flash drive to the GLX.
  2. Press **⇧** + **F2** together to open the Table screen.
  3. Press **F4** to open the **Tables** menu, use the arrow keys to highlight **Show Time** and press **✓**.  
*Result:* Time is added in a column on the left side of the table.
  4. Press **F4** to open the **Tables** menu again, use the arrow keys to highlight **Date/Time** and press **✓**.  
*Result:* The first column now displays the calendar date and time of day.
  5. Press **F4** to open the **Tables** menu again, use the arrow keys to highlight **Export all Data** and press **✓**.  
*Result:* The Export All Data screen opens.
  6. In the Export All Data screen, with **Export File Name** highlighted, press **✓**, type a filename, and press **✓** again.
  7. Press **F4** (**Add All**).  
*Result:* All measurements are added to the list of data to be exported.
  8. Press **F1** (**OK**).  
*Result:* Data is saved to the USB flash drive.
  9. Unplug the USB flash drive from the GLX.
- Connect the USB flash drive to a computer to open the data in mapping software.

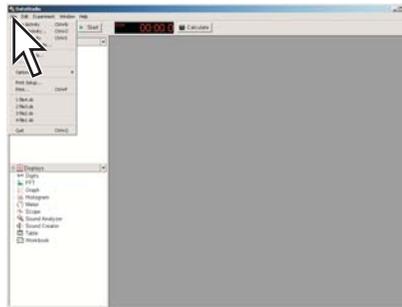
# Appendix D: DataStudio Tech Tips

## Section 1: Starting an experiment

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### 1.1 Opening a file

1. Open the **File** menu and select **Open Activity**.

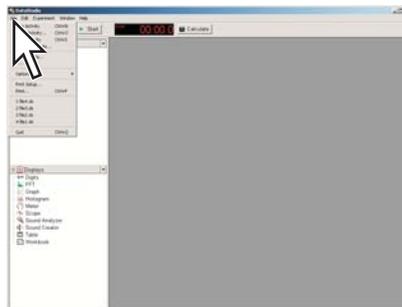


2. In the **Open** dialog box, navigate to the folder that contains the desired file and select the file.
3. Click **Open**.

---

### 1.2 Starting a new experiment

1. Open the **File** menu and select **New Activity**.



2. If you see the Welcome to DataStudio window, select **Create Experiment**.

## Section 2: Setting up measurements and data collection

---

### 2.1 Adding a sensor to your DataStudio experiment

1. If it is not already connected, connect a PASPORT interface (such as a USB Link, PowerLink, or Xplorer GLX) to your computer.

Some interfaces also need to be connected to AC power or turned on.

2. Connect the sensor to the PASPORT interface.

You can plug the sensor directly into the interface or use an extension cable between the sensor and interface.

*Task result:* DataStudio detects the sensor and adds it to your experiment.

## 2.2 Adding multiple sensors to your DataStudio experiment

Depending on which equipment you have, do one of the following:

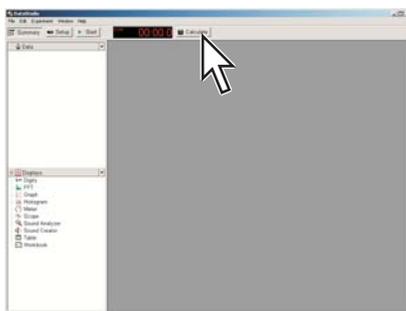
- ◆ If you have a PASPORT interface with multiple ports (such as Xplorer GLX or Power Link), complete these steps:
  - a. If necessary, connect the interface to your computer.
  - b. If necessary, connect the interface to AC power and turn it on.
  - c. Connect all of the sensors to the interface.
- ◆ If you have more than one PASPORT interface, and your computer has a USB port available for each interface, complete these steps:
  - a. Connect each PASPORT interface to a different USB port on your computer.
  - b. Connect a sensor to each PASPORT interface.
- ◆ If you have more than one PASPORT interface, and a USB hub, complete these steps:
  - a. Connect a USB hub to the USB port of your computer.
  - b. Connect the USB hub to AC power.
  - c. Connect all of the PASPORT interfaces to the USB hub.
  - d. Connect a sensor to each PASPORT interface.

*Task result:* DataStudio detects the sensors and adds them to your experiment.

## 2.3 Programming DataStudio to calculate lung volume

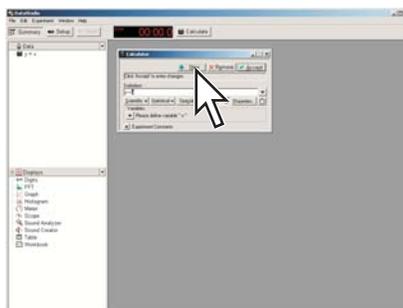
After connecting a spirometer to your DataStudio experiment <sup>(2.1)</sup>, complete these steps:

1. Click **Calculate**.



*Result:* The Calculator window opens.

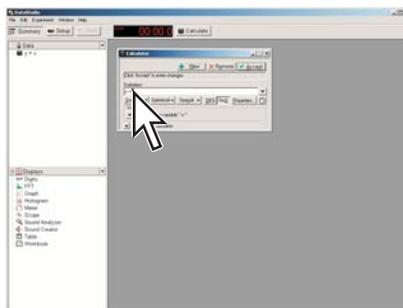
- If a previous calculation that you do not wish to overwrite is shown, click **New**.



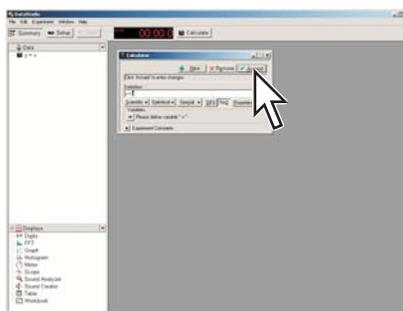
*Result:* A new calculation is started.

- In the definition box, type this expression:  

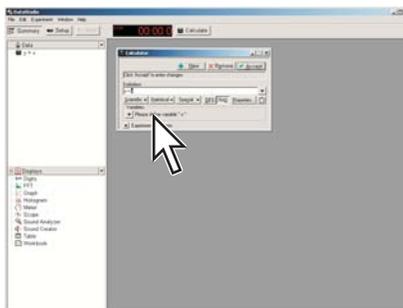
$$\text{Lung Volume} = V_0 - [\text{Total Flow (liters)}]$$



- Click **Accept**.



- Click the button labeled **Please define variable "V0"** and select **Constant**.



- In the box labeled **V0 =**, enter the test subject's functional residual capacity (FRC) in liters.  
 If you do not know the test subject's FRC, enter 2.5.

7. Click **Accept** again.
8. Optionally, close the Calculator window.

*Task result:* The Lung Volume calculation is now available for viewing in a graph, table, or other display.

## Section 3: Calibrating sensors

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### 3.1 Calibrating a carbon dioxide gas sensor

Once the Carbon Dioxide Gas sensor has been calibrated using this method, it retains its calibration, even after it has been unplugged.

1. Take the sensor and an empty sample bottle outdoors.  
If necessary, unplug the sensor from the interface.
2. Fill the sample bottle with fresh outdoor air.
3. Insert the probe of the sensor into the bottle and push the rubber stopper of the sensor into the neck of the bottle.
4. If you have disconnected the sensor, return to the interface and connect the sensor to it.
5. Press the **Calibrate** button on the sensor and hold it for three seconds.

*Result:* The green light illuminates on indicating that calibration is in progress.

6. Wait approximately one minute.  
When the green light flashes slowly, calibration is finished. If the light flashes rapidly (five times per second), an error has occurred and the sensor has not been calibrated.

---

### 3.2 Calibrating a colorimeter

1. Place a cuvette filled with distilled water in the colorimeter and close the lid.
2. Press the **Calibration** button on the sensor.

*Result:* The light in the button illuminates indicating that calibration is in progress.

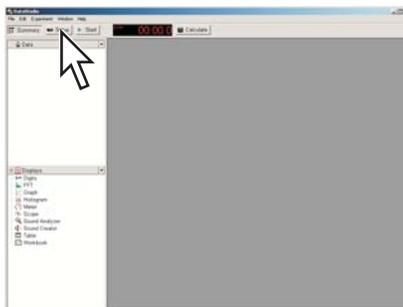
3. Wait for the light to turn off.

---

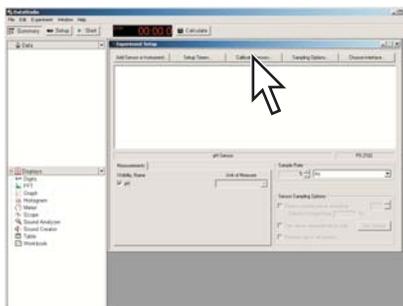
### 3.3 Calibrating a dissolved oxygen sensor

1. Obtain barometric pressure and temperature at your location.
2. Refer to the solubility table included with the sensor and find the standard dissolved oxygen value for the temperature and barometric pressure at your location.

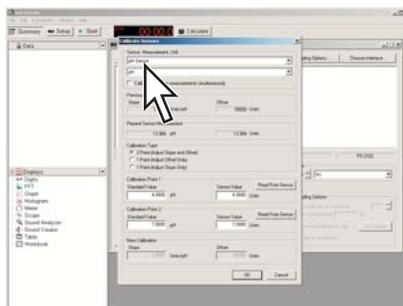
3. Click **Setup**.



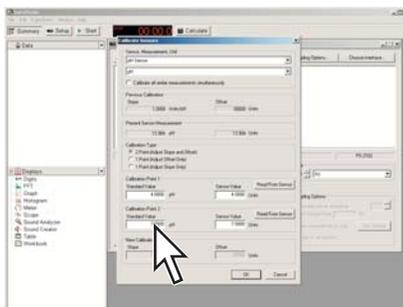
4. Click **Calibrate Sensors**.



5. In the **Calibrate Sensors** dialog box under **Sensor**, **Measurement**, **Unit** select **Dissolved Oxygen Sensor** and **Dissolved Oxygen (mg/L)**.

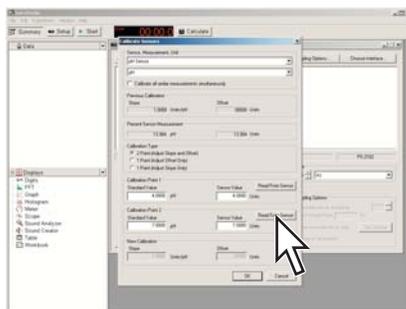


6. Under **Calibration Point 2** in the **Standard Value** box, type the value that you determined from the solubility table.



7. Place about 5 mL (to a height of about 1 cm) of deionized water into a clean soaker bottle. Slip the cap and O-ring of the soaker bottle over the end of the probe.
8. Insert the probe into the soaker bottle and screw on the lid. Adjust the height of the probe to about 2 cm above the top of the water.

9. Shake the soaker bottle vigorously for about 10 seconds. Shake off any large water drops from the membrane at the end of the probe.
10. Under **Calibration Point 2**, click **Read From Sensor**.



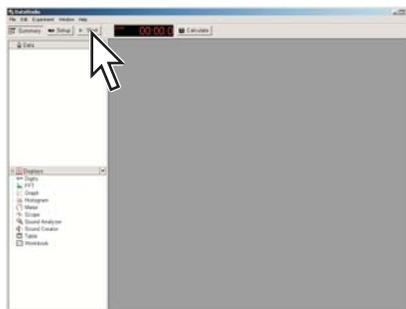
11. Click **OK**.

### 3.4 Calibrating a drop counter

The drop counter is calibrated *after* data is taken, using DataStudio's calculator to correlate the number of drops counted to the volume of liquid dispensed from the burette. The following steps illustrate the calibration procedure. Note that in performing the experiment, these steps will be interspersed with other experiment procedure steps.

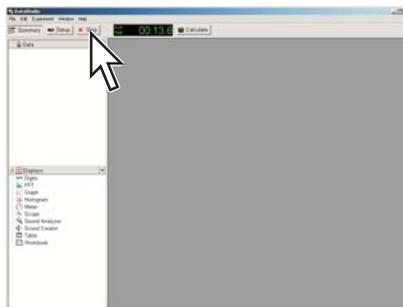
Note: Using this method, you will ignore the "Volume" measurement output directly from the sensor. Instead, volume is calculated by DataStudio. This method takes into account the fact that drop size may be different each time the burette is used. By measuring the total volume dispensed during the actual experiment, a more accurate correlation between volume and drop count is obtained.

1. Set up the drop counter on a stand with a liquid-filled burette, a beaker, stir-plate, and other equipment.  
See your lab instructions for details.
2. Write down the initial volume of liquid in the burette.
3. Click **Start**.

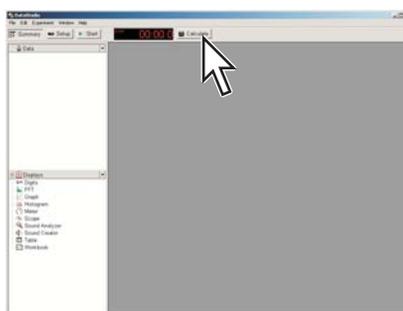


4. Slowly turn the stopcock to start delivering liquid at about 2 drops per second.
5. After the necessary amount of liquid has been dispensed from the burette, close the stopcock.

