

Sabbatical Report

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SABBATICAL PROPOSAL

INTRODUCTION

The two-semester general chemistry sequence (CHEM 1A and CHEM 1B) is composed of lecture as well as an intense laboratory program. The Department has been a regional leader in the use of technology for almost 10 years, beginning with the introduction of Labworks computer-based data-acquisition systems into the teaching laboratory eight years ago. Since Labworks curriculum was very limited, Department members developed their own experiments. This led to the development of the current Department-authored CHEM 1A and CHEM 1B laboratory manuals. They are a compilation of traditional experiments used for many years and innovative experiments employing computers for data acquisition and analysis.

These lab manuals have benefited the Chemistry instructional program in several ways. First, instructors are using experiments that are customized to our facilities and equipment. Second, the price to students is almost half that of commercially produced lab manuals. Third, the publisher of the CHEM 1A and CHEM 1B lab manuals provides royalties to the Department, which has allowed the funding of an Awards Program used to acknowledge outstanding student achievement in the Chemistry department.

The Department-authored laboratory manuals do have some shortcomings. (1) The choice of experiments is limited. Frequently, instructors use the same sequence of experiments several semesters in a row. Students who withdraw or repeat the class perform exactly the same experiments, answer the same questions and write the same lab reports. This is not a pedagogically sound idea. (2) Virtually all of the current experiments consist of a series of procedural steps intended to achieve a predetermined outcome. While this type of experiment is necessary to teach students laboratory techniques and skills, it limits the opportunity for students to work in an environment that encourages discovery of concepts. (3) The Labworks experiments are written with data analysis instructions specific to the Labworks spreadsheet software. Many students have computers at home that can be used to process data with other spreadsheet programs, i.e. Microsoft Excel. Labworks software is very expensive and therefore not readily available to students outside of class.

Technology has advanced very rapidly over the past eight years, leaving our Labworks systems outdated. Experiments and certain laboratory techniques, which once required computers, can now be done with

sophisticated calculators. This technology is called calculator-based-laboratory (CBL) and its use is becoming widespread in science and math labs. Through the budgeting process last year, the Department has received funding to replace the twelve very obsolete and irreparable Labworks systems with thirty-two Texas Instruments calculators equipped with interfaces and probes capable of performing various experiments. In addition, the majority of science majors at Mt. SAC currently possess calculators capable of CBL allowing them to have the ability to process data outside of the classroom.

This technological shift of equipment provides an excellent framework to revise the CHEM 1A and CHEM 1B laboratory manuals. The current lab manuals cannot be used with the CBL systems. Our Labworks experiments need extensive revision and additional experiments from commercial sources must be adopted. My proposal for a sabbatical project entails rewriting the CHEM 1A and CHEM 1B lab manuals. Traditional experiments will be updated. Existing computer experiments will be rewritten to reflect the new CBL technology. New experiments will be developed to increase the selection and breadth. Collaborative group laboratory projects will be developed to allow students the opportunity to participate in open-ended guided-inquiry activities.

The ideas in this sabbatical project were presented at a Department meeting. All full-time Chemistry Department members in attendance unanimously supported this project. It is intended that the new CHEM 1A manual will be used beginning spring 2000 and the CHEM 1B manual will be used beginning fall 2000 semester.

PROJECT ACTIVITIES

1. Gaining Competency with Texas Instruments Calculator-Based-Laboratory (CBL) Technology

Competency using the CBL calculators, interfaces and probes must first be achieved before any experiment revisions can be done. Several types of probes, such as thermistors, pH electrodes, and timers are currently used for data acquisition. The CBL package provides a colorimeter, not currently used with Labworks. All of the current Labworks experiments will be performed repeatedly using CBL technology, until the procedures and data can be consistently reproduced. Additional procedures employing the colorimeter will be developed and tested. If the procedure seems dependable and the data reproducible, an experiment will be written.

2. Revising Existing Labworks® Experiments

CHEM 1A Labworks experiments:

Introduction to Computers and the Labworks Spreadsheet
Properties of Pennies
Computer Programming for Experimental Data Acquisition
Energy Changes
Phase Changes

List of CHEM 1B Labworks experiments:

Reaction Kinetics
Determination of K_{eq}
pH and Buffers
Acid Base Phenomenon
Electrochemistry
Nuclear Decay

Each current Labworks experiment will be rewritten. New introductions to each experiment will be written as needed to inform students of the implementation of CBL technology. Experimental procedures will be revised from Labworks directions to CBL directions. Data analysis instructions will be converted from Labworks spreadsheet to Microsoft Excel, a spreadsheet program readily available to all students in and outside of the chemistry teaching lab. Safety precautions and materials disposal instructions will be updated and added as needed. Once the revisions have been made, copies of each experiment will be circulated to all chemistry instructors who use the manual, for approval.

3. Revising Traditional Experiments

The current traditional experiments will be reviewed for typographical errors, vague language, mistakes and procedural errors. New illustrations will be created where needed. Safety precautions and materials disposal instructions will be updated and added as needed. The current questions will be revised, as necessary and new questions will be written for each experiment. Instructors who have recently used the lab manuals will have opportunity for input. Many have kept notes over the years. Again, once the revisions have been made, copies of each experiment will be circulated to all chemistry instructors who use the manual, for approval.

4. Developing Alternative Experiments

The list of exit skills for CHEM 1A and CHEM 1B will be reviewed. A list of laboratory techniques students will have mastered at the end of each course will be assembled and matched to the experiments. Alternative experiments will be developed, where possible, for experiments that teach

these essential skills to avoid repetition with students who withdraw or repeat the course. For example, titration is currently taught as a two-part experiment in CHEM 1A. Resources will be researched to find an alternative method of teaching titration. A parallel experiment will then be developed. Three new experiments that parallel existing experiments will be developed for each course. Once the experiments have been written, copies of each experiment will be circulated to all instructors who might use the manual, for approval.

5. Developing Group Laboratory Projects

Problems will be selected for which students currently get very little practical experience in the laboratory. The problems must be broad enough so that there is more than one solution, but specific enough that the solutions pertain to the selected topic. Available equipment and supplies will be checked. The experiment will be written with a scenario that presents the problem and a list of materials available to solve the problem. Two group laboratory projects will be developed for each course. Once the protocols for the experiments have been written, copies will be circulated to all chemistry instructors who use the manual, for approval.

Students will form teams and then be given a problem to solve for which they have the theoretical knowledge. They will use tools from the Langford "Quality Learning" program to develop an experimental procedure to solve the problem. The procedures will be peer reviewed. Each group will then perform its experiment and analyze their results.

TIMELINE

Revisions to the CHEM 1A lab manual will be made during Fall 1999 semester. Revisions to the CHEM 1B lab manual will be made during Spring 2000 semester. The same sequence of activities will be followed for both semesters. All laboratory work will be performed in the Mt. SAC Chemistry Department.

- Weeks 1-4** Revise existing traditional experiments (Activity 3). Correlate exit skills with laboratory techniques taught in individual experiments (Activity 4).
- Weeks 5-7** Perform existing Labworks experiments with CBL calculators to gain competence in the technology (Activity 1).
- Weeks 8-9** Revise Labworks experiments for CBL directions (Activity 2).

Weeks 10-15 Research resources for experiments that teach basic laboratory skills with alternate reagents (Activity 4). Select concepts for Group Lab Projects (Activity 5). Develop new experiment procedures.

Weeks 16-17 Write new experiments (Activities 4 & 5).

Week 18 Submit manual to publisher for printing.

ANTICIPATED BENEFITS

Benefits to Me

1. An opportunity to continue to develop a student-centered method of teaching.

The Molecular Science Project has allowed me the opportunity to implement student-centered methods of instruction into CHEM 1A and CHEM 1B lecture. The Molecular Science Project is one of five national curriculum-reform consortia funded by the National Science Foundation. The basis of the project is that traditional lecture is replaced by student-center computer-delivered instruction. I have now taught two courses that employ this type of instruction. Student learning has been very successful as evidence by a comparison of exam scores between traditionally taught sections and student-centered section. I have completed the Langford 4-day "Quality Learning" training and am looking forward to applying these techniques to the laboratory segment of CHEM 1A and CHEM 1B. The group laboratory projects will be designed to utilize Langford tools and techniques.

2. An improved laboratory teaching environment.

Students repeating the course will not have performed all of the experiments already. The boredom and "know-it-all" attitudes should be reduced. The temptation to use data previously acquired, submit old lab reports, supply the answers to questions to other students, etc will be reduced.

3. An opportunity to upgrade my technology skills by implementing the newest in calculator technology.

Most CHEM 1A and CHEM 1B students currently have programmable calculators. This project will encourage me to become at least as proficient in the use of these devices as the students.

Benefits to Students

1. Improvement in the laboratory experience.

Students repeating the course (a significant population) will not have to do all the same experiments over again. They frequently feel that they already know the experiments and therefore do not need to attend lab lectures or listen to safety precautions. Offering new experiments will challenge these students.

2. Improvement in student learning.

The group laboratory projects will allow students to go beyond the "cook-book" approach to laboratory. They will be applying theoretical concepts by developing their own experimental procedures to solve problems, and therefore learning will be increased.

3. Lab manuals customized to the Mt. SAC Chemistry Department equipment and facilities.

Directions should be clearer with fewer changes required. Safety precautions and materials disposal directions will be included.

4. Reduced cost to students.

Department-authored lab manuals cost approximately half of what a commercially produced lab manual costs.

5. Easier access to tools needed for data analysis.

Tools needed for data processing will be more readily available. Students can attach their calculators (~75% of students currently have CBL capable calculators) to the CBL interfaces in class, directly acquire data during experiments and then download the data to their own computers at home. The lab manual will contain directions for data analysis using Microsoft Excel, a program readily available to students.

Benefits to the Instructional Program

1. Experiments are customized to our equipment and facilities.

The experiments will reflect the philosophy of the Department. Safety precautions and materials disposal instructions will be written into each experiment. There should be fewer substances disposed of incorrectly.

2. Wider, more comprehensive choice of experiments.

The new lab manuals will provide a wider choice of experiments to instructors. The traditional experiments will still be present along with alternative experiments that will teach the same skills. CBL experiments will replace the Labworks experiments. Group Laboratory Projects that allow students to develop their own procedures and demonstrate mastery of concepts will also be included.

3. Continuation of Awards Program

Revising the lab manuals rather than adopting commercially produced manuals will maintain the current arrangement with the publisher with respect to royalties. The Chemistry Department Awards Program will continue to be funded.

BACKGROUND AND STATEMENT OF PURPOSE

The purpose of this sabbatical project was to update the Chemistry 1A (CHEM 1A) and Chemistry 1B (CHEM 1B) laboratory manuals. These Department authored lab manuals haven't undergone a major revision in several years, even though significant changes have occurred in the Chemistry 1A and 1B laboratory curriculum.