6. Click **Stop**.

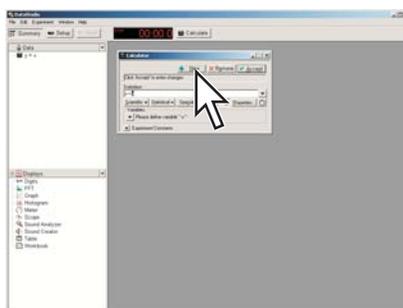


7. Read the final volume of liquid in the burette and determine the net volume that was dispensed.
8. Read the final drop count (the total number of drops dispensed) in a graph or table display.
9. Click **Calculate**.



*Result:* The Calculator window opens.

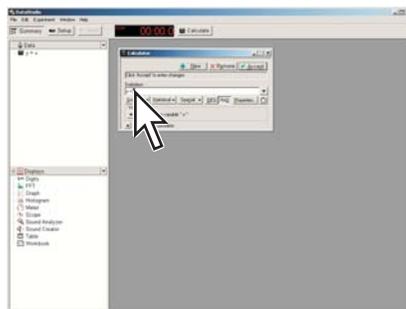
10. If a previous calculation that you do not wish to overwrite is shown, click **New**.



*Result:* A new calculation is started.

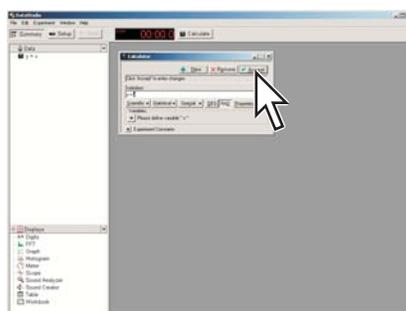
11. In the definition box, type an expression similar to this:  

$$\text{volume} = [\text{Drop Count (drops)}] * 100/3050$$



In this example, “100” is the total volume dispensed (in mL) and “3050” is the final drop count. When you type the expression, use your own values for these two quantities.

12. Click **Accept**.



13. If desired, close the Calculator window.

*Task result:* The calibrated Volume calculation is now available for viewing in a graph, table, or other display.

### 3.5 Calibrating an oxygen gas sensor

Once the Oxygen Gas sensor has been calibrated using this method, it retains its calibration, even after it has been unplugged.

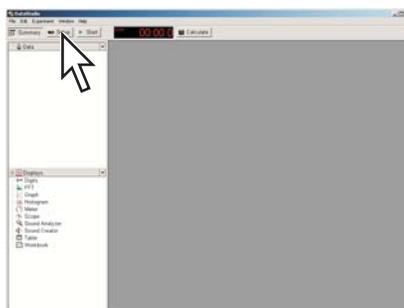
1. Take the sensor and an empty sample bottle outdoors.  
 If necessary, unplug the sensor from the interface.
2. Fill the sample bottle with fresh outdoor air.
3. Insert the probe of the sensor into the bottle and push the rubber stopper of the sensor into the neck of the bottle.
4. If you have disconnected the sensor, return to the interface and connect the sensor to it.
5. Press the **CAL (20.9%)** button on the sensor and hold it for three seconds.

*Task result:* The green light flashes on and off for 4 seconds, indicating that calibration is in progress. When the light stops flashing, calibration is finished. If the light flashes rapidly (5 times per second), an error has occurred and the sensor has not been calibrated.

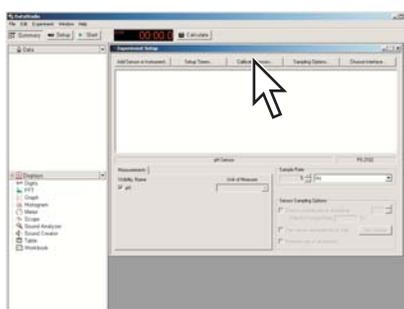
### 3.6 Calibrating a pH sensor

You will need buffer solutions of pH 4 and pH 10 and deionized water.

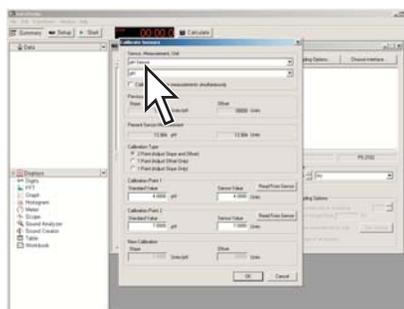
1. Click **Setup**.



2. Click **Calibrate Sensors**.



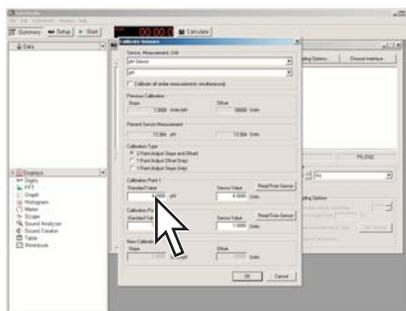
3. In the **Calibrate Sensors** dialog box under **Sensor, Measurement, Unit** select **pH Sensor** (or other sensor with a pH probe) and **pH**.



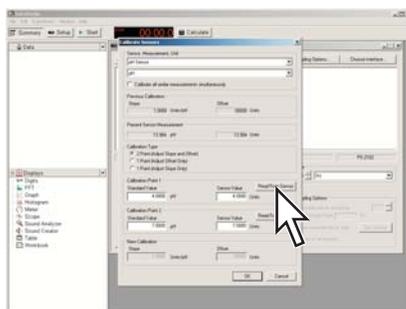
4. Place the pH probe into the pH 4 buffer solution and wait for the reading to stabilize.

The reading is indicated under **Present Sensor Measurement**.

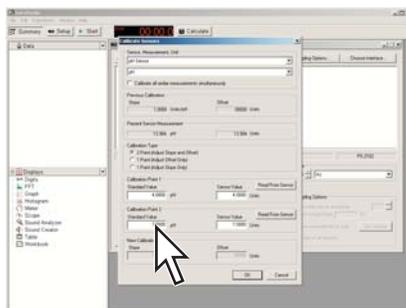
5. Enter 4 (the known pH of the buffer solution) into the **Standard Value** box for **Calibration Point 1**.



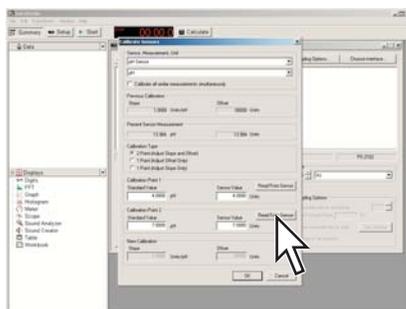
6. Under **Calibration Point 1**, click **Read From Sensor**.



7. Rinse the probe with deionized water.
8. Place the pH probe into the pH 10 buffer solution and wait for the reading to stabilize.
9. Enter 10 (the known pH of the buffer solution) into the **Standard Value** box for **Calibration Point 2**.



10. Under **Calibration Point 2**, click **Read From Sensor**.



11. Click **OK**.

**3.7** Calibrating a turbidity sensor

1. Place a cuvette filled with distilled water in the turbidity sensor and close the lid.
2. Press the **Calibration** button on the sensor.

*Result:* The green light in the button illuminates.

3. When the light starts blinking, replace the cuvette with the standard 100 NTU cuvette (included with the sensor) and close the lid.
4. Press the button again.

*Result:* The light in the button illuminates.

*Task result:* When the light turns off, calibration is complete.

**3.8** Calibrating an ethanol sensor

You will need a solution of 1% ethanol in water. The solution should be at the same temperature as the solutions to be measured.

Once the sensor has been calibrated using this method, it retains its calibration, even after it has been unplugged.

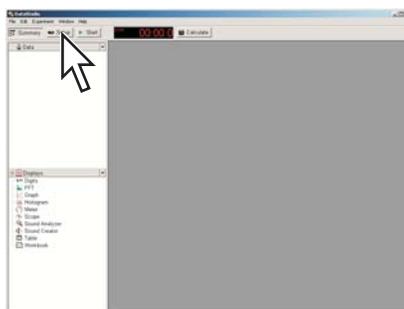
1. Connect the sensor to the interface and wait about 10 minutes for the probe to warm up.
2. Place the probe about 1 cm above the 1% ethanol solution.
3. Observe the ethanol concentration reading in DataStudio and wait until the reading stabilizes.
4. Press and hold the **1% CAL** button on the sensor for 4 seconds.

*Task result:* Immediately after a successful calibration, the sensor's output reads 1%, and the button is illuminated.

**Section 4: Sensor operations****4.1** Using a colorimeter with a specific color of light

The colorimeter makes simultaneous measurements using four different colors. Complete these steps to make measurements from only one color visible.

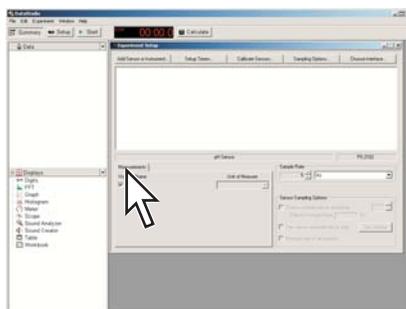
1. Click **Setup**.



*Result:* The Experiment Setup window opens.

- In the Experiment Setup window, click the icon that represents the colorimeter.

*Result:* The complete list of measurements available from the colorimeter appears under the **Measurement** tabs. Note that some of the measurements appear under a second tab.



- Select the check boxes next to the measurements that you want to use in your experiment. Clear the check boxes next to measurements that you do not want to use.

For each color there is a transmittance measurement and an absorbance measurement. Typically, you will select the absorbance or the transmittance measurement (or both) for a single color, and clear the selection of all other measurements.

## 4.2 Setting up a conductivity sensor for a particular sensitivity

When using a conductivity sensor, it is important to select a sensitivity appropriate to the solutions that you plan to test. If the selected sensitivity is too high, the sensor may lack the range necessary to measure your most concentrated solutions. However, if the range is larger than necessary, the measurement precision may not be high enough to detect small conductivity changes or differences.

Complete these steps to select the appropriate sensitivity and range:

- Determine or estimate the highest concentration that you will need to measure.

Use this chart and other resources.

Solution	Conductivity ( $\mu\text{S}/\text{cm}$ )
Ultrapure water	0.05 to 0.75
Drinking water	50 to 1500
Ocean water	~53000

- Select the range that best matches your measurement requirements.

The first column of this chart shows available ranges.

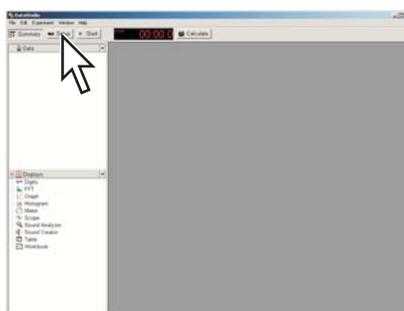
Measurement Range ( $\mu\text{S}/\text{cm}$ )	1x Probe Range Selection	10x Probe Range Selection
0 to 100		-
0 to 1000		
0 to 10000		
0 to 100000	-	

- Select an appropriate probe (either 1x or 10x) and connect it to the sensor.
- Use the buttons on the sensor to select an appropriate range.  
Lights in the buttons indicate which range is selected.
- In DataStudio, make the appropriate measurement visible:
  - Click **Setup**.
  - In the Experiment Setup window, click the icon of the conductivity sensor (or other sensor with a conductivity probe).
  - In the **Measurements** tab, select the conductivity measurement that matches the probe connected to the sensor (either 1x or 10x).
  - Clear the selection of the conductivity measurement that does not match the probe connected to the sensor.

### 4.3

Setting up a rotary motion sensor to measure linear position using the "rack" setting

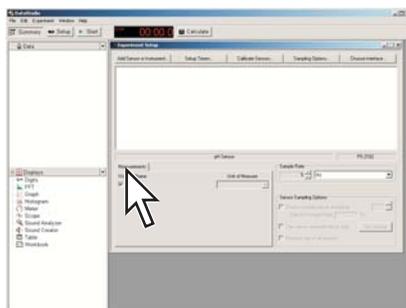
- In the main toolbar, click the **Setup** button.



*Result:* The Experiment Setup window opens.

- If there are multiple sensors, connected, click the icon of the rotary motion sensor in the Experiment Setup window.

3. In the Experiment Setup window under the **Measurements** tab, select the **Linear Position** check box.

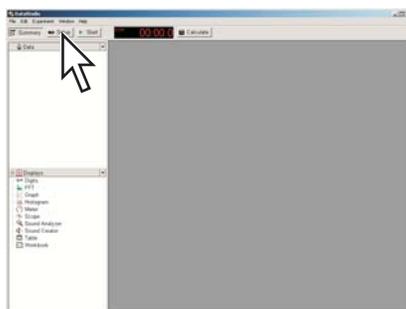


4. Under the **Measurements** tab, clear all other check boxes.
5. Click the **Rotary Motion Sensor** tab.
6. Under **Linear Scale**, select **Rack & Pinion**.
7. Close the Experiment Setup window.

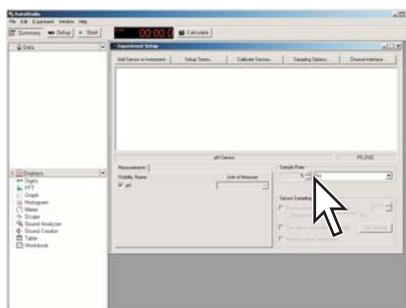
## Section 5: Data measurement setup

### 5.1 Changing the sampling rate

1. Click **Setup**.



2. In the Experiment Setup window, click the icon of the sensor that you wish to change.
3. Under **Sample Rate**, select the units and select the value.

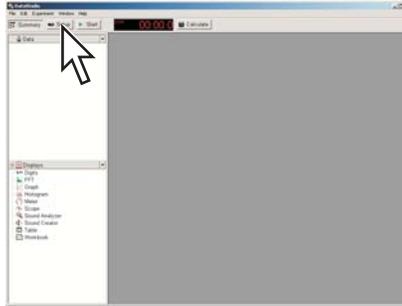


If you select units of Hz, the value indicates the number of samples per second. If you select units of seconds, minutes, or hours, the value indicates the time between samples.

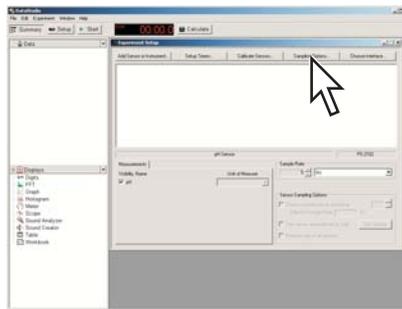
Repeat this task for all other sensors.

## 5.2 Putting DataStudio into manual sampling mode

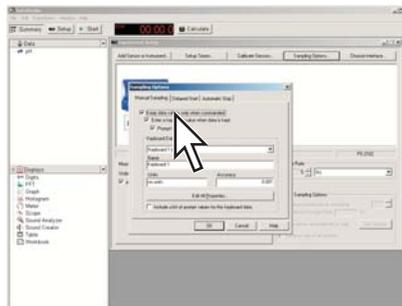
1. Click **Setup**.



2. In the Experiment Setup window, click **Sampling Options**.



3. Select **Keep data values only when commanded**.

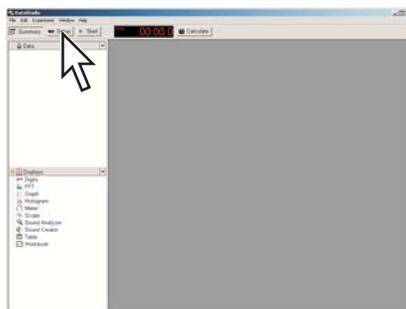


This puts DataStudio into manual sampling mode.

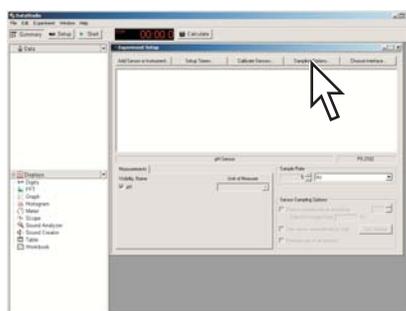
4. For more instructions see Tech Tip 5.2.1 (sampling with manually entered data) and Tech Tip 5.2.2 (sampling without manually entered data).
5. Click **OK**.

### 5.2.1 Putting DataStudio into manual sampling mode with manually entered data

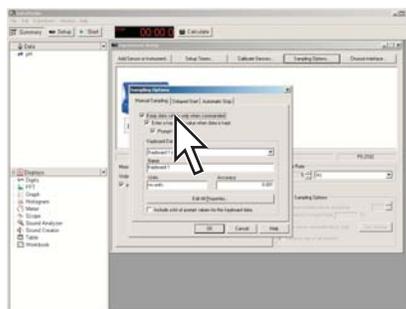
1. Click **Setup**.



2. In the Experiment Setup window, click **Sampling Options**.

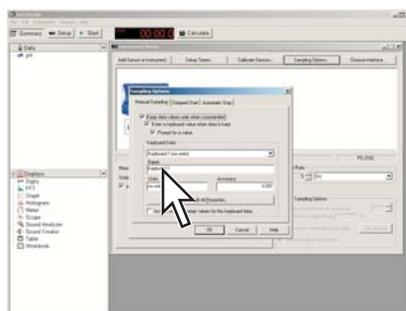


3. Select **Keep data values only when commanded**.

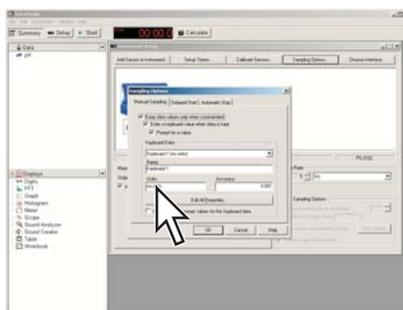


This puts DataStudio into manual sampling mode.

4. In the **Name** box, type the name of the data that you will be manually entering.



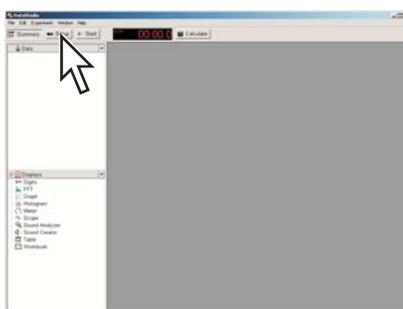
- In the **Units** box, type the units of the data that you will be manually entering.



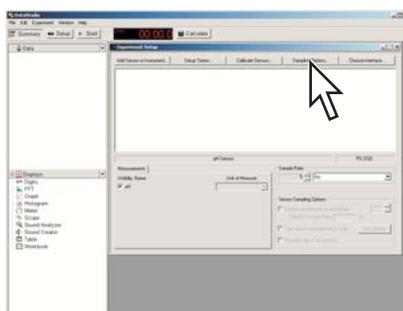
- Click **OK**.
- Create a table to display your manually entered data.<sup>(7.2.1)</sup>
- Add a column to the table to display the sensor measurement that will be recorded alongside the manually entered data.<sup>(7.2.2)</sup>

### 5.2.2 Putting DataStudio into manual sampling mode without manually entered data

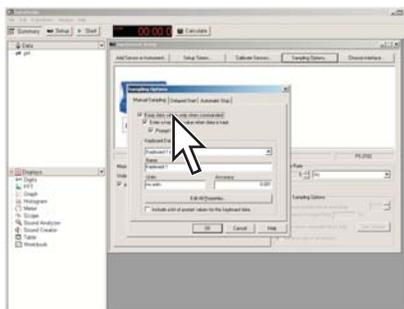
- Click **Setup**.



- In the Experiment Setup window, click **Sampling Options**.

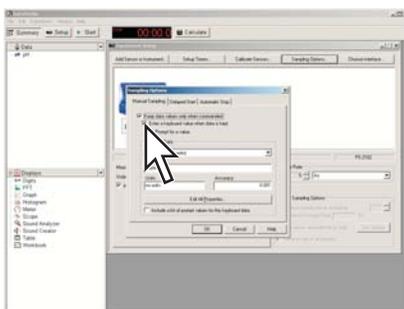


3. Select **Keep data values only when commanded**.



This puts DataStudio into manual sampling mode.

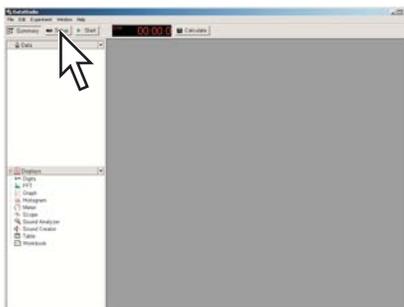
4. Clear the check box labeled **Enter a keyboard value when data is kept**.



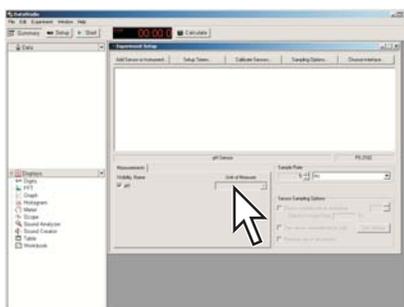
5. Click **OK**.

### 5.3 Changing the units of a measurement

1. Click **Setup**.



2. In the Experiment Setup window, click the icon of the sensor that makes the measurement.
3. In the **Unit of measure** box of the measurement, select the units you want.

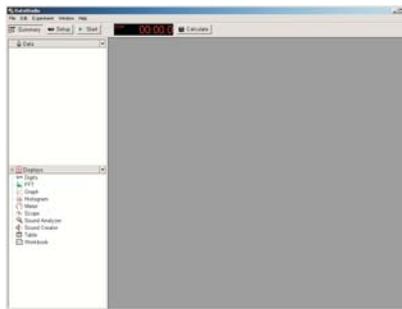


## 5.4 Changing the number of digits with which a variable is displayed

1. If the summary bar is not open, click **Summary**.



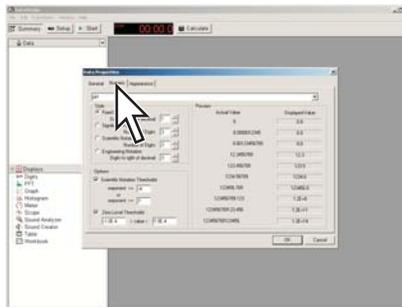
*Result:* The summary bar appears.



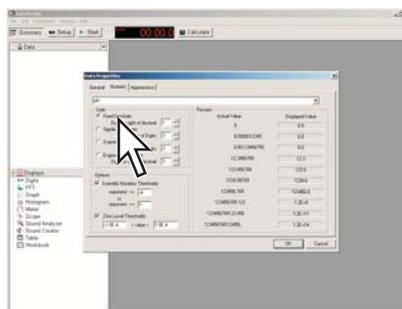
2. In the summary bar, double-click the variable or measurement.

*Result:* The Data Properties window opens.

3. In the Data Properties window, click the **Numeric** tab.



4. Click **Fixed Decimals**.

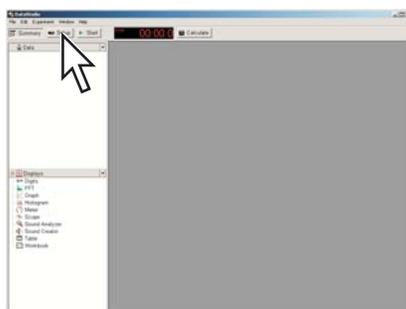


5. In the **Digits to the right of the decimal** box, enter a number.
6. Click **OK**.

## 5.5 Setting up digital timers

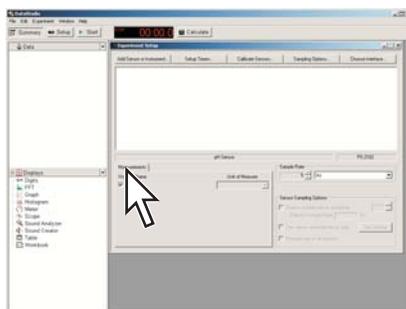
### 5.5.1 Setting up a photogate to measure velocity

1. If it is not already connected, connect a PASPORT interface to your computer.  
(PASPORT interfaces include the Xplorer, Xplorer GLX, PowerLink, and USB Link.)
2. Connect a Digital Adapter to the PASPORT interface.
3. Connect a photogate to the Digital Adapter.  
*Result:* The **Choose sensor or instrument** window appears.
4. Click **Photogate Timing** and click **OK**.  
*Result:* A table display is created automatically.
5. In the main toolbar, click the **Setup** button.



*Result:* The Experiment Setup window opens.

6. In the Experiment Setup window under the **Measurements** tab, confirm that the **Velocity In Gate** check box is selected.



7. Under the **Measurements** tab, clear all other check boxes.
8. Click the **Constants** tab.
9. In the **Flag Length** box, type the length (in meters) of the flag that will pass through the photogate.
10. Close the Experiment Setup window.
11. Find the table display that was automatically created earlier.
12. Click the column heading of the second column in the table and select **Velocity In Gate**.

*Task result:* DataStudio is now set up to measure and display the magnitude of velocity of an object passing through the photogate.

## 5.5.2

Setting up a photogate to measure the period of a pendulum

1. If it is not already connected, connect a PASPORT interface to your computer.

(PASPORT interfaces include the Xplorer, Xplorer GLX, PowerLink, and USB Link.)

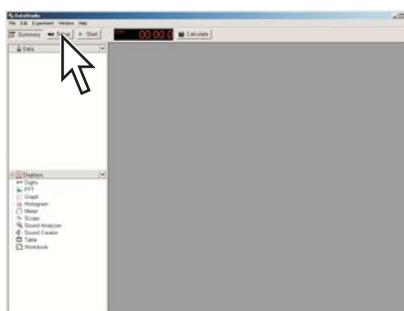
2. Connect a Digital Adapter to the PASPORT interface.
3. Connect a photogate to the Digital Adapter.

*Result:* The **Choose sensor or instrument** window appears.

4. Click **Photogate and Pendulum** and click **OK**.

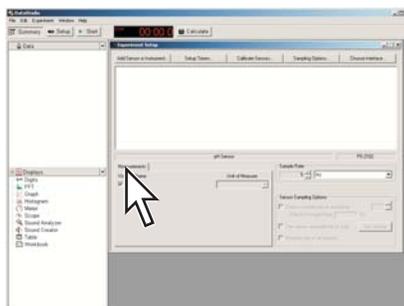
*Result:* A table display is created automatically.

5. In the main toolbar, click the **Setup** button.



*Result:* The Experiment Setup window opens.

6. In the Experiment Setup window under the **Measurements** tab, confirm that the **Period** check box is selected.

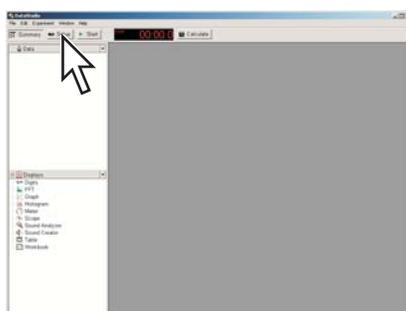


7. Under the **Measurements** tab, clear the **Velocity** check box.
8. Close the Experiment Setup window.
9. Find the table display that was automatically created earlier.
10. Click the column heading of the second column in the table (labeled **Velocity**) and press the Delete key to remove the column.