The Mt. San Antonio College Department of Chemistry has been a leader in the use of technology in the student laboratory. In 1991, the Department installed twelve IBM PS 2 computers equipped with Labworks data acquisition systems. Since very little curriculum was available for these systems, Department faculty members developed and wrote programs and procedures for experiments that would utilize the technology. These experiments were eventually combined with other non-technology based ("traditional") experiments in use to produce two Department authored laboratory manuals; Chemistry 1A (CHEM 1A) and Chemistry 1B (CHEM 1B).

The use of the Labworks data acquisition systems dramatically changed the way experiments were performed. Labworks quickly and automatically stored data on a computer disk as it was read. The data could be loaded into the Labworks Spreadsheet program where calculations could be performed on entire columns of numbers. Graphs could be quickly plotted, allowing students to visualize the results of their experiments, often the same day that the experiment was performed. Students were able to focus on the concepts that the experiments were designed to convey, instead of spending hours performing calculations, one number at a time, with a calculator. Computers were not readily available at the time. The Mt. SAC chemistry computer lab was the first hands-on experience with computers for many students.

Ten years is an eon on the technology timeline. Most students now have ready access to computers and sophisticated spreadsheet programs such as Microsoft Excel. Many students have graphing calculators. Two years ago, the College recognized the obsolescence of the Labworks systems and granted the Department the funding to upgrade its technology. Texas Instrument graphing calculators (TI-86) and calculator based laboratory (CBL) data acquisition systems were purchased. Eighteen modern computers became available to the teaching laboratories as equipment in the Department's Molecular Science and Molecular Modeling Learning Center was replaced. The Department was in the same predicament as in 1991; very sophisticated equipment with little available curriculum.

The course material has evolved over the last ten years. Lecture textbooks have changed over time. Some topics that occurred in the same chapter were originally grouped together in experiments. For example, the topics of pH and buffers were covered in the same chapter in 1991. Now these two topics occur in different chapters. It is difficult for students to explore these two topics in an experiment when the material is presented at different times in lecture. The acid-base experiments should be revised to make the topics that they cover consistent with the current configuration in textbooks. Also, the course outline for CHEM 1B has been revised. Material on organic chemistry and biochemistry is no longer covered, but several experiments remain in the lab manual.

The Department has also recently changed its method of handling student laboratory waste. Almost all substances in the laboratories are now collected for disposal. Only a very limited list of approved substances can be disposed of by pouring down the drains or placing in trashcans. Substances used and/or produced, that need to be collected, are assigned tracking numbers. Laboratory waste containers are labeled with these tracking numbers and

placed in the laboratories. Directions for disposing of laboratory wastes need to be inserted into each experiment.

The purpose of this project was to address all of these issues by updating the CHEM 1A and CHEM 1B lab manuals. The experiments that require Labworks technology were rewritten to reflect the new CBL technology. The nontechnology-based experiments were updated to be consistent with the current lecture textbook. New experiments were developed to increase the selection. Guided-inquiry experiments were added to allow students to participate in open-ended activities that require them to apply their knowledge. All experiments now include instructions for proper disposal of laboratory waste.

PROJECT ACTIVITIES

A. GAINING COMPETENCE WITH NEW TECHNOLOGY

The first month of my sabbatical leave was spent becoming familiar with new hardware and software. The starting point was learning the basic functions of the Texas Instruments 86calculator (TI-86). These calculators are extremely powerful, complex devices with more computing capacity than many early personal computers. They go way beyond adding, subtracting, multiplying and dividing. Becoming competent with the TI-86 required going through the tutorial drills in the calculator owner's manual, line by line and keystroke by keystroke, until the function could be performed without looking at references.

Next, the TI-86 had to be interfaced with the Calculator-Based-Laboratory (CBL) data acquisition system. The TI-86 program that runs the CBL system, Vernier's CHEMBIO program, is not part of the calculator's factory programming. The most current version is available on the Texas Instrument Web site. Downloading the CHEMBIO program required the installation of Graphlink software and hardware, which provided an interface between the TI-86 and a computer. Once the logistics of having the TI-86 communicate with the computer and downloading programs into the calculator were worked out, all 32 student TI-86 calculators were programmed with CHEMBIO.

The data acquisition system (TI-86 and CBL interface) was finally ready to use! The system comes with several probes. The thermistor is used to measure temperature. The pH electrode measures acidity and basicity. The colorimeter measures the intensity of specific wavelength of light passing through a solution. The electrical leads measures current or voltage.

Vernier's CHEMBIO program proved to be incredibly user-friendly with well-written directions. The thermistor was selected as the probe to use to gain competence with CBL interface unit. This activity went extremely smoothly. Several experiments were performed with the thermistor. The data acquisition proved to be very dependable and easy to use. Experiments were then performed with the pH electrode, colorimeter and voltage leads. These probes proved to be as reliable and user-friendly as the thermistor.

The publisher of the Department lab manuals, DAY AND NITE, suggested updating the look of the lab manuals by switching from word processing software (Microsoft Word) to desktop publishing software (Adobe Page Maker). Page Maker was installed on my computer. The Word versions of current experiments were imported and reformatted using Page Maker software.

B. REVISING EXISTING LABWORKS EXPERIMENTS

All of the existing experiments written for use with Labworks needed to be revised. Some experiments only used Labworks for analyzing data using the spreadsheet program. These experiments needed to have directions written for data analysis using the Microsoft Excel spreadsheet program. Some experiments used both the Labworks interface connected to probes for data acquisition and the spreadsheet program for data analysis. These experiments needed to have procedures developed and directions written using the CBL systems to collect data as well as new instructions for data analysis using Excel.

The basic format of the experiments was not changed. Each experiment is presented in six sections: Purpose, Introduction, Materials, Procedure, Data Analysis and Questions. The experiments reflect the new method of collecting laboratory waste. The Materials section now lists laboratory waste containers with tracking numbers specific to the experiment. The Procedure section includes directions for disposing of laboratory waste into the correct laboratory waste containers.

Following is a list of experiments that were written for use with Labworks and the revisions that were made.

Revised CHEM 1A Labworks Experiments:

a. Introduction to Computers and the Labworks Spreadsheet

The original experiment had two goals. One goal was to teach students how to use the Labworks spreadsheet program. This part of the experiment consisted of three exercises that required students to input data into the spreadsheet, perform calculations on the data and plot graphs of the data. The lists of data were supplied to the student as a handout in class.

The second goal was to acquaint students with the Labworks interface. This part of the experiment provided instructions for the student to connect the thermistor probe to the Labworks interface and collect data, monitoring the temperature of hot and cold water. The emphasis of the experiment was on the spreadsheet program.

The revised experiment has only one goal: to teach students to use Excel to analyze data. The new CBL data acquisition is completely separate from the Excel spreadsheet used for data processing. A new experiment, "Introduction to the Calculator Based Laboratory System", has been developed to teach students to use the CBL system.

The revised experiment uses the same data lists as in the original experiment. The data lists are included in the Procedure section of the lab manual instead of being supplied as a student handout. The new Procedure provides detailed instructions on inputting data, performing calculations, plotting graphs, and adding trendlines using Microsoft Excel. The new experiment uses the same method of instruction as in the original experiment. The Procedure is written as a step by step tutorial, providing many illustrations of what the computer screen should look like. See Appendix A for a copy of the experiment.

b. Properties of Pennies

The Introduction section of this experiment was rewritten to provide interesting background information about pennies. The original experiment required students to input measurements made of the mass, diameter and height of pennies into the Labworks spreadsheet program. The Data Analysis section provided instructions for using the Labworks spreadsheet program to perform calculations and plot graphs. The experiment has been revised so that Data Analysis instructions are written for use with Excel.

c. Computer Programming for Experimental Data Acquisition

The goal of this experiment was for students to write a program that directed the Labworks interface panel to measure temperature with the thermistor. The original Labworks systems had very limited programs available for methods of collecting data. Programs had to be written for almost every experiment that was developed. Fortunately, Labworks provided a very user-friendly method of writing programs, so an experiment was developed to provide students with an opportunity to actually write and use a program. The CHEMBIO program has wonderful choices for methods of collecting data but almost no capacity to write new programs. Therefore, the program writing activity has been eliminated from the CHEM 1A curriculum.

d. Energy Changes

The original experiment included 3 Parts: Conservation of Energy, Heat of Neutralization and Enthalpy of Reaction. All of the data was collected with Labworks interfaces and then analyzed using the Labworks spreadsheet program.

The revised version of the experiment includes four parts: Conservation of Energy, Heat of Neutralization with HCl, Heat of Neutralization with HCl and H₂SO₄ and Enthalpy of Reaction. Heat of Neutralization has been split into two parts (Heat of Neutralization with HCl and Heat of Neutralization with HCl and H₂SO₄). These two new parts together require approximately half of the original amounts of reagents as in the original experiment. The Procedure section has been rewritten to provide instructions for using the CBL system for data collection. The Data Analysis section has been revised to provide instructions written for Excel.

e. *Phase Changes*

The data collected in this experiment proved to be very unreliable. As a result, it has currently been dropped from the curriculum.

Revised CHEM 1B Labworks experiments:

a. *Reaction Kinetics*

This experiment used phenolphthalein to follow the hydrolysis of tert-butyl bromide. Tert-butyl bromide is an organic compound that raises many safety concerns. Therefore, this experiment has been dropped from the curriculum. It has been replaced by new kinetics experiments: “Kinetics of the Crystal Violet Reaction Using Initial Rates of Reaction”, “Kinetics of the Crystal Violet Reaction Using Integrated Rate Laws” and “Chemical Kinetics”.

b. *Determination of K_{eq}*

The original Data Analysis section required students to input Spectronic 20 measurements made from equilibrium solutions into the Labworks spreadsheet program. The Data Analysis section provided instructions for using the Labworks spreadsheet program to perform calculations and plot graphs. The experiment has been revised so that Data Analysis instructions are written for use of the Excel spreadsheet program.

c. *pH and Buffers*

At the time this experiment was written the topics of pH and buffers occurred in the same chapter of the textbook adopted for CHEM 1B. These two topics occur in different chapters in the current textbook. The topic of hydrolysis, originally included in the experiment “Acid Base Phenomenon” occurs in the same chapter as pH in the current

textbook. The topic of buffers occurs in a later chapter. The original experiment has been revised to include the topics of pH and hydrolysis. A new experiment, "Buffers", has been developed. (see new experiments)

The Introduction section of the original experiment has been rewritten to include the topic of hydrolysis as well as pH. The directions for Hydrolysis have been transferred from the Procedure section of "Acid Base Phenomenon" to this experiment.

The Procedure section was originally written with instructions for collecting data using the Labworks interfaces. These instructions have been rewritten for using the CBL systems for data collection.

The Data Analysis section provided instructions for using the Labworks spreadsheet program to perform calculations and plot graphs. The experiment has been revised so that Data Analysis instructions are written for use with Excel.

d. Acid Base Phenomenon

The original experiment covered the topics of acid-base titration curves and hydrolysis. The topic of hydrolysis has been removed, as explained above. The experiment now only covers the topic of acid-base titration curves and has been renamed "Titrating a Weak Acid with a Strong Base".