*Task result:* DataStudio is now set up to measure and display the period of a pendulum passing through the photogate.

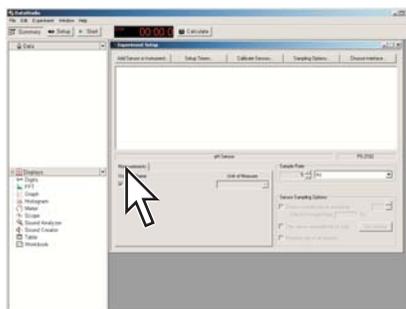
### 5.5.3 Setting up a Free Fall Adapter to measure time of fall

1. If it is not already connected, connect a PASPORT interface to your computer.  
(PASPORT interfaces include the Xplorer, Xplorer GLX, PowerLink, and USB Link.)
2. Connect a Digital Adapter to the PASPORT interface.
3. Connect a Free Fall Adapter to the Digital Adapter.  
*Result:* The **Choose sensor or instrument** window appears.
4. Click **Free Fall Adapter** and click **OK**.  
*Result:* A table display is created automatically.
5. In the main toolbar, click the **Setup** button.



*Result:* The Experiment Setup window opens.

6. In the Experiment Setup window under the **Measurements** tab, confirm that the **Time of Fall** check box is selected.



7. Under the **Measurements** tab, clear the **Acceleration** check box.
8. Close the Experiment Setup window.
9. Find the table display that was automatically created earlier.
10. Click the column heading of the second column in the table (labeled **Acceleration**) and press the Delete key to remove the column.

*Task result:* DataStudio is now set up to measure and display the time for an object to fall from the clip to the pad of the Free Fall Adapter.

### 5.5.4 Setting up photogates to measure time between gates

1. If it is not already connected, connect a PASPORT interface to your computer.

(PASPORT interfaces include the Xplorer, Xplorer GLX, PowerLink, and USB Link.)

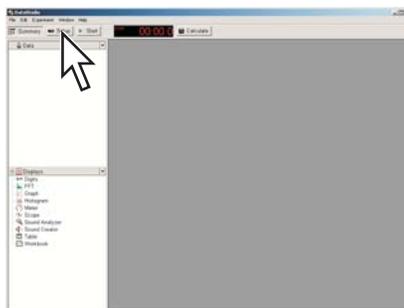
2. Connect a Digital Adapter to the PASPORT interface.
3. Connect a photogate to the Digital Adapter.

*Result:* The **Choose sensor or instrument** window appears.

4. Connect a second photogate to the Digital Adapter.
5. Click **Photogate Timing** and click **OK**.

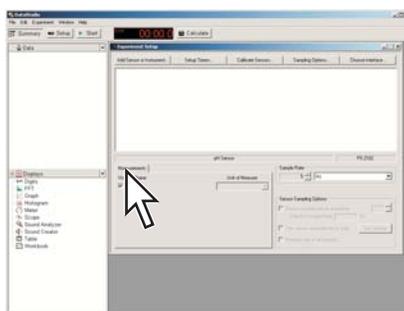
*Result:* A table display is created automatically.

6. In the main toolbar, click the **Setup** button.



*Result:* The Experiment Setup window opens.

7. In the Experiment Setup window under the **Measurements** tab, confirm that the **Time Between Gates** check box is selected.



8. Under the **Measurements** tab, clear the **Time in Gate** check box.
9. Close the Experiment Setup window.
10. Find the table display that was automatically created earlier.
11. Click the column heading of the second column in the table (labeled **Time in Gate**) and press the Delete key to remove the column.

*Task result:* DataStudio is now set up to measure and display the time for an object to pass from one photogate to the other.

## Section 6: Collecting and working with data

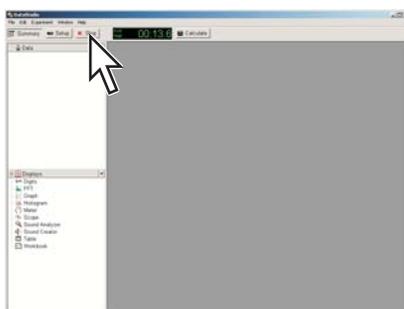
### 6.1 Monitoring live data

1. Create a digits display to show the measurement (or measurements) that you wish to monitor. ♦(7.3.1, 7.3.2)
2. In the **Experiment** menu, click **Monitor Data**.



*Result:* Data Studio starts displaying live data.

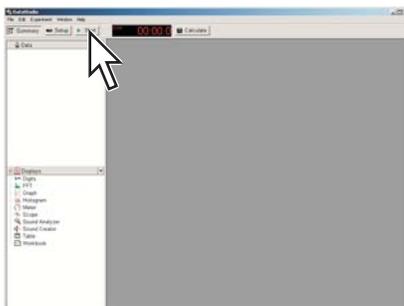
3. To stop monitoring, click **Stop**.



### 6.2 Recording a run of data

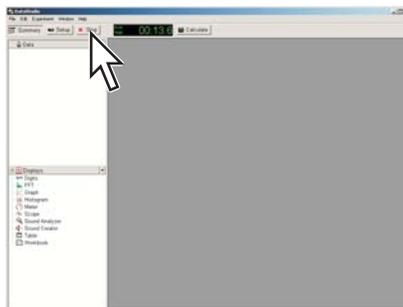
Complete these steps to record a data run in normal sampling mode:

1. Click **Start** to start data recording.



The **Start** button is replaced by the **Stop** button.

2. Click **Stop** to stop data recording.

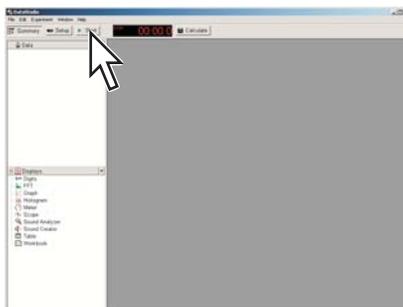


Repeat the steps above to collect another data run.

### 6.3 Recording a set of manually sampled data

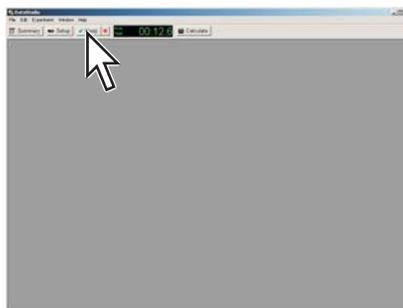
If DataStudio is in manual sampling mode <sup>(5.2)</sup>, complete these steps to record a data set:

1. Click **Start**.



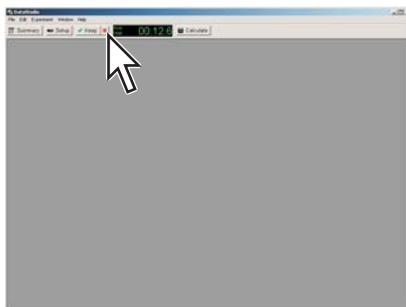
*Result:* A new data set is started and the **Start** button is replaced by the **Keep** button.

2. When you are ready to record a point, click **Keep**.



3. If DataStudio prompts you to enter keyboard data, enter a number.  
Note: You must enter a number, not text.
4. Each time you wish to record another data point, click **Keep**.

5. When you have recorded the *entire* data set, click the **Stop** button.



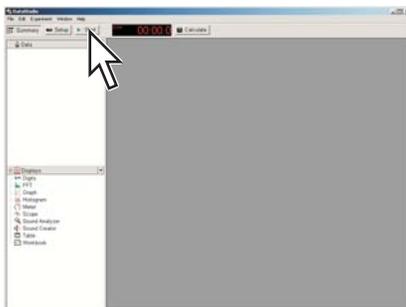
*Result:* The data set is stopped.

Note: If you accidentally stop data collection early (by clicking the **Stop** button instead of **Keep**), you will need to start over again from the beginning. If desired, delete the incomplete data set before starting again. ♦(8.1)

---

### 6.3.1 Starting a manually sampled data set

- ♦ Click **Start**.

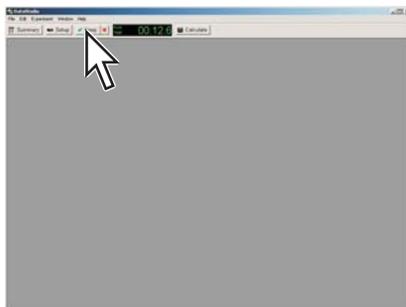


*Result:* A new data set is started and the **Start** button is replaced by the **Keep** button.

---

### 6.3.2 Recording a manually sampled data point

1. When you are ready to record a point, click **Keep**.



2. If DataStudio prompts you to enter keyboard data, enter a number.

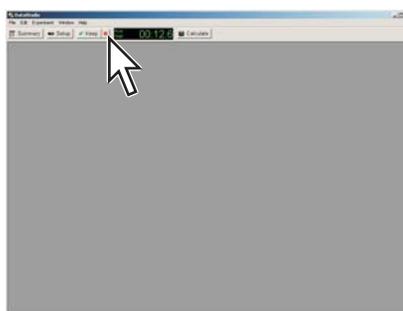
Note: You must enter a number, not text. You may need to assign a number to identify each sample and enter the number instead of the name of the sample.

Note: If you accidentally stop data collection early (by clicking the **Stop** button instead of **Keep**), you will need to start over again from the beginning. If desired, delete the incomplete data set before starting again. <sup>(8.1)</sup>

---

### 6.3.3 Stopping a manually sampled data set

- ◆ When you have recorded the *entire* data set, click the **Stop** button.



*Result:* The data set is stopped.

## Section 7: Data display

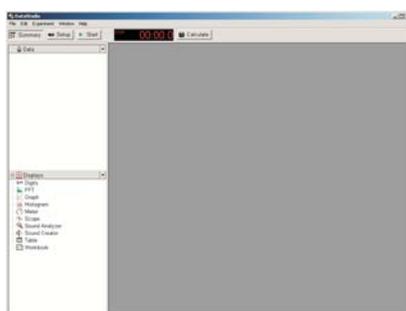
### 7.1 Graph

#### 7.1.1 Displaying data in a graph

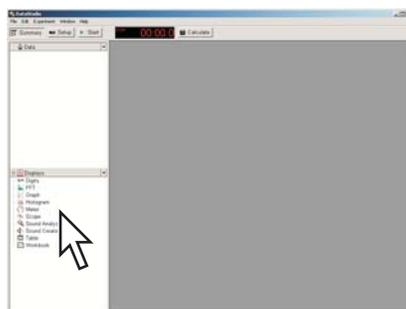
1. If the summary bar is not visible, click **Summary**.



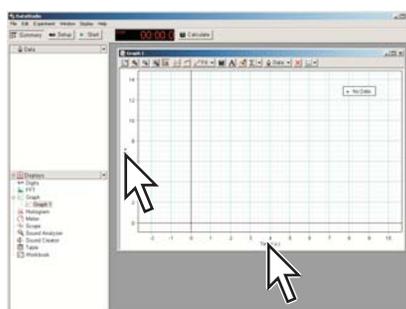
*Result:* The summary bar appears.



2. In the summary bar, double-click **Graph**.



3. If the Please Choose a Data Source window appears, click the measurement that you would like to see on the y-axis; then click **OK**.
4. Optionally, click the axis labels to change the variables on the x- and y-axes.



## 7.1.2 Adjusting the scale of a graph

To adjust the x- and y-scales, or the domain and range of a graph, do one or more of the following:

- ◆ Click the **Scale to fit** button.



*Result:* The graph adjusts to fit all data.

- ◆ Stretch or compress the graph by dragging:

- a. Point to one of the numbers labeling the x- or y-divisions so that the scale cursor appears.



- b. Drag the cursor left or right (on the x-axis) or up or down (on the y-axis).

- ◆ Move the graph by dragging:

- a. Point to the x- or y-axis (or point the bottom or left edge of the graph if the axes are not visible) so that the move cursor appears.



- b. Drag the graph left, right, up, or down.

- ◆ Zoom in on a selected region:

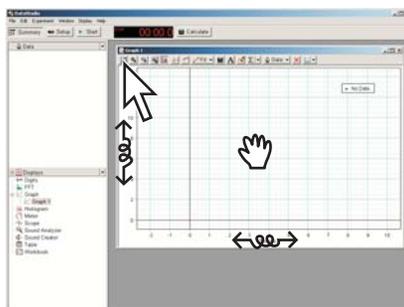
- a. Drag the cursor diagonally on the graph to draw a rectangular outline selecting the points that you would like to zoom in on.

*Result:* The selected points are highlighted.

- b. Click the **Scale to fit** button.

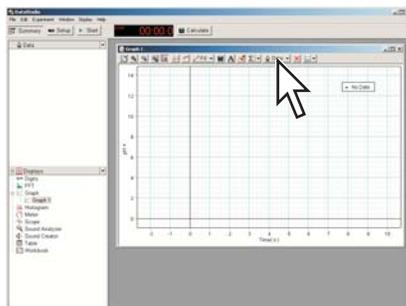


*Result:* The graph adjusts to fit the selected data.