The Procedure section was originally written with instructions for collecting data using the Labworks interfaces. These instructions have been rewritten for using the CBL systems for data collection. The Data Analysis section provided instructions for using the Labworks spreadsheet program to perform calculations and plot graphs. The experiment has been revised so that Data Analysis instructions are written for use with Excel. See Appendix A for a copy of the experiment.

e. *Electrochemistry*

This experiment was originally designed as a collaborative, guided-inquiry experiment where students designed and built a galvanic cell. Students wrote a Labworks program to instruct the interface to measure voltage.

The new experiment has changed very little from the original experiment. The directions to write a Labworks program have been removed from the Procedure section. Directions for using the CBL system to measure voltage have been included in the Procedure section.

f. *Nuclear Decay*

This experiment was based on Labworks simulation capability. Databases were developed to simulate the decomposition of several radioactive substances. These databases were entered into the simulation mode of the Labworks program. Students accessed these databases in the form of a real time simulation of nuclear decay.

C. WRITING NEW TECHNOLOGY-BASED EXPERIMENTS

New CBL experiments were written for both courses. Some of the new experiments allow students to explore concepts not previously included in the lab curriculum. Other new experiments replace ones that didn't always work well or used reagents that raised safety issues. The basic format of the new experiments follows that of the old format. Each experiment is presented in six sections: Purpose, Introduction, Materials, Procedure, Data Analysis and Questions. The experiments include the new method of collecting laboratory waste. The Materials section lists laboratory waste containers with tracking numbers specific to the experiment. The Procedure section includes directions for disposing of laboratory waste into the correct laboratory waste containers. The Procedure sections is written with directions for CBL systems. The Data Analysis section is written with directions for Excel.

Following is a list of the new CBL experiments developed. Each listing includes a description of the goal and a brief description of the experiment. The source of the experiment is also included, if it is not a completely original experiment.

New CHEM 1A Technology-Based Experiments

a. Introduction to the Calculator Based Laboratory System

This experiment was developed to teach students how to use the CBL system, including how to transfer data saved on the calculator during an experiment to a computer equipped with Excel. The experimental procedure has students connect all parts of the system together, using the thermistor as the probe. Instructions are given in a step by step, tutorial manner. A series of measurements of hot and cold water are made, which are

automatically stored on by the TI-86. Instructions are then given to rename the data file and to transfer it to Excel. See Appendix B for a copy of the experiment

b. CheR₂Y Solutions

This experiment is designed to teach students to use the CBL colorimeter, to prepare solutions and to prepare and use a standard curve. The colorimeter measures the intensity of a specific wavelength of light passing through a solution. A search was made to select a solution that was readily available, absorbed light within the wavelength capacity of the colorimeter and could be disposed of by pouring it down the drain. The light-absorbing solution chosen was cherry Kool-Aid.

A solution was prepared by pouring the entire contents of a cherry Kool-Aid envelope into a 2 liter container, adding water (no sugar!) to the calibration mark and stirring until the solid all dissolved. Seven serial dilutions were made. The absorbance of the solutions were read using all three wavelengths of the colorimeter. It was determined that the solution absorbed the most light at 565 nm. The concentration of the solution versus the absorbance was plotted, using Excel. The first five points on the graph appeared linear. This provided the linear limits of the colorimeter. The student procedure was based on diluting solutions and preparing unknowns within the linear range of the colorimeter. See Appendix B for a copy of the experiment. See Appendix E for an example of data collected during development.

New CHEM 1B Technology-Based Experiments

a. Kinetics of the Crystal Violet Using Initial Rates of Reaction

The concepts included in the study of kinetics are very difficult to comprehend. This experiment is designed to illustrate the concept of determining the order of a reactant in a reaction using the method of initial rates of reaction. A reaction is performed over a very short period of time and the initial rate of reaction is determined. The procedure is repeated, doubling the concentration of one of the reactants. The rates for both experiments are compared to determine the effect that doubling the concentration of a reactant has on the rate of reaction. This concept is new to the CHEM 1B laboratory curriculum.

The reaction between crystal violet and sodium hydroxide was selected. The book "Chemistry with CBL"¹, purchased with the CBL systems, contained a version of a kinetics experiment with crystal violet. This experiment was used as the basis for the "Kinetics of the Crystal Violet Using Initial Rates of Reaction" experiment and for the "Kinetics of the Crystal Violet Using Integrated Rate" experiment.

It was decided to vary the concentration of the sodium hydroxide and keep the concentration of crystal violet constant. A series of dilutions of sodium hydroxide were prepared, ranging from 0.025 M to 2.00 M. The methodology was worked out using the CBL colorimeter and a series of experiments were performed. The initial rates were determined by plotting absorbance versus time and calculating the slope of the line. It was determined that the best results occurred when 0.50 M and 1.0 M sodium hydroxide were used. The reaction was determined to be first order with respect to sodium hydroxide. See Appendix B for a copy of the experiment. See Appendix E for examples of data collected during development.

b. Kinetics of the Crystal Violet Reaction Using Integrated Rate Laws

In this version of the crystal violet reaction, the reaction proceeds and data is collected for five minutes. Three different graphs are plotted of the data: concentration versus time (zero order), natural log of concentration versus time (first order) and inverse of concentration versus time (second order). Linear regression trendlines are added to the graphs and the most linear plot is selected to determine the order. The experiment is repeated at three different temperatures. The data from all three experiments are processed to determine the energy of activation for the crystal violet reaction.

The order of reaction determined from this experiment is with respect to crystal violet, since it is the species that is monitored by the colorimeter. The procedure was carried out over a wide range of temperatures to determine what would work for student use. Very low temperatures (below 15°C) resulted in condensation on the cuvettes. High temperatures (above 30°C) resulted in the experiment proceeding too fast for the colorimeter to accurately monitor. The methodology was worked out and many trials of the procedure performed. The reaction was determined to be first order with respect to crystal violet.

This experiment replaces "Reaction Kinetics". The same concepts are covered in both experiments. The new experiment requires only two lab periods, as opposed to three to four lab periods for the original experiment. Crystal violet and sodium hydroxide can be used safely as disposed of by pouring down the drain. The t-butyl bromide cannot easily be purchased for student use any more, due to safety concerns. The crystal violet reaction is extremely reproducible. The t-butyl bromide reaction proved to be notoriously undependable from year to year. See Appendix B for a copy of the experiment. See Appendix E for examples of data collected during development.

c. Chemical Kinetics

This experiment is based on “the Reaction of a Food Colorant with Sodium Hydroxide”². This experiment is written as a collaborative, guided – inquiry experiment, using CBL systems for data collection. Dr. Iraj Behbahani prepared a handout of the experiment for use in CHEM 1B classes the semester before my sabbatical leave. The handout and feedback from instructors, who used the experiment were used to prepare the version that appears in the current lab manual.

This experiment requires students to work in groups and write their own experimental protocol, based on a scenario or a series of guiding questions. Students must have a clear understanding of the topics of kinetics, the reaction being studied and the use of the CBL system to successfully complete the experiment. . See Appendix B for a copy of the experiment. See Appendix E for data collected during development. See Appendix F for a copy of the original JCE experiment

d. Buffers

Understanding how buffer solutions work is very complex, requiring application of a complex formula called the Henderson – Hasselbach equation. Students seem to get hung up on using the equation without really understanding how a buffer solution is prepared or really works. The first part of this experiment is based on the “Buffers” portion of “pH and buffers” from the original lab manual. Students follow instructions to prepare a buffer. The effects of adding acid and base on both the buffer and water are compared. The relevant sections of that “Acid-Base Phenomenon” have been transferred to this experiment. The second part of the experiment, the students prepare buffer at a specified concentration and molarity. The students must create their own instructions to prepare the buffer, including

calculating the amounts needed of each substance. The students must then calculate the amount of acid and base needed to change the pH of the buffer by a specified amount. The success of their calculations is then measured by carrying out the additions of acid and base to portions of their buffer solution. The CBL system is used to measure all pH values. See Appendix B for a copy of the experiment.

D. WRITING NEW NONTECHNOLOGY – BASED EXPERIMENTS

The new nontechnology-based experiments are mostly guided-inquiry experiments that require students to create their own protocol for carrying out the experiment. This requires students to go beyond the “cookbook” method of performing experiments where they follow a procedure to arrive at a predetermined answer. Guided-inquire experiments require students to apply the concepts that they are supposed to be learning. Guided-inquire experiments developed follow two possible forms: students answer a series of guiding questions to develop a protocol or a problem is presented as a scenario and the students must respond to the scenario

Following is a list of new nontechnology-based experiments.

New CHEM 1A Nontechnology Experiments

a. The 10 mL Transfer

This experiment is written as a collaborative, guided – inquiry experiment. Students work in groups and write their own experimental protocols, based on the scenario of determining the best instrument to transfer exactly 10 mL of water. Students must have a basic understanding of the scientific method and density to successfully perform this experiment. Basic lab skills such as using a graduated cylinder, pipetting and making observations are reviewed. See Appendix C for a copy of the experiment.

b. Normality of an Unknown Aqueous Acid

This experiment is designed as an alternative to “Determining the Equivalent Mass of a Solid Acid”. Students work individually to develop their own protocols to determine the

normality of an unknown, aqueous acid. Student must understand the concepts of equivalents, normality, and volumetric analysis to successfully perform this experiment.

New CHEM 1B Nontechnology Experiments

a. Analysis of a Sodium Chloride Solution

This experiment is written as a collaborative, guided – inquiry experiment. Students work in groups to develop a protocol to prepare and analyze a solution. The scenario presented requires the students to submit their protocol and results in an attempt win a contract to prepare and analyze solutions. Students must have an understanding of chemical properties, colligative properties and solution preparation to successfully complete this experiment. Basic lab skills such as are weighing, using a graduated cylinder, using a volumetric flask, maintaining a lab notebook are reviewed. See Appendix C for a copy of the experiment.

b. An Equilibrium Activity

Equilibrium involves understanding the concept that forward and reverse reactions are occurring at the same time, even though no visible changes may be noticeable. This experiment is based on “Equilibrium: A Teaching/Learning Activity”³. It is designed to provide a visual, hands-on simulation that demonstrates the concept of equilibrium at the beginning of discussion of the topic. Different colored beads are used to simulate reactants and products in a hypothetical reaction. Students are designated as either products (P), reactants (R) or recorder. Products and reactants are given a predetermined number of beads to begin. Instructions are given to exchange a number of beads at exactly the same time. The recorder notes the total number of beads that P and R have after each exchange. The result of each

activity is that, eventually, the number of beads possessed by P and R will remain constant, even though they are still exchanging beads.

c. *Chemical Tangerine*

Versions of this experiment, under the title “Optimum Reaction Conditions”⁴ have been around the Department for at least twelve years. I have tried several versions with my classes over the years and have never had good results. The experiment is based on the reaction that occurs between acetic anhydride and ferrocene (yellow) in the presence of heat and acid to produce two colored products (orange and tan). The amount of ferrocene, time of heating, type of acid and amount of acid can all be altered to determine the optimum reaction conditions to yield the most orange product with the least amount of tan product and unreacted yellow ferrocene. Most students ended up with unreacted (yellow) ferrocene and no orange or tan products.

My daughter chose to try to work the bugs out of this experiment for her high school science project last year. She used a much wider scope of conditions than in the versions I have tried. She did end up with sets of conditions that yielded significant quantities of orange and tan products.

I combined the last version of the experiment that I had tried along with her procedure to develop a guided-inquiry scenario experiment. Students must determine a method of efficiently producing a dye called “Chemical Tangerine” for the fashion industry. They are only allowed to try eight different combinations of reactants to find the optimum conditions that will produce the orange-colored dye. Students must have an understanding of basic experiment design to successfully complete this experiment. See Appendix C for a copy of the experiment. See appendix F for a copy of the original handout.

E. REVISING TRADITIONAL EXPERIMENTS

Many experiments in the current lab manuals are not technology (computer) based. The concepts that they teach, such as titration, observations measurements, are very traditional part of the curriculum. Some just needed minor updating and editing. Others underwent more extensive revisions, such as having examples added or being split into two separate experiments. All existing experiments were revised to include directions for proper disposal of laboratory waste.

Following is a list of the nontechnology-based experiments that were revised. The types of revision are included.

Revised CHEM 1A Traditional Experiments

a. Introduction to the Chemical Laboratory

This experiment was originally developed to acquaint students fresh from high school or CHEM 2A with the CHEM 1A laboratory. It had many different parts, required two to three lab periods to complete and was very difficult for students to write a report for. Since students at this point (beginning) of the semester are new to the art of writing lab reports, the reports were extremely difficult to grade. The result was that students frequently didn't receive feedback on their lab reports until they had turned in at least one additional report.

The experiment had been split into two manageable experiments: "Measurements" and "Physical and Chemical Properties". The appropriate parts of the original experiment were cut and pasted into the two new experiments. New material was written to create two complete experiments.

b. *Stoichiometry*

This experiment underwent very minor editing.

c. *Neutralization Reaction*

This experiment was split into two experiments: “Standardization of a Sodium Hydroxide Solution” and “Determination of the Equivalent Mass of an Unknown Solid Acid”. The existing material was cut and pasted into the two new experiments. New material was written to obtain two complete experiments.

d. *Properties of Gases*

This experiment underwent very minor editing.

e. *Molecular Bonding and Geometry*

The introduction of this experiment was revised to include examples of most of the concepts covered by the experiment. The tables that were in the original experiment were updated to a more professional look. See Appendix D for copies of the original and new versions of the experiment.

f. *Analysis of a Salt Mixture*

This experiment underwent very minor editing.

g. *Quantitative precipitation of PbCrO₄*

The waste produced in this experiment is very expensive to dispose of. No teacher has used this experiment for several years. It has been removed from the curriculum.

h. *Equivalent Weight of an Unknown Reducing Agent*

This experiment illustrates the same concepts as “Standardization of a Sodium Hydroxide Solution” and “Determination of the Equivalent Mass of an Unknown Solid

Acid”. “Equivalent Weight of an Unknown Reducing Agent” underwent minor revision, but was moved forward in the manual to follow these two experiments.

Revised CHEM 1B Traditional Experiments

a. *Principles of Equilibrium*

This experiment underwent minor revision. One part was removed since it was redundant with the section that followed. See Appendix D for copies of the original and new versions of this experiment.

b. *Qualitative Analysis of Group 1 Cations*

This experiment underwent minor revision. The flow chart was created with a computer. The boxes in the previous version were hand-drawn.

c. *Qualitative Analysis: A Computer Simulation*

This experiment underwent very minor revision.

d. *Oxidation Reduction Reactions*

This experiment was very long with many different parts. It was difficult for students to record data and write a report. It was difficult for the instructors to grade the reports. Therefore, the experiment was split into two separate experiments: “Oxidation-Reduction Reactions and Activity Series” and “Oxidation-Reduction Reactions”. The first experiment was moved to the CHEM 1A manual as it correlated with material earlier in the lecture textbook. The second experiment remains in the CHEM 1B manual. It is more streamlined and should be easier for students and instructors to manage.

- e. *Organic Nomenclature and Molecular Structure, Properties of Hydrocarbons, Reactions of Organic Compounds, Synthesis and Analysis of Aspirin, Polymerization*

The course outline was revised for CHEM 1B several years ago. These experiments are now longer consistent with the course material and have been removed from the curriculum.

CONCLUSION

I have achieved the goals that I set when I began this project. I was able to keep to the timeline I set for myself. I received continuous input from my colleagues as the experiments were developed. Several colleagues were excited enough about the new equipment that they volunteered to field test experiments before the lab manuals were even finished. The completed CHEM 1A and CHEM 1B lab manuals truly reflect what I envisioned when I proposed this project.

Personal Benefits

The most obvious benefit to me is the relaxation and reinvigoration that occurred during my sabbatical leave. The change of pace was wonderful, leaving me refreshed and eager to return to the classroom (to try out these new experiments)!

The second most obvious benefit is that I have become comfortable with the use of a TI-86 graphing calculator. More than half of the students in my current CHEM 1B classes have some form of graphing calculator. I know the power of these calculators and have found that most students do not know how to use most of the functions. I am now able to point out features that will make problem solving easier for students. I encourage students to bring the handbooks to class to learn how to use some of the graphing functions.

Another benefit is that I really know how to use the CBL systems. I can quickly detect where the problem lies when a student is having difficulty with the system.

One benefit that I am seeing as the semester progresses is that the experiments are working as intended. My CHEM 1B classes have now performed four of the new experiments. "Analysis of a Sodium Chloride Solution", a guided-inquiry group activity, had students working together, discussing chemistry on the first day of class! The two crystal

violet experiments went smoothly, with very few technical glitches. I have found students to be much more concerned about disposing of laboratory materials, quickly calling my attention to full waste containers, and cautioning other students to wait until the containers have been emptied.

Benefits to the Students

The immediate benefit to the students is that they are no longer crammed into a small room with twelve computer systems when they perform a technology-based experiment. The Department purchased thirty-two systems, enough so that two classes can perform technology-based experiments, in their own classroom, at the same time, with no shortage of equipment.

Also, data processing is performed with Excel, which, unlike Labworks spreadsheet, is readily available to students. Students can even use their own calculator to run the CBL system and collect the data. They can download the data directly into their own computers at home, if they wish. The Department has an adequate number of computers available for students to download their data from Department calculators during class time. The students store the data on their own disks and process the data at their convenience.

The advantage of the guided-inquiry experiments is that students must actually apply concepts in order to write experimental protocols. They must be able to design a procedure that will collect the data needed to solve the problem. If they don't really understand a concept, they are forced to review it to successfully complete the experiment.

Another benefit to the students is that the experiments are written specifically for the equipment at Mt. SAC. It is not necessary for students to write down a list of corrections to the procedure. This is especially true regarding the disposal of laboratory waste.

The lab manuals are still published by Day and Night. This keeps the price of the Department lab manuals to approximately half of what a commercially produced lab manual costs.

Benefits to the Instructional Program

The largest benefit to the Instructional Program is that the experiments reflect the philosophy of the department. I continually sought input from colleagues regarding how experiments should be developed or revised. Copies of experiments were circulated to Department members for feedback.

The laboratory curriculum should run smoothly and safely, since the experiments are specific to Mt. SAC facilities and the new equipment. Experiments that raise safety issues have been eliminated or replaced by safer choices. Safety precautions and the new materials disposal instructions are written directly into each experiment eliminating the need to give corrections to the procedure at the beginning of class.

The selection of experiments has increased (see appendix G). There are alternative experiments for several topics covered, especially those at the beginning of the semester. Students repeating the course should not be performing exactly the same experiments.

The Chemistry Department will still be regarded as a leader in the innovative use of technology in the classroom.

Appendix A

Examples of Revised Labworks Experiments

CHEM 1A

Original – Introduction to Computers and the Labworks Spreadsheet

New – Introduction to Spreadsheets

CHEM 1B

Original – Acid/Base Phenomena

New – Titrating a Weak Acid with A Strong Base

#3 - Introduction to Computers and the Labworks Spreadsheet

PURPOSE:

These hands-on exercises provide an opportunity to become familiar with the use of the Labworks Spreadsheet software which is available in the Mt. San Antonio College Chemistry Computer Lab and the Natural Science Computer Lab.

This experiment is designed to introduce the student to the Labworks hardware and software through some simple and practical wet and dry chemical exercises.

DISCUSSION:

The computer hardware used in the Chemistry 1A/1B classes at Mt. San Antonio College are IBM PS/2 - Model 30 computers. These computers include a central processing unit (CPU), an enhanced keyboard, a video graphics analyzer (VGA) monitor, and two 3 1/2" disk drives. Connected to each computer is an interface panel from SCI Technologies, which allows the student to acquire experimental data through the use of measurement-specific sensors. **Care should be taken to prevent the spilling of chemicals onto any of the hardware in the chemistry computer laboratory. Report any such spills immediately to the instructor.**

The computer software used in the Chemistry 1A or 1B classes will vary depending upon the needs of the student. Computer simulations and tutorials are available for all students, whenever time permits, covering a range of chemistry topics: scientific notation, significant figures, dimensional analysis, the periodic table, the mole concept, nomenclature, stoichiometry, oxidation-reduction reaction, equilibrium, aqueous solutions, qualitative analysis, and organic chemistry. The software which drives the lab interface panel, **Labworks**, will be the software of choice during laboratory experiments. Labworks will be used in three primary modes: writing programs to use for data acquisition during experiments, the actual experimental data acquisition, and data analysis (spreadsheet and graphical) after the experiment is completed.

Computer spreadsheet programs (software) have become very popular as a means to organize and manipulate large quantities of data. The data (actual numbers) are entered into columns of the spreadsheet, and then the program can perform computations on entire columns of numbers very quickly. Spreadsheet programs are well suited for use in chemistry classes because they can reduce the amount of time spent in the repetitive and lengthy chemical calculations required. If the spreadsheet software also contains a graphing option (Labworks does contain this option), large amounts of data can be displayed graphically in several different formats with the addition of just a few keystrokes.

The Labworks Spreadsheet is very user friendly! It is menu-driven, which means that the options available for the student to choose will always be displayed on the monitor screen. Helpful messages concerning each menu selection also appear on the monitor whenever a

selection is highlighted. It is not necessary to know a computer language or have had prior experience with a computer to be successful with the Labworks software.

The software that controls the operation of the computer and manages the data files is called the **Disk Operating System**, or **DOS**. Students will have limited exposure to DOS in the chemistry computer laboratory, but it is an important tool for utilizing the various computer software, including Labworks.