### 7.1.3 Displaying multiple data runs on a graph

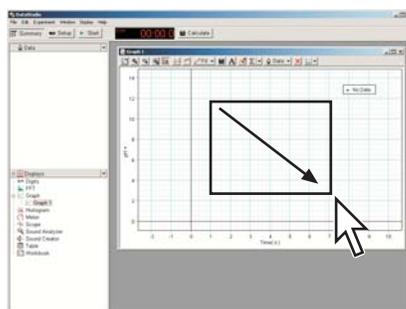
1. In the toolbar of the graph, click **Data**.



2. Select the runs that you want to see in the graph.

### 7.1.4 Selecting data points in a graph

1. If two or more data runs are visible, click the data run that you want to select points from.
2. Drag the pointer diagonally on the graph to draw a rectangular outline.

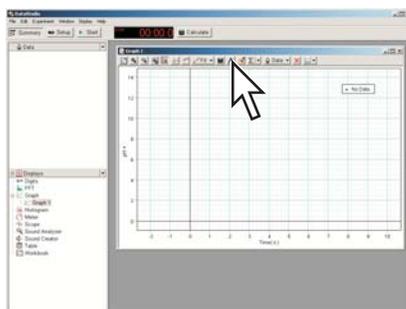


*Result:* Data points within the bounding outline are selected and highlighted.

*Task result:* Functions such as scale-to-fit, statistics, and curve fits will apply only to the selected points.

### 7.1.5 Adding a note to a graph

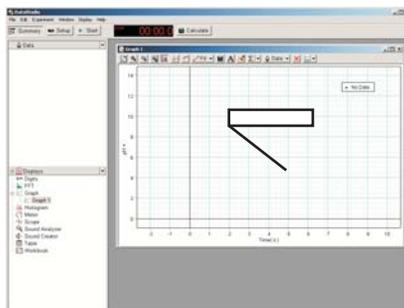
1. In the toolbar of the graph, click the **Note** button.



2. Click where you want the note to point.
3. Type the text of the note.

4. Click **OK**.

*Result:* The note appears on the graph.



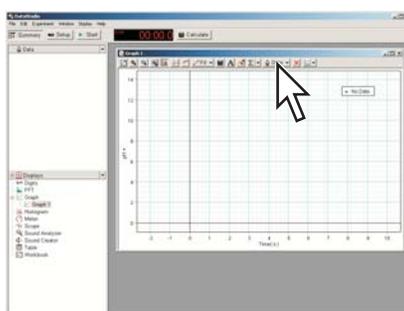
5. Drag the note to position it on the graph.

### 7.1.6 Removing a note from a graph

1. Click the note.
2. Press the Delete key on your keyboard.

### 7.1.7 Showing and hiding data runs in a graph

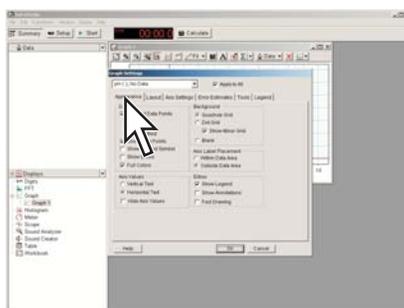
1. In the toolbar of the graph, click **Data**.



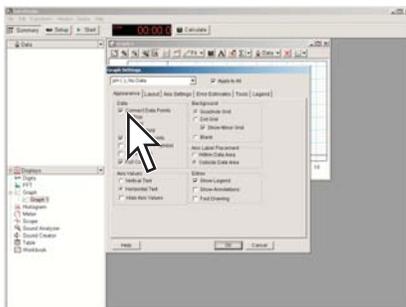
2. Select a run that you wish to see or cancel the selection of a run that you wish to hide.

### 7.1.8 Showing and hiding connecting lines between data points

1. Double-click the middle of the graph.  
*Result:* The Graph Settings window opens.
2. Click the **Appearance** tab.



3. Select or clear the **Connect Data Points** check box.

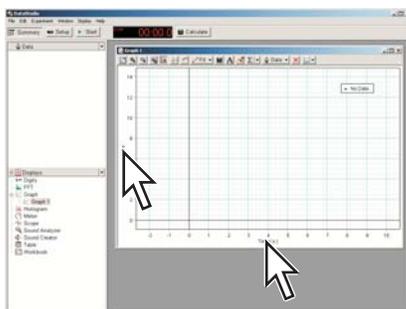


4. Click **OK**.

---

### 7.1.9 Changing the variable on the x- or y-axis

1. Click the measurement name on the x- or y-axis label



*Result:* A menu appears.

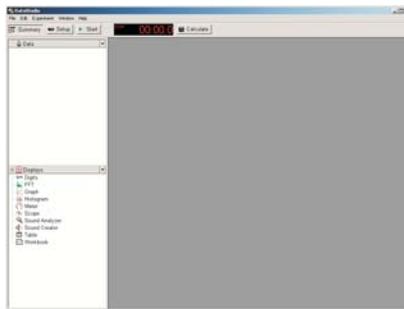
2. Click the measurement that you want.

### 7.1.10 Displaying multiple variables on the y-axis

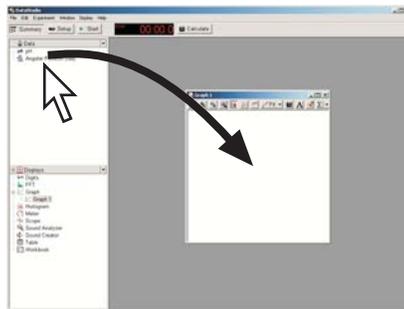
1. If the summary bar is not visible, click **Summary**.



*Result:* The summary bar appears.



2. Drag the desired additional measurement from the summary bar to the center of the graph.



### 7.1.11 Displaying multiple graphs

- ◆ To add a graph in its own window, complete the steps in Tech Tip 7.1.1.

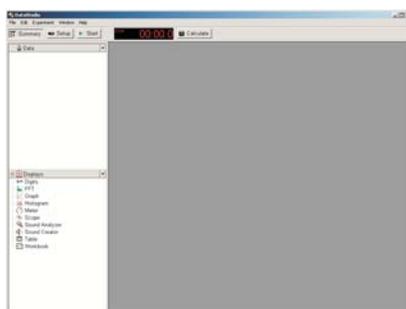
- ◆ To add an additional graph to an existing graph window, complete these steps:

Note: The two measurements will appear on separate graphs in the same window only if they have different units. If the two measurements have the same units, they will appear in the same graph.

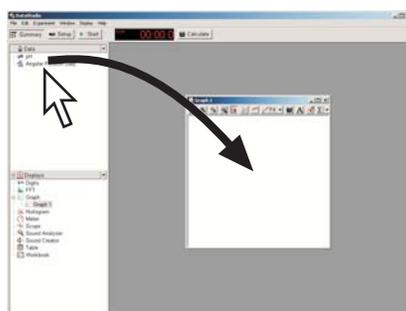
- a. If the summary bar is not visible, click **Summary**.



*Result:* The summary bar appears.

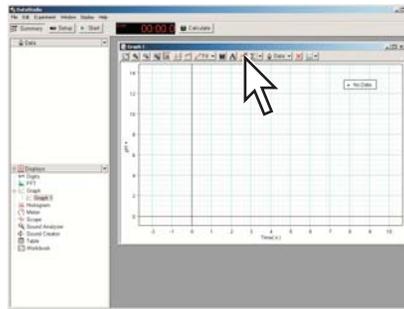


- b. Drag the measurement from the summary bar to the center of the graph.



### 7.1.12 Drawing a prediction

1. In the toolbar of the graph, click the **Prediction** button.



2. Click and drag on the graph to draw a prediction.

## 7.2 Table

### 7.2.1 Displaying data in a table

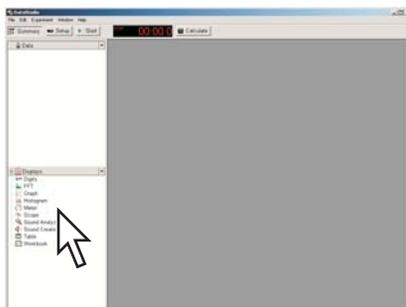
1. If the summary bar is not visible, click **Summary**.



*Result:* The summary bar appears.



2. In the summary bar, double-click **Table**.



3. If the Please Choose a Data Source window appears, click the measurement that you would like to see; then click **OK**.
4. Optionally, add additional columns<sup>(7.2.2)</sup>.

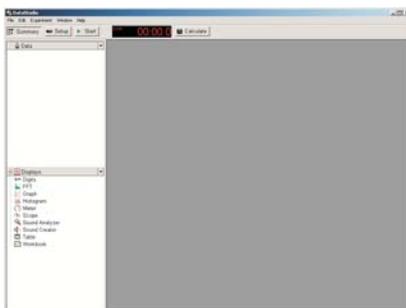
---

## 7.2.2 Adding a measurement to a table

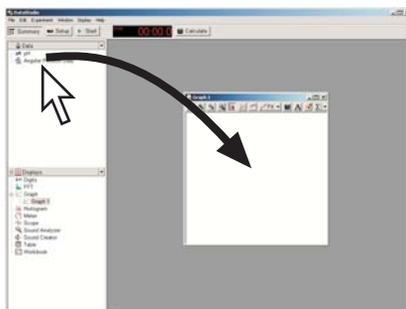
1. If the summary bar is not visible, click **Summary**.



*Result:* The summary bar appears.

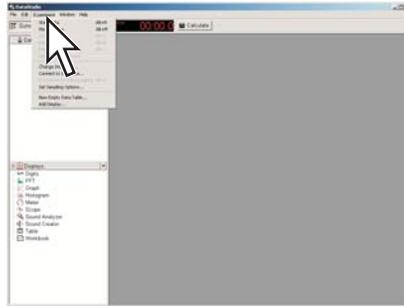


2. Drag the measurement from the summary bar to the center of the table.



### 7.2.3 Manually entering data into a table

1. Open the **Experiment** menu and select **New Empty Data Table**.



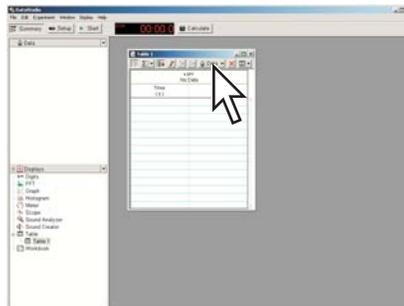
*Result:* An editable table appears.

2. Click the table.
3. Type numbers into the cells of the table. Press the Enter or Return key after typing in each cell.

*Task result:* The manually entered data set appears in the summary bar and is available for viewing in a graph or other display.

### 7.2.4 Showing and hiding data runs in a table

1. In the toolbar of the table, click **Data**.



2. Select a run that you wish to see or cancel the selection of a run that you wish to hide.

### 7.2.5 Changing the variable displayed in a column

1. Click the measurement name near the top of the table.

*Result:* A menu appears.

2. In the menu, click the desired variable.

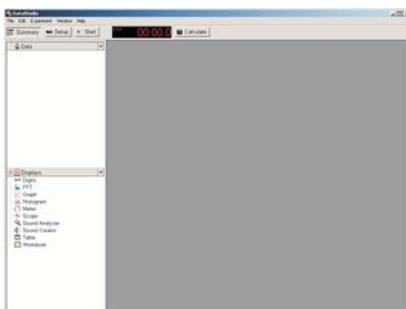
## 7.3 Digits display

### 7.3.1 Displaying data in a digits display

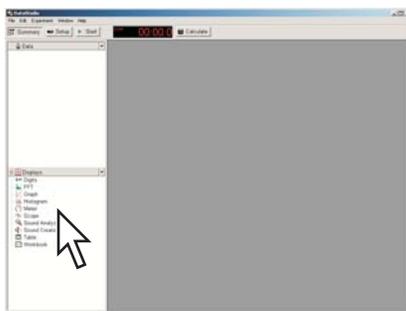
1. If the summary bar is not visible, click **Summary**.



*Result:* The summary bar appears.



2. In the summary bar, double-click **Digits**.



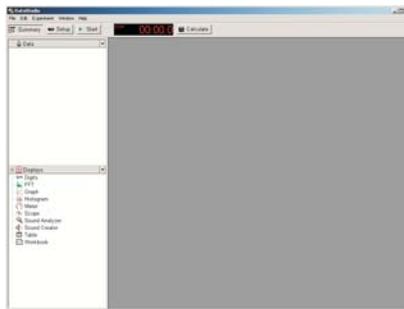
3. If the Please Choose a Data Source window appears, click the measurement that you would like to see; then click **OK**.

### 7.3.2 Adding a measurement to a digits display

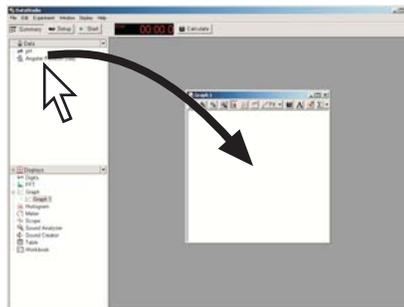
1. If the summary bar is not visible, click **Summary**.



*Result:* The summary bar appears.

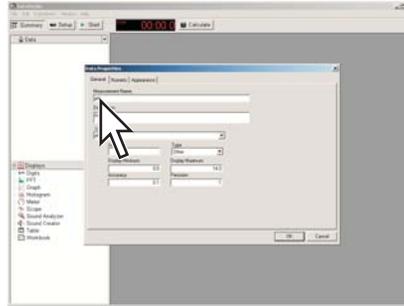


2. Drag the measurement from the summary bar to the middle of the digits display.





3. Enter the new name in the **Measurement Name** box.



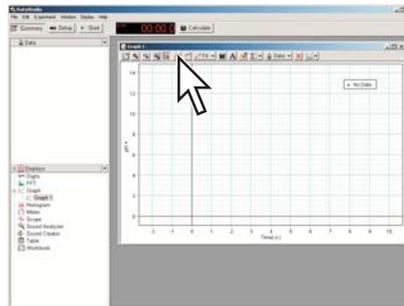
4. Click **OK**.

## Section 9: Analyzing data

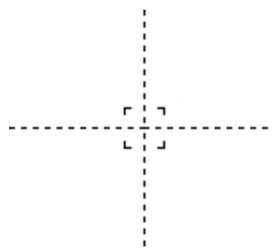
### 9.1

Finding the coordinates of a point in a graph

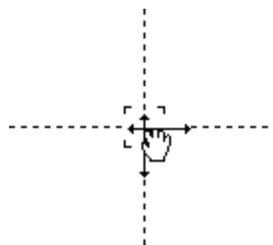
1. In the toolbar of the graph, click the **Smart Tool** button.



*Result:* The Smart Tool appears in the middle of the graph.



2. Point the mouse to the center of the Smart Tool so that the Smart Tool cursor appears.



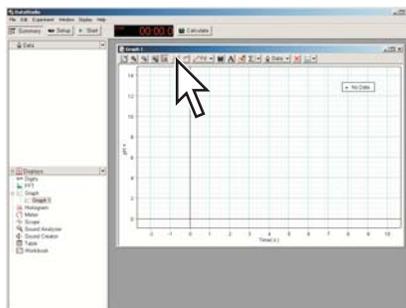
3. Drag the Smart Tool to a point on the graph.

*Result:* The coordinates of the Smart Tool are displayed.

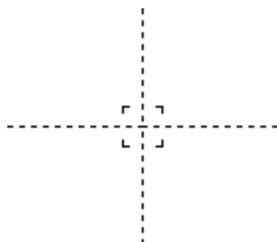
## 9.2

Measuring the difference between two points in a graph

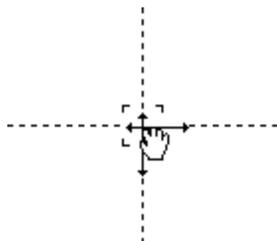
1. In the toolbar of the graph, click the **Smart Tool** button.



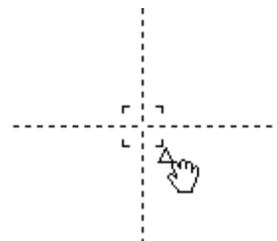
*Result:* The Smart Tool appears in the middle of the graph.



2. Point the mouse to the center of the Smart Tool so that the Smart Tool cursor appears.



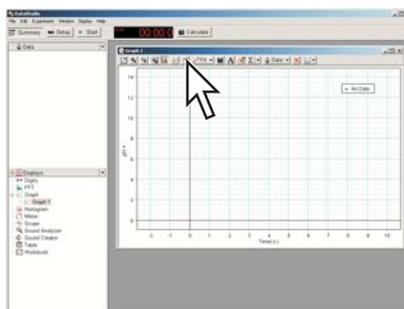
3. Drag the Smart Tool to a point on the graph.
4. Point the mouse to the corner of the Smart Tool so that a delta cursor appears.



5. Drag the cursor to a second point on the graph.  
The x- and y-differences are displayed.

### 9.3 Finding the slope at a point on a data plot

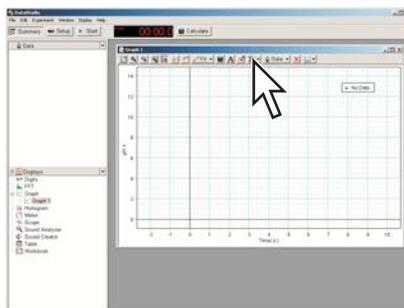
1. In the toolbar of the graph, click the **Slope Tool** button.



2. Drag the Slope Tool to a point on the graph.  
The slope is displayed.
3. To find the coordinates of the point where the Slope Tool is, follow the steps in Tech Tip 9.1.

### 9.4 Viewing statistics of data

- ◆ In the toolbar of the graph, click the **Statistics** button.

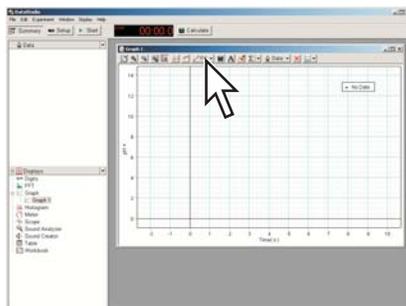


*Result:* Statistics appear.

- ◆ To select or hide different types of statistics, click the arrow next to the **Statistics** button.
- ◆ To view the statistics of a portion of a data set, select that portion. <sup>◆(7.1.4)</sup>

## 9.5 Applying a curve fit

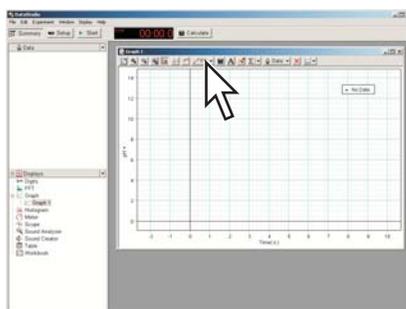
1. In the toolbar of the graph, click **Fit**.



2. Select the desired curve fit.  
The fit appears on the graph.
3. Optionally, select part of the data set for the curve fit to be applied to.  $\diamond(7.1.4)$

## 9.6 Finding the slope and intercept of a best-fit line

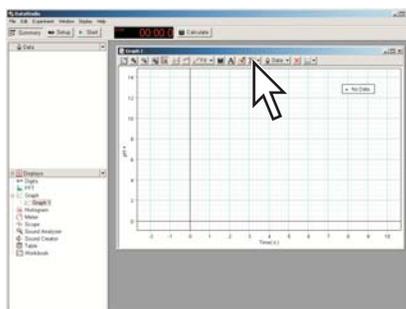
1. In the toolbar of the graph, click **Fit**.



2. Select **Linear Fit**.  
The slope and intercept of the best-fit line are displayed.
3. Optionally, select part of the data set for the linear fit to be applied to.  $\diamond(7.1.4)$

## 9.7 Finding the area under a curve

1. In the toolbar of the graph, click the arrow next to the **Statistics** button.



*Result:* A menu opens

- In the menu, click **Area**.  
*Result:* The area under the curve is displayed.
- Optionally, select part of the data set for the area to be applied to. <sup>◆(7.1.4)</sup>

## Section 10: Data operations

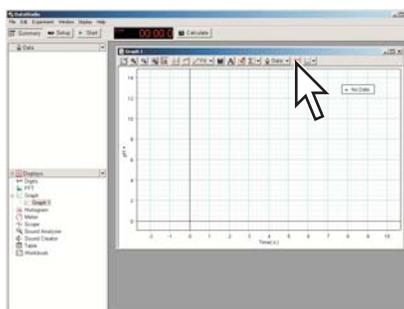
### 10.1 Editing manually entered data.

- Display the manually entered data set in a table. <sup>◆(7.2.1)</sup>
- Click a cell in the table.
- Type new number into the cell and press the Enter or Return key.

Repeat this task to edit data in other cells.

### 10.2 Deleting data points

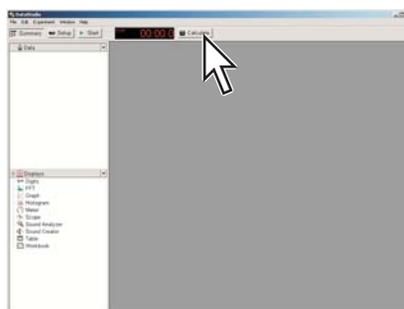
- In a graph or table, select the data point (or points) to be deleted. <sup>◆(7.1.4)(7.2.3)</sup>
- In the toolbar of the graph, click the **Remove Selected Data** button.



- If DataStudio prompts you to make an editable copy of the data, click **OK**.

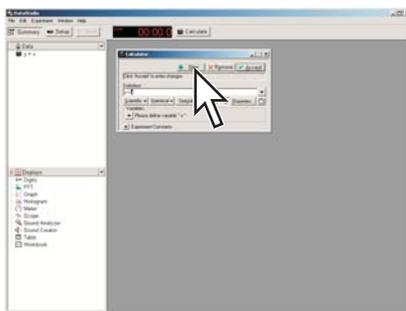
### 10.3 Creating calculated data

- Click **Calculate**.



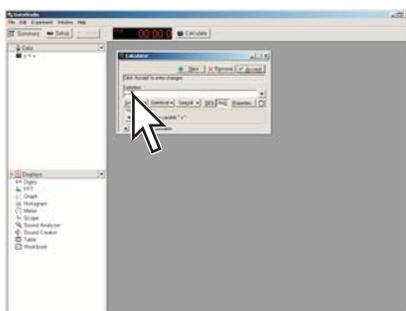
*Result:* The Calculator window opens.

2. If a previous calculation that you do not wish to overwrite is shown, click **New**.



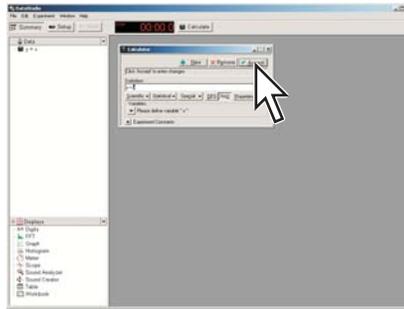
*Result:* A new calculation is started.

3. In the definition box, type an expression.

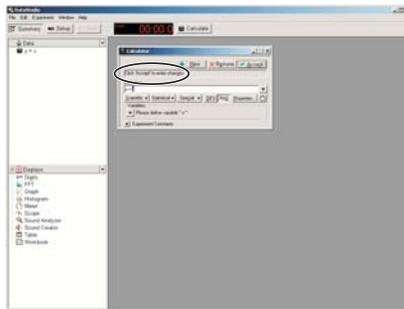


If you have been given an expression in your lab instructions, type it into the calculator exactly as it appears in the instructions. Entering the expression exactly as given will allow DataStudio to automatically associate variables in the expression with sensor measurements.

4. Click **Accept**.



If the expression has been entered correctly, a message appears in the Calculator window indicating that the calculation is complete.



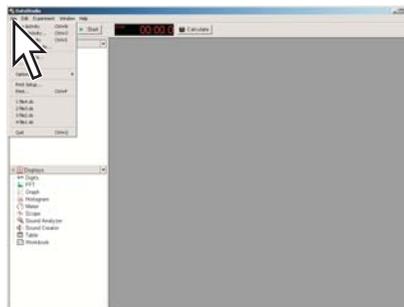
5. Optionally, close the Calculator window.

*Task result:* The calculation is now available for viewing in a graph, table, or other display.

## Section 11: Saving and sharing your experiment

### 11.1 Saving your experiment

1. Open the **File** menu and select **Save Activity As**.

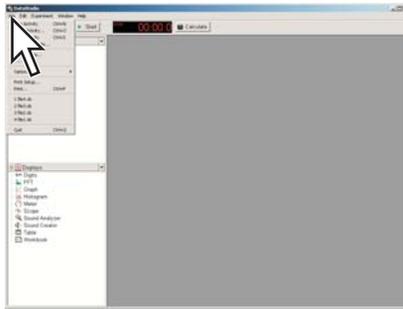


2. Select a folder
3. Type a filename and click **Save**.

### 11.2 Printing

1. Click the graph, table, or other window that you would like to print.

2. Open the **File** menu and select **Print**.

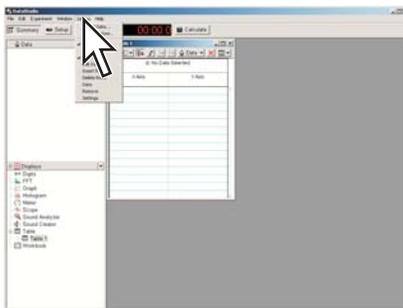


3. Click **OK**.

---

### 11.3 Exporting data for use in mapping software

1. Display the data that you wish to export in a table. <sup>◆(7.2.1)</sup>
2. Open the **Display** menu and select **Export Data**.



3. Select a folder.
4. Type a filename and click **Save**.

# Appendix E: Correlations to Education Standards

This appendix lists the National Science Content Standards that these labs address. Additional information regarding other standards these labs address can be found in the section, "About Correlations to Standards."

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## Scientific Inquiry

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### Correlation to the National Science Education Standards

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Identify questions and concepts that guide scientific investigations
    - ◆ Design and conduct scientific investigations
    - ◆ Use technology and mathematics to improve investigations and communications
    - ◆ Formulate and revise scientific explanations and models using logic and evidence
    - ◆ Recognize and analyze alternative explanations and models
    - ◆ Communicate and defend a scientific argument
  - ◆ Understanding about scientific inquiry
    - ◆ Scientists usually inquire about how physical, living, or designed systems function
    - ◆ Scientists conduct investigations for a wide variety of reasons
    - ◆ Scientists rely on technology to enhance the gathering and manipulation of data
    - ◆ Mathematics is essential in scientific inquiry
    - ◆ Scientific explanations must adhere to criteria such as: a proposed explanation must be logically consistent; it must abide by the rules of evidence; it must be open to questions and possible modification; and it must be based on historical and current scientific knowledge
    - ◆ Results of scientific inquiry—new knowledge and methods—emerge from different types of investigations and public communication among scientists
2. Content Standard E: Science and Technology
  - ◆ Abilities of technological design
    - ◆ Identify a problem or design an opportunity
    - ◆ Propose designs and choose between alternative solutions
    - ◆ Implement a proposed solution
    - ◆ Evaluate the solution and its consequences
    - ◆ Communicate the problem, process, and solution
  - ◆ Understandings about science and technology
    - ◆ Scientists in different disciplines ask different questions, use different methods of investigation, and accept different types of evidence to support their explanations
    - ◆ Science often advances with the introduction of new technologies

- ◆ Creativity, imagination, and a good knowledge base are all required in the work of science and engineering
- ◆ Science and technology are pursued for different purposes

## **Lab 1: Determining the Empirical Formula of a Compound**

---

### **Correlation to the National Science Education Content Standards**

Content Standard A: Science as Inquiry

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.