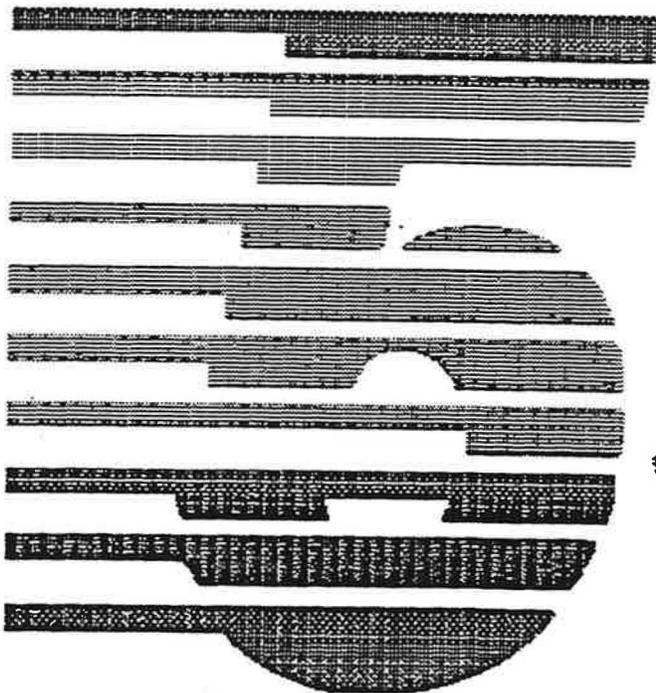
The exercises that follow are meant to serve as an introduction to the Labworks Spreadsheet. Basic operations such as data acquisition, data analysis and graphing will be covered. Students should feel free to explore the choices available until they are comfortable with the software.

Entering the Labworks Spreadsheet

The chemistry instructor will turn on the computers, monitors and printers in the Chemistry Department's Computer Laboratory. **DO NOT TURN ON THE COMPUTERS OR MONITORS BY YOURSELF!** Be sure to read the Computer and Diskette Rules in the General Information Section of the Chemistry Lab Manual.

A. Network Menu

When the computers in the Chemistry Department's Computer Lab or in the Natural Science Computer Lab (they are all networked together) are initially started up (booted up), the opening screen will look like this:



FIFTH GENERATION SYSTEMS

presents

Digital Access Network

Consult the Supervisor's Manual
for instructions on placing
your company's logo here!

Press any key...

To enter the network's main menu, press any key. The main menu has many choices:

```

Welcome to the Natural Science Network
Chemistry Lab - Main Menu

Good Morning...Please Enter Your Selection

A) LabWorks 3.2
B) KC? Discoverer
C) Inorganic Qual Analysis
D) Organic Nomenclature
E) WChem
F) DOS Functions
G) Anti-Virus
H) Select Network Printer

Direct Access Network Version 5.1

F1 Help

Monday May 20, 1996 9:42:33 am
```

B. Selecting Menu Options

Menu options are selected by highlighting an option and then pressing the Enter key. To highlight a menu option, use the Arrow keys (darker in color) located between the alphabet keys and number keys, on the lower right side of the computer keyboard. Follow this procedure whenever there are instructions to select a menu option.

C. Selecting a Printer

If you are planning to print anything from your use of the spreadsheet, you must select a printer first. Highlight the **Select Printer** option from the network main menu, and then select the letter of the printer closest to your computer. Return to the network main menu. Now you are ready to enter Labworks.

D. Entering LABWORKS

On the network main menu, select the **Labworks 3.2** option. Press any key. The Labworks title screen will appear:

```

L a b W o r k s
Version 3.2

Copyright (C) 1993 SCI Technologies, Inc.
All Rights Reserved
```

Press any key to begin

The Labworks main menu can be obtained by pressing any key:

- CALIBRATE SENSORS
- BUILD/EDIT EXPERIMENT
- PERFORM EXPERIMENT
- SPREADSHEET
- SETTINGS
- DOS SHELL
- DELETE FILES
- EXIT

Calibrate sensors such as pH probes, thermistors, etc.

Using the Labworks Spreadsheet

Select **SPREADSHEET** from the Labworks main menu. The Spreadsheet entrance screen will appear:

Welcome to the LabWorks
spreadsheet for the laboratory

Copyright (c) 1993 SCI Technologies, Inc.
All Rights Reserved

Press any key to move to an actual spreadsheet display. The Labworks Spreadsheet is arranged with a menu bar across the top and two sets of columns below. Again, the arrow keys on the computer keyboard are used to highlight and select menu options. Located at the bottom of the screen are some additional options which may be selected by using the "F" keys (located across the top of the keyboard).

UNTITLED								
Column	Setup	Data	Analysis	Graph	Stats	Print	File	Quit
	Data Acquisition				Data Analysis			EXIT
No.	A	B	C	D	E	F	G	

Exit to DOS
F1 F2 Edit F3 F4 Grid F5Graph F6D Row F7I Row F8 F9 Help 10 Quit

Data can be entered into columns A through D, which are designated Data Acquisition. Data can be either entered manually using the keyboard, acquired during an experiment through the Interface Panel, or recalled from a stored file.

Columns E through G are titled Data Analysis and are used for math functions and operations such as performing the log or square root. The selected function for the data analysis column will compute that function on an entire column of data (in column A, B, C, or D).

A. Labeling and Formatting Columns

Columns must first be labeled and formatted before data is entered. To do this, select **Column Setup** from the menu bar. The following options will appear under column A:

UNTITLED							
Column Setup	Data	Analysis	Graph	Stats	Print	File	Quit
Data Acquisition				Data Analysis			
No.	A	B	C	D	E	F	G
	Format Label Initialize/Delete						

1. Format

Format allows data to be displayed in either decimal or scientific notation. Very large and very small numbers should be displayed in scientific notation. **Format** will also allow the user to choose how many places past the decimal point are needed.

2. Label

Use the **Label** option to title a column. Letters and symbols can be used in titles. All data columns must be titled.

3. Initialize/Delete

The **Initialize/Delete** option will turn a column on and off. Columns that are needed for data should be initialized. When this option is chosen, a row of zeroes will appear in that column. The **Delete** option should be used for columns that are not needed.

When all of the choices have been made for a single column, use the right arrow key to go on to the next column. Continue in this manner to the right, setting up all columns that are needed to accommodate the data that will be entered. Choose **Delete** for any column that is not needed. The spreadsheet is now ready to accept data.

B. Entering Data into the Spreadsheet

Choose the **DATA** option to type in data from the keyboard. The zeroes in column A will be highlighted. Type in the first number using either set of number keys: the line of numbers across the top of the keyboard or the block of numbers on the lower right of the keyboard. (Be sure that the Number Lock [**Num Lock**] key is pressed to use the lower right number keys.) Press the enter key when each number is completed.

If more than one column was initialized, the zeroes in the next column will now be highlighted. If only one column was initialized, the highlight will move down to the next row in the same column. Continue data entry until the last number is reached and press the escape key [**ESC**] (upper left side of keyboard). **DO NOT PRESS THE ENTER KEY AFTER THE LAST NUMBER HAS BEEN TYPED IN!** This will cause the highlight to move to another row of data. If this should accidentally occur, press the F6 key to erase the unneeded row.

C. Editing Data

Use the **Data** option on the menu bar to re-enter any data column and edit (change) the original data. Use the arrow keys to move to the desired column and row of data. Simply type in the new data and press Enter. Use the Esc key to exit the data column.

D. Analyzing Data

Once all of the data has been entered into the spreadsheet, the **Analysis** option can be used to perform computations. Selecting **Analysis** from the menu bar will shift the highlight to column E and the following options will appear:

UNTITLED							
Column Setup	Data	Analysis	Graph	Stats	Print	File	Quit
	Data Acquisition				Data Analysis		
No.	A	B	C	D	E	F	G
					Select function Unused Recompute column Format		

1. Select Function

Choosing **Select Function** will present the functions and operators available to carry out calculations in the Labworks Spreadsheet.

It is necessary to determine the exact formula needed for the calculation that will be carried out. The formulas must be typed in correctly for the computer to carry out the calculation.

List of Functions and Operations

Abbreviation	Definition
+	addition
-	subtraction
*	multiplication
/	division
^ or **	exponent
ABS	absolute value
ARCTAN	inverse tangent
COS	cosine
DELTA	the difference between sequential data points in a column
DERIV	derivative
EXP	exponential
FACT	factorial
INTEG	integral
LN	natural log (base e)
LOG	logarithm (base 10)
SIN	sine
SQR	square the number (x^2)
SQRT	square root of the number
STEPPD	move down data points in the column
STEPUP	move up data points in the column
SUM	cumulative sum of the column

The letter of the column which will have the function performed on it must be enclosed in parentheses. For example, to calculate the square root of all of the values in column A, type in **SQRT(A)** with no spaces. The square root of each value in column A will appear in the corresponding row of the analysis column. The formula will appear as the label of the analysis column.

It is possible to label an analysis column by using the square brackets []. Information inside of these brackets is interpreted as a message, not as a function. Before typing the formula, type in the column label enclosed in square brackets. Whatever is in the square brackets will appear as the column label, and will also appear as the axis label if that column is later used for graphing.

2. Unused

This option informs the computer that no computation will be

C. Titling the Graph

Use an appropriate and descriptive title for each graph. The printers will be accepting print commands from many computers, and untitled graphs are difficult to distinguish from each other.

UNTITLED							
Column Setup	Data	Analysis	Graph	Stats	Print	File	Quit
	Data Acquisition				Data Analysis		
No.	A	B	C	D	E	F	G
Enter a graph title (make it descriptive):							

D. Plotting the Graph

To display the graph, select **Plot**. The following types of plots can be generated:

1. **Scatter Graph** - data points will appear individually with no line connecting them
2. **Line Graph** - data points connected sequentially by a line
3. **Line Graph, No Points** - line graph with no points on it
4. **Bar Graph** - bars extending up from the x axis (takes a long time to generate)
5. **Linear Regression** - calculates least squares equation and displays a linear equation ($y = mx + b$) which reflects the data, along with a correlation coefficient

Printing the Spreadsheet or Graph

One copy of the spreadsheet or graph may be printed using the **Print** option. Use a photocopier to make additional copies. If a printed copy of the image on the screen is all that is desired, press the **Print Screen** key on the upper right of the keyboard for a single printed copy. A spreadsheet with many rows of data should not be printed.

UNTITLED							
Column Setup	Data	Analysis	Graph	Stats	Print	File	Quit
	Data Acquisition						
No.	A	B	C	D	E	F	G
					Print data table Print graph Print data table and graph		

Exercise care when tearing off the printout from the continuous roll of computer paper exiting the printer. Be sure the entire printout is out of the printer, and that the paper is not pulled obliquely (not

Exercise care when tearing off the printout from the continuous roll of computer paper exiting the printer. Be sure the entire printout is out of the printer, and that the paper is not pulled obliquely (not evenly) from the printer. Improper tear-offs can result in severe printer jamming, and extended down-time for the printers. If jamming occurs, please inform the instructor and let the instructor clear the jam. Also, do not advance the paper in order to tear-off the printout, as this will cause future printouts to print undesirably across the perforations.