### **Correlation to the AP Chemistry Topic Outline**

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III. Reactions

B. Stoichiometry

3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

V. Laboratory: Chemical Calculations

1. Percentage composition
2. Empirical and molecular formulas from experimental data
5. Stoichiometric relations using the concept of the mole

## **Lab 2: Determine the Percentage of Water in a Hydrate**

---

### **Correlation to the National Science Education Content Standards**

Content Standard A: Science as Inquiry

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.

---

**Correlation to the AP Chemistry Topic Outline**

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III. Reactions

B. Stoichiometry

3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

V. Laboratory: Chemical Calculations

1. Percentage composition
2. Empirical and molecular formulas from experimental data

---

**Lab 3: Determine the Molar Mass of a Volatile Liquid**

---

**Correlation to the National Science Education Content Standards**

Content Standard A: Science as Inquiry

1. Content Standard A: Science as Inquiry

- ◆ Abilities necessary to do scientific inquiry
  - ◆ Use technology and mathematics to improve investigations and communications.
- ◆ Understandings about scientific inquiry
  - ◆ Mathematics is essential in scientific inquiry.

2. Content Standard B: Physical Science

- ◆ Structure of atoms
  - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.

**Correlation to the AP Chemistry Topic Outline**

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II. States of Matter

A. Gases

1. Laws of ideal gases
  - a. Equation of state for an ideal gas
2. Kinetic-molecular theory
  - a. Interpretation of ideal gas laws on the basis of this theory
  - b. Avogadro's hypothesis and the mole concept
  - c. Dependence of kinetic energy of molecules on temperature

V. Laboratory: Chemical Calculations

3. Molar masses from gas density, freezing-point, and boiling-point measurements
4. Gas laws, including the ideal gas law, Dalton's law, and Graham's law

## Lab 4: Molecular Weight by Freezing Point Depression

---

### Correlation to the National Science Education Content Standards

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ The structure and properties of matter
    - ◆ The physical properties of compounds reflect the nature of the interactions among its molecules.
    - ◆ Solids, liquids, and gases differ in the distances and angles between molecules or atoms and therefore the energy that binds them together.

### Correlation to the AP Chemistry Topic Outline

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#### II. States of Matter

##### B. Liquids and solids

1. Liquids and solids from the kinetic-molecular viewpoint
2. Phase diagrams of one-component systems
3. Changes of state, including critical points and triple points
4. Structure of solids; lattice energies

#### V. Laboratory: Chemical Calculations

5. Stoichiometric relations using the concept of the mole

## Lab 5: Molar Volume of a Gas

---

### Correlation to the National Science Education Content Standards

#### Content Standard A: Science as Inquiry

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.

**Correlation to the AP Chemistry Topic Outline**

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## II. States of Matter

## A. Gases

1. Laws of ideal gases
  - a. Equation of state for an ideal gas
2. Kinetic-molecular theory
  - a. Interpretation of ideal gas laws on the basis of this theory
  - b. Avogadro's hypothesis and the mole concept
  - c. Dependence of kinetic energy of molecules on temperature

## III. Reactions

## B. Stoichiometry

3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

## V. Laboratory: Chemical Calculations

4. Gas laws, including the ideal gas law, Dalton's law, and Graham's law

**Lab 6: Standardizing a Solution of Sodium Hydroxide****Correlation to the National Science Education Content Standards**

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ Chemical reactions
    - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.

**Correlation to the AP Chemistry Topic Outline**

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## III. Reactions

## A. Reaction types

1. Acid-base reactions

## B. Stoichiometry

3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

- V. Laboratory: Chemical Calculations
5. Stoichiometric relations using the concept of the mole

### **Lab 7: Acid–Base Titration**

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1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ Chemical reactions
    - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.

#### **Correlation to the AP Chemistry Topic Outline**

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#### III. Reactions

##### A. Reaction types

1. Acid-base reactions

##### B. Stoichiometry

3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

##### C. Equilibrium

1. Concept of dynamic equilibrium, physical and chemical; Le Chatelier's principle; equilibrium constants
2. Quantitative treatment
  - b. Equilibrium constants for reaction in solution
    - (1) Constants for acids and bases:  $pK$ ;  $pH$

- V. Laboratory: Chemical Calculations
5. Stoichiometric relations using the concept of the mole

### **Lab 8: Oxidation–Reduction Titration**

---

#### **Correlation to the National Science Education Content Standards**

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science

- ◆ Structure of atoms
  - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
- ◆ Chemical reactions
  - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.

### **Correlation to the AP Chemistry Topic Outline**

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#### III. Reactions

##### A. Reaction types

##### 3. Oxidation-reduction reactions

- a. Oxidation number
- b. The role of the electron in oxidation-reduction

##### B. Stoichiometry

2. Balancing of equations including those for redox reactions
3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

#### V. Laboratory: Chemical Calculations

5. Stoichiometric relations using the concept of the mole

## **Lab 9: Mole Relationships in a Chemical Reaction**

### **Correlation to the National Science Education Content Standards**

#### 1. Content Standard A: Science as Inquiry

- ◆ Abilities necessary to do scientific inquiry
  - ◆ Use technology and mathematics to improve investigations and communications.
- ◆ Understandings about scientific inquiry
  - ◆ Mathematics is essential in scientific inquiry.

#### 2. Content Standard B: Physical Science

- ◆ Structure of atoms
  - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
- ◆ The structure and properties of matter
  - ◆ The physical properties of compounds reflect the nature of the interactions among its molecules.
- ◆ Chemical reactions
  - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.

**Correlation to the AP Chemistry Topic Outline**

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III. Reactions

A. Reaction types

2. Precipitation reactions

B. Stoichiometry

2. Balancing of equations including those for redox reactions
3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

V. Laboratory: Chemical Calculations

1. Percentage composition
2. Empirical and molecular formulas from experimental data
5. Stoichiometric relations using the concept of the mole

**Lab 10: Determine the Equilibrium Constant for a Chemical Reaction**

---

1. Content Standard A: Science as Inquiry

- ◆ Abilities necessary to do scientific inquiry
  - ◆ Use technology and mathematics to improve investigations and communications.
- ◆ Understandings about scientific inquiry
  - ◆ Mathematics is essential in scientific inquiry.

2. Content Standard B: Physical Science

- ◆ Structure of atoms
  - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
- ◆ The structure and properties of matter
  - ◆ The physical properties of compounds reflect the nature of the interactions among its molecules.
- ◆ Chemical reactions
  - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.

**Correlation to the AP Chemistry Topic Outline**

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III. Reactions

C. Equilibrium

1. Concept of dynamic equilibrium, physical and chemical; Le Chatelier's principle; equilibrium constants
2. Quantitative treatment
  - b. Equilibrium constants for reactions in solution

V. Laboratory: Chemical Calculations

8. Equilibrium constants and their applications, including their use for simultaneous equilibria

---

**Lab 11: Using Different Indicators for pH Determination**

---

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ Chemical reactions
    - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.

**Correlation to the AP Chemistry Topic Outline**

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## III. Reactions

## A. Reaction types

1. Acid-base reactions

## C. Equilibrium

1. Concept of dynamic equilibrium, physical and chemical; Le Chatelier's principle; equilibrium constants
2. Quantitative treatment
  - a. Equilibrium constants for reactions in solution
    - (1) Constants for acids and bases;  $pK$ ;  $pH$

## V. Laboratory: Chemical Calculations

5. Stoichiometric relations using the concept of the mole

---

**Lab 12: Determination of the Rate of the Decomposition of Hydrogen Peroxide**

---

**Correlation to the National Science Education Content Standards**

## Content Standard A: Science as Inquiry

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ Chemical reactions

## Appendix E: Correlations to Education Standards

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- ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.
- ◆ Catalysts, such as metal surfaces, accelerate chemical reactions.

### Correlation to the AP Chemistry Topic Outline

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#### II. States of Matter

##### A. Gases

1. Laws of ideal gases
  - a. Equation of state for an ideal gas
  - b. Partial pressures

#### III. Reactions

##### A. Reaction types

3. Oxidation-reduction reactions

##### D. Kinetics

1. Concept of rate of reaction
2. Use of experimental data and graphical analysis to determine reactant order, rate constants, and reaction rate laws
4. Energy of activation; the role of catalysts

#### V. Laboratory: Chemical Calculations

11. Kinetics calculations

## Lab 13: Enthalpy of a Chemical Reaction

---

### Correlation to the National Science Education Content Standards

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ Chemical reactions
    - ◆ Chemical reactions may release or consume energy.

### Correlation to the AP Chemistry Topic Outline

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#### III Reactions

##### E. Thermodynamics

2. First law: change in enthalpy; heat of formation; heat of reaction; Hess's law; heats of vaporization and fusion; calorimetry

3. Second law: entropy; free energy of formation; free energy of reaction; dependence of change in free energy on enthalpy and entropy changes
- V. Laboratory: Chemical Calculations
10. Thermodynamic and thermochemical calculations

---

## Lab 14a: Separation and Analysis of Cations

---

### Correlation to the National Science Education Content Standards

Content Standard A: Science as Inquiry

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ The structure and properties of matter
    - ◆ The physical properties of compounds reflect the nature of the interactions among its molecules.
  - ◆ Chemical reactions
    - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.

### Correlation to the AP Chemistry Topic Outline

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III. Reactions

- A. Reaction Types
  1. Acid-base reactions
  2. Precipitation reactions
  3. Oxidation-reduction reactions
- B. Stoichiometry
  1. Ionic and molecular species present in chemical systems: net ionic equations
  2. Balancing of equations including those for redox reactions

---

## Lab 14b: Analysis of Anions

---

### Correlation to the National Science Education Content Standards

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry

### 2. Content Standard B: Physical Science

- ◆ Structure of atoms
  - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
- ◆ The structure and properties of matter
  - ◆ The physical properties of compounds reflect the nature of the interactions among its molecules.
- ◆ Chemical reactions
  - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.

### **Correlation to the AP Chemistry Topic Outline**

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#### III. Reactions

##### A. Reaction types

2. Precipitation reactions
3. Oxidation-reduction reactions

##### B. Stoichiometry

1. Ionic and molecular species present in chemical systems: net ionic equations
2. Balancing of equations including those for redox reactions

#### IV. Descriptive Chemistry

1. Chemical reactivity and products of chemical reactions

## **Lab 15a: Synthesis of a Coordination Compound**

---

### **Correlation to the National Science Education Content Standards**

#### Content Standard A: Science as Inquiry

##### 1. Content Standard A: Science as Inquiry

- ◆ Abilities necessary to do scientific inquiry
  - ◆ Use technology and mathematics to improve investigations and communications.
- ◆ Understandings about scientific inquiry
  - ◆ Mathematics is essential in scientific inquiry.

##### 2. Content Standard B: Physical Science

- ◆ Structure of atoms
- ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
- ◆ Chemical reactions
  - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.

**Correlation to the AP Chemistry Topic Outline**

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## III. Reactions

## A. Reaction types

1. Acid-base reactions; concepts of Arrhenius, Brønsted-Lowry, and Lewis; coordination complexes; amphoterism
2. Precipitation reactions

## B. Stoichiometry

3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

## V. Laboratory: Chemical Calculations

1. Percentage composition
5. Stoichiometric relations using the concept of the mole

**Lab 15b: Analysis of a Coordination Compound****Correlation to the National Science Education Content Standards**

## Content Standard A: Science as Inquiry

## 1. Content Standard A: Science as Inquiry

- ◆ Abilities necessary to do scientific inquiry
  - ◆ Use technology and mathematics to improve investigations and communications.
- ◆ Understandings about scientific inquiry
  - ◆ Mathematics is essential in scientific inquiry.

## 2. Content Standard B: Physical Science

- ◆ Structure of atoms
- ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
- ◆ Chemical reactions
  - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.

**Correlation to the AP Chemistry Topic Outline**

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## II. States of Matter

## C. Solutions

1. Types of solutions and factors affecting solubility

## III. Reactions

## A. Reaction types

1. Acid-base reactions; concepts of Arrhenius, Brønsted-Lowry, and Lewis; coordination complexes; amphoterism
2. Precipitation reactions

## V. Laboratory: Chemical Calculations

5. Stoichiometric relations using the concept of the mole

## Lab 16: Gravimetric Determination of a Precipitate

---

### Correlation to the National Science Education Content Standards

Content Standard A: Science as Inquiry

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ Chemical reactions
    - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.

### Correlation to the AP Chemistry Topic Outline

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III. Reactions

A. Reaction types

2. Precipitation reactions

B. Stoichiometry

3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

V. Laboratory: Chemical Calculations

1. Percentage composition

2. Empirical and molecular formulas from experimental data

## Lab 17a: Absorption Spectra

---

### Correlation to the National Science Education Content Standards

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ The structure and properties of matter
    - ◆ The physical properties of compounds reflect the nature of the interactions among its molecules.