Saving or Retrieving a Data File

UNTITLED						
Column Setup	Data	Analysis	Graph	Stats	Print	File Quit
	Data Acquisition				D	Load data file Save data file Clear Spreadsheet
No.	A	B	C	D	E	

A. Saving Data

To save the data of a spreadsheet or graph, use **Save Data File** under the menu bar's **File** option. The storage location, as well as a name (maximum of 8 letters) for the data file, must be entered in order for the file to be saved properly. For example, to save Trial One data to the student's data disk stored in the right-hand disk drive (drive b:) of the computer, enter **b:trialone**. It is a good idea to record the name of the data file in the lab notebook at the same time.

B. Retrieving Data

Use **Load Data File** under the menu bar's **File** option to retrieve a data file from a computer diskette or from the computer network. The correct location and name of the file must be entered, for retrieval to occur. Incorrectly typed locations or file names will result in the error message "File not found".

Exiting Labworks

Select the **QUIT** option from the menu bar to exit Labworks. If necessary, remove the student diskette from the right-hand disk drive of the computer. **DO NOT TURN OFF THE COMPUTER OR MONITOR.** The instructor will do this for all students.

MATERIALS:

Pure solids
ice
Pure liquid
hot water
Stockroom equipment
IBM PS/2 Model 30 computer
thermistor
labworks/interface panel
Locker equipment
student disk
thermometer
2 beakers

PROCEDURE:

Part 1: Computer Worksheet

1. Complete computer worksheet and turn in to instructor.

Part 2: Spreadsheet Analysis **NOTE: network = f**

1. The instructor will turn on the electrical power to the computer and the Labworks interface panel, and will log the computer onto the network. **DO NOT FLIP THE POWER SWITCHES ON THE COMPUTER OR MONITOR WITHOUT THE INSTRUCTOR'S PERMISSION.** The monitor should show the main menu of the network at this time.
2. Use the up and down arrow keys on the right side of the keyboard to position the highlighted bar on **G) Select Network Printer**, then press **Enter**. Select **A) Printer A** if using computers 1-4, **B) Printer B** if using computers 5-8, or **C) Printer C** if using computers 9-12. Press **Enter**.
3. Scan each student disk for viruses (See Appendix E), then select Labworks 3.2 from the network main menu.
4. Insert the student disk into the right-hand disk drive (drive b:), making sure the metal is inserted first and the label side is up.
5. Complete the spreadsheet exercises given as a handout by your instructor, and turn the results in to your instructor.

Part 3: Calibration of Thermistor

1. Obtain a thermistor from the stockroom. Handle it with care, as it can break as easily as a thermometer.
2. Calibrate the thermistor (see Appendix A.), using thermometer, ice water and hot tap water.
3. When the calibration is completed, exit Labworks.

4. Eject the student disk from the disk drive by pushing the little blue button.
5. Return the thermistor to the stockroom technician or instructor.

DATA ANALYSIS:

Part 2: Spreadsheet Analysis

1. Check each of the graphs printed for Exercises to insure that it has a title, and both axes are labelled and properly scaled. Turn in all printed graphs and spreadsheets from the exercises, per instructor's directions.

QUESTIONS:

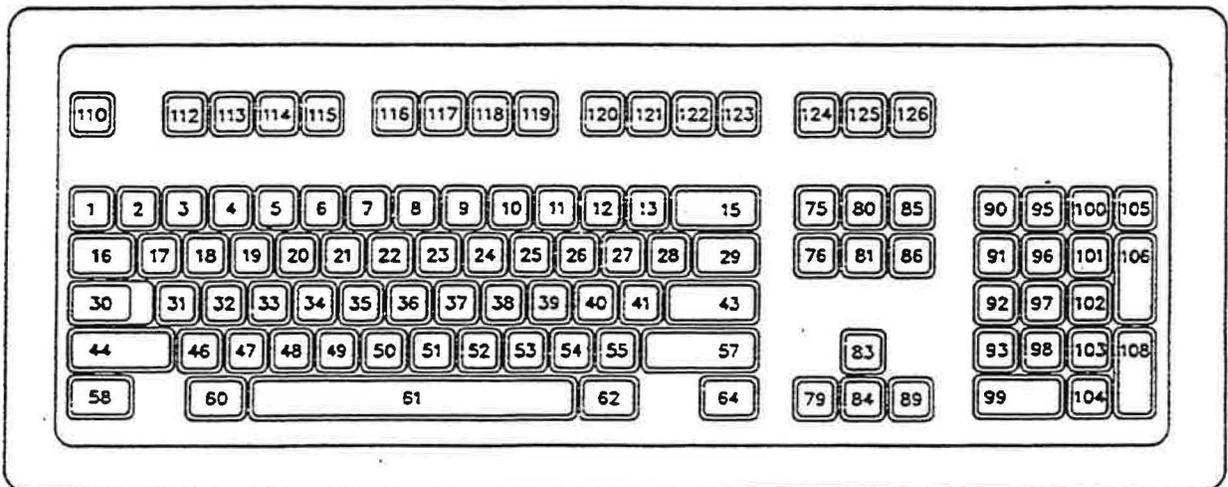
1. What can be done with the "Scaling" option in the Graphing Mode of Labworks 3.2?
2. List the series of steps required to manually enter new data into a clear Labworks 3.2 spreadsheet.
3. How can incorrectly entered data in Column C line 10 be edited?
4. How can the grid in the Graphing Mode be removed?
5. What is the most critical step in the calibration of a thermistor?

Computer Worksheet

Locate the following keys on the computer keyboard, placing the key number next to the corresponding key name.

- _____ Enter (2 of them)
- _____ F1
- _____ Backspace
- _____ Esc (Escape)
- _____ Tab
- _____ Insert
- _____ Print Screen
- _____ Delete (2 of them)
- _____ Ctrl (Control)
- _____ Alt (Alternate)
- _____ : (colon)
- _____ Arrow keys (4 of them)

Enhanced Keyboard



COMPUTERS AND SPREADSHEET ANALYSIS

The purpose of this part of the experiment is to guide the student through two exercises designed to make the student familiar and comfortable with the preparation and presentation of a computer spreadsheet. Through this work, the student will see that the spreadsheet is a useful and convenient tool when performing numerous graphing and calculational tasks.

Exercise # 1 Spreadsheet Basics

From a hypothetical set of data for two variables the student will construct a spreadsheet and then plot the best possible graph to discover what relationships, if any, exists between the variables. Here is the data:

<u>x</u>	<u>y</u>
1.0	4.80
2.0	6.80
3.0	9.80
4.0	12.10
5.0	19.50
6.0	17.00
7.0	20.00
8.0	23.50
9.0	24.70
10.0	28.50

In a timely manner before coming to the lab assigned for the spreadsheet work (see the lab schedule), prepare a graph, done neatly by hand, of the given data. Bring this graph to lab on the designated day; it will be part of the lab report. Think about graphing, i.e., graph paper with a sufficiently fine grid; a suitable scale for each axis; labeling of axes; spreading the data over the axes in order to use more than half (preferably all) of the graph paper; a title for the graph, etc. Note that x values will be plotted on the horizontal axis and y values on the vertical axis. Determine the slope and the intercept from the handmade graph and record the values on the plot. What algebraic equation connects the x and y values?

Now in lab the x,y data points will be plotted by the computer and then a comparison made of the two graphs, slopes, intercepts and algebraic equations. By the way, before you constructed the handmade graph, did you review graphing by studying the appropriate pages in the lab manual and the lecture text?

Column Setup

The first option you will use in the Spreadsheet is the Column Setup. The Column Setup option will allow you to label and format the columns to be used for entering the data into the spreadsheet. The following steps will set the column for your exercise:

1. With the highlight bar on Column Setup, use the down arrow key to move the bar through Set data columns to Column A, then press the Enter key. This will highlight the format for the data entering in Column A.
2. Hit the Enter key. Select the desired format(decimal or scientific. If you select the decimal format, you will be asked to select the number of decimal places next.). When done, hit the Enter key again. You have now set the format for Column A data.
3. Move the bar to Label and press the Enter key. This will allow you to label Column A.
4. Type the letter "x" for the label, then press the Enter key.
5. Using the right arrow, move the bar to Column B. Repeat steps 1,2,3 and 4 for Column B, except type the letter "y" for the label instead of "x".
6. Use the right arrow to position the bar back to Column Setup.

These six steps have set the columns in the spreadsheet to manually accept you x and y data.

Data

The Data option allows you to manually enter data or edit data in the spreadsheet.

1. Using the right arrow, move the highlighted bar to Data, then press the down arrow to highlight Enter/Edit and press the Enter key.
2. Proceed to type in the x,y pairs into the columns A and B of the spreadsheet. Pressing the Enter key after each piece of data enter that data. Use the right arrow to go from Column A to B and for entering the subsequent data points.
Note: After the last piece of data has been typed, press the Esc key, not the Enter key. The Esc key indicates to the computer that you have finished entering your data points. If you have already hit the Enter key(oops!), you will have a zero in the seventh data line. In this case, press the F6 key to delete row 7, then press the Esc key.
3. You should now have a table of your data points.

Graph

Now that you have entered your data points into the spreadsheet, you are ready to graph your data.

1. Using the right arrow, position the highlight bar to Graph, then use the down arrow to position the bar on X-axis and press the Enter key.
2. Now position the highlighted bar on Column A and press Enter. Steps 1 and 2 have made the data in column A of your spreadsheet the x-axis for your graph.
3. Graph will now be highlighted. With the down arrow, position the highlighted bar on Y-axis 1 and press the Enter key.
4. Using the down arrow, position the bar to Column B and press Enter. Steps 3 and 4 have made the data in column B of your spreadsheet the y-axis for your graph.
5. Now highlight the Title option and press Enter. Type in a descriptive title and press Enter.

6. Now highlight the Scaling option and press Enter. You can now scale your graph by selecting Min x (Minimum value of x), Max x (Maximum value of x), Min Y1, Max y1, one at a time, and typing in the desired values. Alternatively, you may select Automatic Scaling and let the computer scale the graph for you.
7. Press Esc key when you are finished with scaling.
8. You are now ready to plot the data points and construct the graph. Move the highlighted bar to Plot and press Enter. You will now have several types of graphs to choose from. Try Scatter graph and Line graph to see the difference in the plots. With a graph on the screen, pressing the Enter or Esc keys will return you to the spreadsheet, allowing you to try another type of graph.
9. The most useful graph for the purpose of this exercise is the Linear Regression option. This option will mathematically fit your data points to the best straight line. After choosing this option, you will want to Plot Full Range. With the Linear Regression option, you will see an equation like $y = mx + b$ printed at the top of your graph. This is the equation for the best straight line fit for the data points. The number "m" is the slope of the line; "b" is the y-intercept. "Corr: 0.9----" is also printed next to the equation. This is an indication of how well the generated equation for the straight line fits the data points. If all of the data points fall exactly on the line, "corr = 1". "Corr:0.9981", for example, means that the equation fits the data points quite well.

Editing Your Data

Looking closely at your graph, you will see that the fifth data point seems to fall much further away from the line than the other data points. Let's examine how the graph would look if you eliminated this data point.