**Correlation to the AP Chemistry Topic Outline**

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- I. Structure of Matter
  - A. Atomic theory and atomic structure
    - 1. Evidence for the atomic theory
    - 4. Electron energy levels; atomic spectra, quantum numbers, atomic orbitals

---

**Lab 17b: Colorimetric Analysis**

---

**Correlation to the National Science Education Content Standards**

- 1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
    - ◆ Understandings about scientific inquiry
- 2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ The structure and properties of matter
    - ◆ The physical properties of compounds reflect the nature of the interactions among its molecules.

**Correlation to the AP Chemistry Topic Outline**

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- II. States of Matter
  - C. Solutions
    - 2. Methods of expressing concentration
- V. Laboratory: Chemical Calculations
  - 6. Mole fractions; molar and molal solutions

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**Lab 18: Separation by Liquid Chromatography**

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**Correlation to the National Science Education Content Standards**

- 1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
    - ◆ Understandings about scientific inquiry
      - ◆ Mathematics is essential in scientific inquiry.
- 2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ The structure and properties of matter

## Appendix E: Correlations to Education Standards

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- ◆ Atoms interact with one another by transferring or sharing electrons that are furthest from the nucleus.
- ◆ The physical properties of compounds reflect the nature of the interactions among its molecules.

### Correlation to the AP Chemistry Topic Outline

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#### I. Structure of matter

##### B. Chemical bonding

1. Binding forces
  - a. Types: ionic, covalent, metallic, hydrogen bonding, van der Waals (including London dispersion forces)
  - b. Relationships to states, structure, and properties of matter
  - c. Polarity of bonds, electronegativities

## Lab 19: Properties of Buffer Solutions

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### Correlation to the National Science Education Content Standards

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ Chemical reactions
    - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.

### Correlation to the AP Chemistry Topic Outline

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#### III. Reactions

##### A. Reaction types

1. Acid-base reactions

##### B. Stoichiometry

3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

##### C. Equilibrium

1. Concept of dynamic equilibrium, physical and chemical; Le Chatelier's principle; equilibrium constants

2. Quantitative treatment
    - b. Equilibrium constants for reactions in solution
      - (1) Constants for acids and bases;  $pK$ ;  $pH$
      - (3) Common ion effect; buffers; hydrolysis
- V. Laboratory: Chemical Calculations
5. Stoichiometric relations using the concept of the mole

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## Lab 20: Determination of Electrochemical Series

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### Correlation to the National Science Education Content Standards

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ Chemical reactions
    - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.

### Correlation to the AP Chemistry Topic Outline

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- I. Structure of Matter
- A. Atomic theory and atomic structure
    5. Periodic relationships including, for example, atomic radii, ionization energies, electron affinities, oxidation states
- III. Reactions
- A. Reaction types
    3. Oxidation-reduction reactions
      - a. Oxidation number
      - b. The role of the electron in oxidation-reduction
      - c. Electrochemistry: electrolytic and galvanic cells
- V. Laboratory: Chemical Calculations
9. Standard electrode potentials and their use

## Lab 21: Electroplating

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### Correlation to the National Science Education Content Standards

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ Chemical reactions
    - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.

### Correlation to the AP Chemistry Topic Outline

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#### I. Structure of Matter

##### A. Atomic theory and atomic structure

5. Periodic relationships including, for example, atomic radii, ionization energies, electron affinities, oxidation states

#### III. Reactions

##### A. Reaction types

3. Oxidation-reduction reactions
  - a. Oxidation number
  - b. The role of the electron in oxidation-reduction
  - c. Electrochemistry: electrolytic and galvanic cells; Faraday's laws

#### V. Calculations

7. Faraday's law of electrolysis

## Lab 22a: Organic Synthesis I—Preparation

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### Correlation to the National Science Education Content Standards

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.

- ◆ The structure and properties of matter
  - ◆ Carbon atoms can bond to one another in chains, rings, and branching networks to form a variety of structures, including synthetic polymers, oils, and the large molecules essential to life.
- ◆ Chemical reactions
  - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.
  - ◆ Catalysts, such as metal surfaces, accelerate chemical reactions.

### **Correlation to the AP Chemistry Topic Outline**

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#### III. Reactions

##### A. Reaction types

###### 1. Acid-base reactions

##### B. Stoichiometry

###### 3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

#### IV. Descriptive Chemistry

##### 3. Introduction to organic chemistry: hydrocarbons and functional groups (structure, nomenclature, chemical properties)

#### V. Laboratory: Chemical Calculations

##### 5. Stoichiometric relations using the concept of the mole

## **Lab 22b: Organic Synthesis II—Analysis**

### **Correlation to the National Science Education Content Standards**

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ The structure and properties of matter
    - ◆ Carbon atoms can bond to one another in chains, rings, and branching networks to form a variety of structures, including synthetic polymers, oils, and the large molecules essential to life.
  - ◆ Chemical reactions
    - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.

**Correlation to the AP Chemistry Topic Outline**

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III. Reactions

A. Reaction types

1. Acid-base reactions

B. Stoichiometry

3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

IV. Descriptive Chemistry

- 3 Introduction to organic chemistry: hydrocarbons and functional groups (structure, nomenclature, chemical properties)

V. Laboratory: Chemical Calculations

5. Stoichiometric relations using the concept of the mole; titration calculations

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**Lab 23: Determination of a Solubility Product**

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**Correlation to the National Science Education Content Standards**

Content Standard A: Science as Inquiry

1. Content Standard A: Science as Inquiry

- ◆ Abilities necessary to do scientific inquiry
  - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.

2. Content Standard B: Physical Science

- ◆ Structure of atoms
  - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.

**Correlation to the AP Chemistry Topic Outline**

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II. States of Matter

B. Liquids and solids

1. Liquids and solids from the kinetic-molecular viewpoint

III. Reactions

C. Equilibrium

1. Concept of dynamic equilibrium, physical and chemical; Le Chatelier's principle; equilibrium constants
2. Quantitative treatment
  - a. Equilibrium constants for reactions in solution
    - (2) Solubility product constants and their applications to precipitation and the dissolution of slightly soluble compounds

V. Calculations

5. Stoichiometric relations using the concept of the mole; titration calculations

---

**Lab 24: Determining  $K_a$  by Half-Titration of a Weak Acid**

---

**Correlation to the National Science Education Content Standards**

Content Standard A: Science as Inquiry

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.

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## III. Reactions

## A. Reaction types

1. Acid-base reactions

## B. Stoichiometry

3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

## C. Equilibrium

1. Concept of dynamic equilibrium, physical and chemical; Le Chatelier's principle; equilibrium constants
2. Quantitative treatment
  - b. Equilibrium constants for reactions in solution
    - (1) Constants for acids and bases:  $pK$ ;  $pH$

## V. Calculations

5. Stoichiometric relations using the concept of the mole; titration calculations
11. Kinetics calculations

---

**Lab 25: Order of Reaction**

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**Correlation to the National Science Education Content Standards**

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.

### 2. Content Standard B: Physical Science

- ◆ Structure of atoms
  - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
- ◆ The structure and properties of matter
  - ◆ The physical properties of compounds reflect the nature of the interactions among its molecules.
- ◆ Chemical reactions
  - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.
  - ◆ Chemical reactions can take place in time periods ranging from the few femtoseconds ( $10^{-15}$  seconds) required for an atom to move a fraction of a chemical bond distance to geologic time scales of billions of years.

### Correlation to the AP Chemistry Topic Outline

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#### II. States of Matter

##### C. Solutions

##### 2. Methods of expressing concentration

#### III. Reactions

##### D. Kinetics

##### 1. Concept of rate of reaction

#### V. Laboratory: Chemical Calculations

##### 11. Kinetics calculations

## Lab 26: Conductometric Titration

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### Correlation to the National Science Education Content Standards

#### Content Standard A: Science as Inquiry

##### 1. Content Standard A: Science as Inquiry

- ◆ Abilities necessary to do scientific inquiry
  - ◆ Use technology and mathematics to improve investigations and communications.
- ◆ Understandings about scientific inquiry
  - ◆ Mathematics is essential in scientific inquiry.

##### 2. Content Standard B: Physical Science

- ◆ Structure of atoms
  - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.

**Correlation to the AP Chemistry Topic Outline**

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## III. Reactions

## A. Reaction types

1. Acid-base reactions
2. Precipitation reactions

## B. Stoichiometry

1. Ionic and molecular species present in chemical systems: net ionic equations
3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

## V. Laboratory: Chemical Calculations

5. Stoichiometric relations using the concept of the mole; titration calculations

**Lab 27: Identifying an Unknown Metal**

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**Correlation to the National Science Education Content Standards**

## Content Standard A: Science as Inquiry

## 1. Content Standard A: Science as Inquiry

- ◆ Abilities necessary to do scientific inquiry
  - ◆ Use technology and mathematics to improve investigations and communications.
- ◆ Understandings about scientific inquiry
  - ◆ Mathematics is essential in scientific inquiry.

## 2. Content Standard B: Physical Science

- ◆ Structure of atoms
  - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.

**Correlation to the AP Chemistry Topic Outline**

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## II. States of Matter

## A. Gases

1. Laws of ideal gases
  - a. Equation of state for an ideal gas
2. Kinetic-molecular theory
  - a. Interpretation of ideal gas laws on the basis of this theory
  - b. Avogadro's hypothesis and the mole concept

## III. Reactions

## A. Reaction types

3. Oxidation-reduction reactions
  - a. Oxidation number

## B. Stoichiometry

3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

V. Laboratory: Chemical Calculations

4. Gas laws, including the ideal gas law, Dalton's law, and Graham's law

## **Lab 28: Molecular Interaction in Ethanol and Acetone**

---

### **Correlation to the National Science Education Content Standards**

Content Standard A: Science as Inquiry

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ The structure and properties of matter
    - ◆ The physical properties of compounds reflect the nature of the interactions among its molecules.

### **Correlation to the AP Chemistry Topic Outline**

(Source: Copyright © 2009. The College Board. Reproduced with permission. <http://apcentral.collegeboard.com>.)

- I. Structure of Matter
  - B. Chemical bonding
    1. Binding forces
      - a. Types: ionic, covalent, metallic, hydrogen bonding, van der Waals (including London dispersion forces)
- II. States of Matter
  - A. Gases
    1. Laws of ideal gases
      - a. Equation of state for an ideal gas
    2. Kinetic-molecular theory
      - a. Interpretation of ideal gas laws on the basis of this theory
      - b. Avogadro's hypothesis and the mole concept

## **Lab 29: Exploring Gas Laws**

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### **Correlation to the National Science Education Content Standards**

Content Standard A: Science as Inquiry

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry

- ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
    - ◆ Structure of atoms
      - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.

### Correlation to the AP Chemistry Topic Outline

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#### II. States of Matter

##### A. Gases

1. Laws of ideal gases
  - a. Equation of state for an ideal gas
2. Kinetic-molecular theory
  - a. Interpretation of ideal gas laws on the basis of this theory
  - c. Dependence of kinetic energy of molecules on temperature

#### V. Laboratory: Chemical Calculations

4. Gas laws, including the ideal gas law, Dalton's law, and Graham's law

## Lab 30: Determination of the $K_a$ Values of Two Isomeric Multi-Protic Acids

### Correlation to the National Science Education Content Standards

#### Content Standard A: Science as Inquiry

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.

### Correlation to the AP Chemistry Topic Outline

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#### III. Reactions

##### A. Reaction types

1. Acid-base reactions

##### C. Equilibrium

1. Concept of dynamic equilibrium, physical and chemical; Le Chatelier's principle; equilibrium constants
2. Quantitative treatment
  - a. Equilibrium constants for reactions in solution
    - (1) Constants for acids and bases;  $pK$ ; pH

V. Calculations

5. Stoichiometric relations using the concept of the mole; titration calculations

## **Lab 31: Determining the Half-Life of an Isotope**

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### **Correlation to the National Science Education Content Standards**

Content Standard A: Science as Inquiry

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
    - ◆ Radioactive isotopes are unstable and undergo spontaneous nuclear reactions, emitting particles and/or wave-like radiation.

### **Correlation to the AP Chemistry Topic Outline**

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I. Structure of Matter

- A. Atomic theory and atomic structure
  3. Atomic number and mass number; isotopes
- C. Nuclear chemistry: nuclear equations, half-lives, and radioactivity; chemical applications

## **Lab 32: The Breathalyzer™ Test for Alcohol**

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### **Correlation to the National Science Education Content Standards**

1. Content Standard A: Science as Inquiry
  - ◆ Abilities necessary to do scientific inquiry
    - ◆ Use technology and mathematics to improve investigations and communications.
  - ◆ Understandings about scientific inquiry
    - ◆ Mathematics is essential in scientific inquiry.
2. Content Standard B: Physical Science
  - ◆ Structure of atoms
    - ◆ Matter is made of minute particles called atoms, and atoms are composed of even smaller components.
  - ◆ Chemical reactions
    - ◆ A large number of important reactions involve the transfer of either electrons (oxidation/reduction reactions) or hydrogen ions (acid/base reactions) between reacting ions, molecules, or atoms.
    - ◆ Catalysts, such as metal surfaces, accelerate chemical reactions.

**Correlation to the AP Chemistry Topic Outline**

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III. Reactions

A. Reaction types

3. Oxidation-reduction reactions

B. Stoichiometry

3. Mass and volume relations with emphasis on the mole concept, including empirical formulas and limiting reactants

V. Laboratory: Chemical Calculations

1. Percentage composition
5. Stoichiometric relations using the concept of the mole