1. Go back to the spreadsheet and select Data. Move the highlighted bar to Enter/Edit and press Enter.
2. Use the down arrow key to position the bar on data point no. 5.
3. Press the F6 key. This will delete this data point.
4. Now press the Esc key to return to the main menu of the spreadsheet.
5. Go back to the Graph option and replot the Linear Regression line. Look at the corr:value of the graph and the overall appearance of the graph. Do you see any difference?

Note that you may not delete a data point just because you feel like it. There must be a good, scientific reason for the eliminating of a point, e.g., a procedural error, a mathematical/statistical analysis etc.

Once you have seen the graph, return to the main menu of the spreadsheet.

6. Finally, position the highlighted bar to Quit and select Exit. Answer No to Save data file. You have now completed Exercise 1.

Exercise 2: Spreadsheet Complexities

Now that the data points from Exercise 1 has been plotted and the linear(right?) relationship between x and y values discovered, additional data will be processed to see what other math function capabilities have been programmed in the computer.

1. Follow the instructions on "booting up" the computer(if turned off) and select Spreadsheet and wait for the program to load.
2. Following steps 1-6 in the Column Setup and steps 1-3 of Data in Exercise 1, enter the following data set into the spreadsheet.

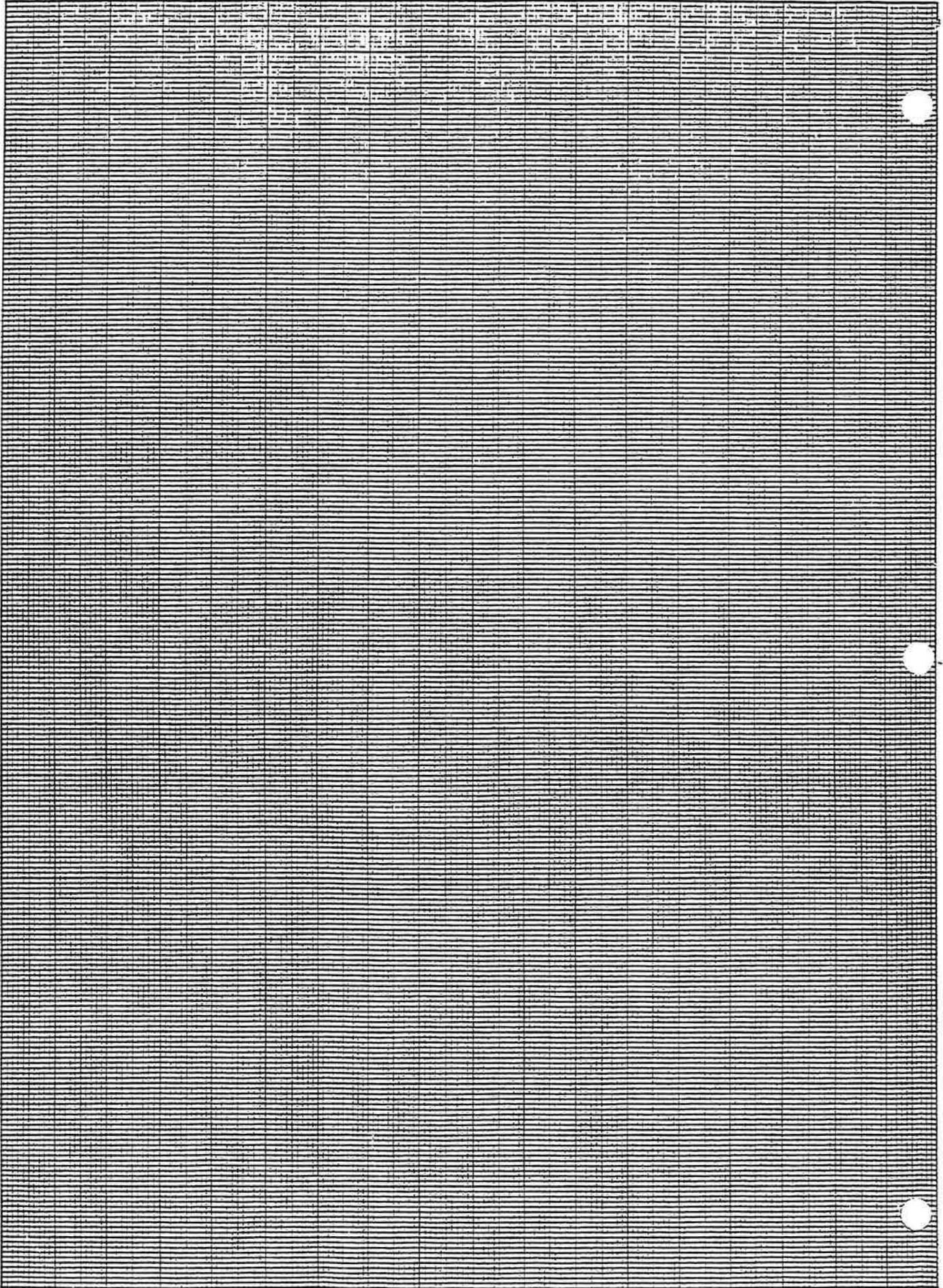
<u>x</u>	<u>y</u>
0.00	1.50
0.78	2.50
1.51	4.00
2.33	9.00
3.09	14.00
3.86	26.00
4.11	35.00
4.50	46.50
4.98	70.00
5.39	90.00

In science, a great deal of information can be learned from interpreting graphical data and mathematically manipulating the data. With the data entered into the spreadsheet, you are now ready to graph and analyze the data. So, let's complete the exercise, let's do it! You will plot this data in four(4) different ways: x v. y , x v. $1/y$, x v. $\log(y)$ and x v. $(y)^{1/2}$.

1. Using the right arrow key, highlight Analysis, then press Enter. Column E of the Data Analysis will be highlighted.
2. Using the down arrow, highlight Select Function, then press Enter.
3. A menu will appear from which you can select the function you need to manipulate the data. Enter the formula for one of your graphs in Column E. For example, for the graph of $\log(y)$ v. x , type $E = \log(b)$. Press Enter to calculate $\log(y)$ values.
4. Follow steps 2 and 3 and enter the formulas for the other two graphs; one in Column F and one in Column G.
5. Following steps 1-6 in the Graph portion of Exercise 1, plot the data and construct the graphs, one at a time. Which graph appears linear?
6. You have now completed Exercise 2. Assemble your spreadsheets and graphs into a lab report and turn the report in to your lab instructor.

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Introduction to Spreadsheets

Experiment

4

PURPOSE:

The purpose of this experiment is to guide the student through two exercises designed to make the student familiar and comfortable with the preparation and presentation of a computer spreadsheet. Through this work, the student will see that the spreadsheet is a useful and convenient tool for performing numerous calculations and graphing tasks.

DISCUSSION:

The computer software used in the Chemistry 1A and 1B classes will vary depending upon the needs of the student. Computer simulations, and tutorials are available for all students, covering a range of chemistry topics: scientific notation, significant figures, dimensional analysis, the periodic table, the mole concept, nomenclature, stoichiometry, oxidation-reduction reaction, equilibrium, aqueous solutions, qualitative analysis, organic chemistry and molecular modeling. Word processing is also available.

Computer spreadsheet programs have become very popular as a means of organizing and manipulating large quantities of data. The data is entered into columns of the spreadsheet, and then the program can perform computations on entire columns of numbers very quickly. Spreadsheet programs are well suited for use in chemistry classes because they can reduce the amount of time spent in the repetitive and lengthy calculations required.

Microsoft Excel (Excel) is a spreadsheet program that provides the features fundamental to the Mt. SAC Chemistry program. The program is user friendly, although practice is needed to master the many options. Excel has the capacity to handle large quantities of numerical data. Text, such as labels, titles and column headings can be easily inserted and formatted to give the spreadsheet a professional, polished look. The Excel charting capabilities allows data to be displayed as graphs in many different formats. A trendline such as linear regression can be added with just a few keystrokes.

Exercise 1 will serve as an introduction to basic Excel operations. The hypothetical set of data for two variables contained in Exercise 1 will first be hand - plotted by the student. The Y-intercept and slope will be calculated. The data will then be entered into cells of an Excel worksheet. The cells will be formatted and the computer will plot a graph of the data. A linear regression trendline, including the slope and Y-intercept of the line, will be added and the worksheet will be printed. The two graphing methods can then be compared.

Exercise 2 will reinforce the fundamental skills introduced in Exercise 1 and also provide instructions for performing calculations with Excel. The data for Exercise 2 will be entered into a new Excel worksheet. The data will be analyzed by calculating the inverse, log and square root of the Y column. Four graphs will be plotted: Y versus X, 1/Y versus X, log (Y) versus X and square root (Y) versus X. Linear regression trendlines will be added. The student will determine which plot is the most linear.

Even though Excel is a user-friendly program, understanding new software can be very challenging and time consuming. Read through the entire experiment carefully before coming to lab. Try to form a mental picture of the steps required for the various basic operations. Illustrations of screens and dialog boxes are included in the directions. Words that appear *italicized* direct the user to press that key on the computer or mouse. For example, *ENTER* means to press the Enter key on the computer. Words that appear in quotation marks are choices on a menu or dialog box. For example, "File" is found on the Excel menu toolbar.

The mouse has two buttons. *Click* directs the user to use the mouse to place the cursor on an icon or selected word on a menu and quickly press the left button. This is the most common use of the mouse. *Double-click* refers to pressing the left mouse button twice in rapid succession. *Right-click* refers to pressing the right mouse button. This is the least common use of the mouse. *Drag* refers to holding down the left mouse button until the cursor is moved to a desired location.

Materials and Equipment:

Equipment

- Computer equipped with Microsoft Excel software
- Printer connected to the computer
- Formatted student disk

PROCEDURE:

Part 1 Hand Plot of Exercise 1 Data

1. Before coming to the first laboratory period assigned for Introduction to Spreadsheets, prepare a graph, done neatly by hand, of the data given for Exercise 1. Bring this graph to lab on the designated day. It will be part of the lab report.
 - a. Note that X values will be plotted on the horizontal axis and Y values on the vertical axis.
 - b. Think about graphing, i.e., graph paper with a sufficiently fine grid; a suitable scale for each axis; labeling of axes; spreading the data over the axes in order to use more than half of the graph paper; a title for the graph.
 - c. Determine the slope and Y intercept from the graph and record these values on the plot.

d. Exercise 1 data:

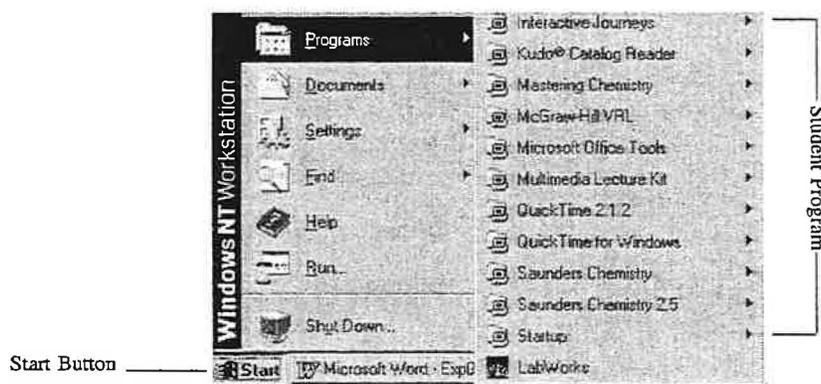
<u>X</u>	<u>Y</u>	<u>X</u>	<u>Y</u>
1.0	4.80	6.0	17.00
2.0	6.80	7.0	20.00
3.0	9.80	8.0	23.50
4.0	12.10	9.0	24.70
5.0	19.50	10.0	28.50

Part 2 Computer Plot of Exercise 1 Data

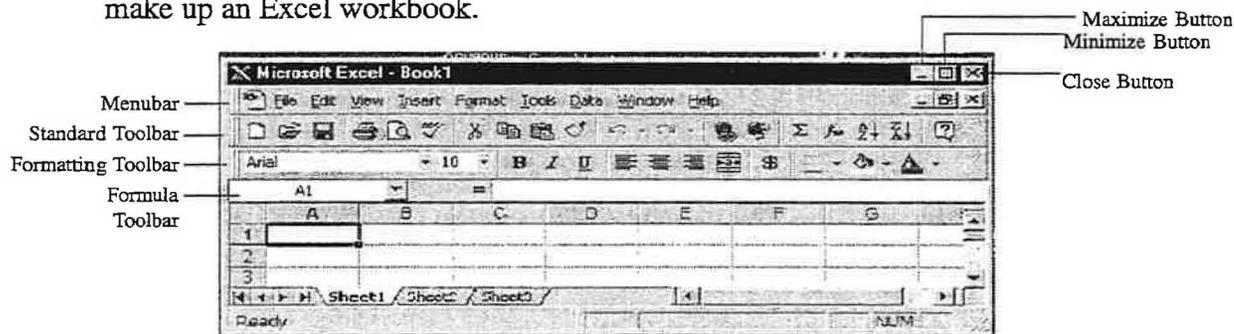
The following parts of the experiment will be performed in a computer lab with a lab partner(s). The instructor or technician will turn on the electrical power to the computer and will log the computer onto the network. **DO NOT FLIP THE POWER SWITCHES ON THE COMPUTER OR MONITOR WITHOUT THE INSTRUCTOR'S PERMISSION**

A. Opening Microsoft Excel

1. Use the mouse to place the pointer on "Start" (lower left corner of screen). *Click* to open the Windows menu. Place the pointer on the Windows menu. As you move the pointer to an item on the menu, the word will become highlighted in blue and a submenu will appear. Highlight "Programs". The students programs will be appear.



2. Now move the pointer to "Microsoft Excel". When it is highlighted, *click*. This will open the Microsoft Excel program and a blank worksheet will appear. Several worksheets make up an Excel workbook.

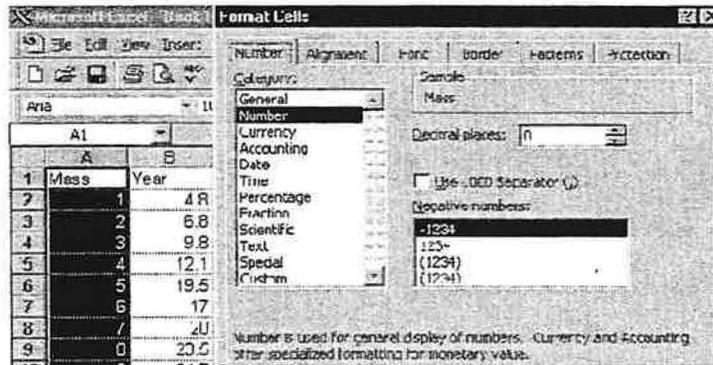


B. Entering Data Into the Spreadsheet

1. The cursor has become a thick, white cross pointer (). Use this cursor to move around the spreadsheet and select the cells that will be used. Place the cursor in a cell and *click* the mouse button. This will activate that cell and allow information to be entered.
2. Begin Exercise 1 by activating cell A1 and typing a heading for the first column of data. Press **Tab** when finished. The heading will appear in cell A1. The cursor will move to the right, activating the next cell (B1).
3. Type in the second column heading. Press **Enter** when finished. The cursor will move to the cell under the first column heading (A2). The mouse and the arrow keys on the keyboard as well as the Tab and Enter keys may be used to move the cursor.
4. Enter the data for Exercise 1 into the appropriate cells under the two column headings. Notice that zeroes are not shown. The cells must be formatted to the correct number of decimal places.

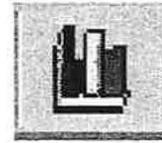
C. Formatting and Editing the Appearance of the Spreadsheet and Graph

1. The “Format Cells” dialog box can be used to format individual cells and entire columns. Highlight the cell(s) or column(s) to be formatted. Go to the menu toolbar and *Click* “Format” and then “Cells” to open the “Format Cells” dialog box.



2. *Click* the “Number” tab to format numerical entries. Choose the “Number” category to designate the amount of decimal places in a cell or column or choose “Scientific” to convert data to scientific notation. Format both columns in Exercise 1 to reflect the appropriate number of significant digits. *Click* “OK”.
3. *Click* the “Alignment” tab to designate where in the cell the entry will be placed. Align the data in both columns to be placed in the center of the cell, both horizontally and vertically. *Click* “OK”.
4. *Click* the “Font” tab to format the size and font of text. *Click* “Times New Roman” as the font. *Click* “12” as the size of the font. *Click* “OK”.

D. Plotting Graphs Using Chart Wizard as a Guide



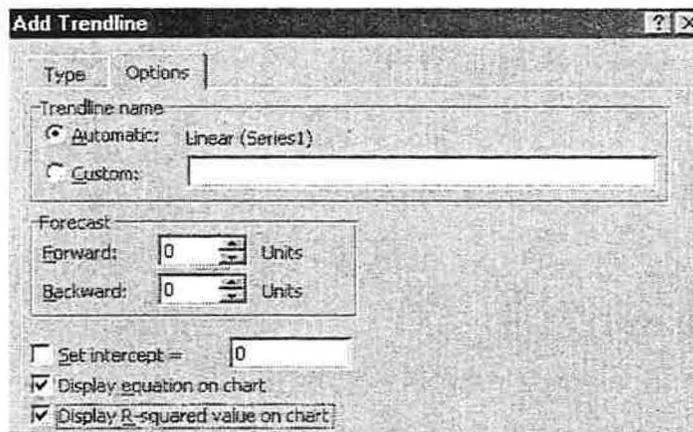
1. **Click** the Chart Wizard icon on the formatting tool bar to open the series of dialog boxes:
 - a. Step 1 – Chart Type. Select type of graph that will be plotted. **Click** the “Standard Types” tab. Highlight “XY (Scatter)” as the chart type and **click**. **Click** “Scatter” as the subtype from the five choices given. **Click** “Next”.
 - b. Step 2 – Chart Source Data. Select the Data Range for the graph. **Click** the “Data Range” tab. **Click** “columns” if it hasn’t already been automatically selected. The data will automatically be plotted. **Click** “Next”.
 - c. Step 3 – Chart Options. Complete the graph. Several tabs will appear on the dialog box. When finished with the following choices, **click** “Next”.
 - (i) “Titles” provides a means to label both axes and to title the graph. Enter appropriate labels for the X and Y axis and a title for Exercise 1.
 - (ii) “Axes” allows the user to designate whether values should appear on the X and Y axes. Be sure that both axes have a number scale.
 - (iii) “Gridlines” permits the user to include major and minor X and Y gridlines. Exercise 1 should include only major X and Y gridlines.
 - (iv) Use the “Legend” tab to place the legend for the chart at the bottom of the page.
 - (v) “Data Labels” permits each data point to have its associated values printed near the point. This option will not be used for Exercise 1.
 - d. Step 4 – Chart Location. There are two choices for “Chart Location”. **Click** the second choice; as object in Sheet 1. The graph will be inserted onto the same sheet as the data. **Click** “Finish”.
 - e. Once the chart has been completed, change the plot area color to white; place the cursor on the gray area of the graph. “Plot area” should appear below the cursor. **Double click**. The “Format Plot Area” dialog box will appear providing a choice of “Borders” and “Area”. Go to “Area” and **click** the white box, and then “OK”. The gray area on the graph will become white.
 - f. The complete graph appears as a small box on the spreadsheet containing the data for Exercise 1. Place the graph below the spreadsheet data; place the cursor on the active graph (the graph is active if black rectangles appear around the perimeter) and hold down the left mouse key. The cursor will become a four-headed arrow. Drag the graph to the desired location and then release the mouse key.

- g. Resize the graph to the instructor's specifications; place the arrow cursor on one of the black rectangles on the perimeter of the graph so that it becomes a two-headed arrow (\leftrightarrow). Hold down the left mouse key and drag the arrow until the graph is the appropriate size and then release the mouse key.

2. The choices made while navigating through the Chart Wizard dialog boxes can be easily altered. Simply place the cursor on the chart and *click*. The chart is now the active portion of the worksheet. Then *click* "Chart" on the menu toolbar. The titles of the Chart Wizard dialog boxes will appear as a drop down menu and can be activated by highlighting and clicking. "Chart" only appears on the toolbar when a chart is active.

E. Adding Trendlines to Charts

1. *Click* the chart on the Exercise 1 workbook to activate it. *Click* "Chart" on the menu toolbar. *Click* "Add Trendline". The Add Trendline dialog box will appear.

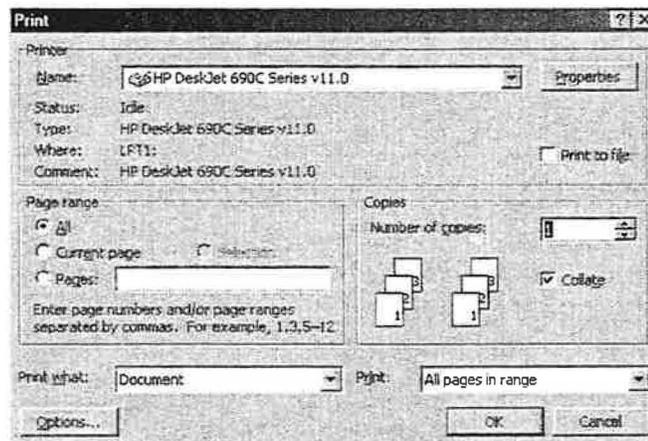


- a. Click "Linear" from the Trend/Regression types.
- b. Click the "Options" tab.
- c. Click "Display R-squared value on chart" and "Display equation on chart". The linear equation and regression coefficient will appear on the graph.

F. The Print Preview and Print Features

1. The Print Preview feature permits the workbook to be viewed exactly as it will be paged, before it is printed. This allows the user to approve the appearance of a document or make changes prior to printing. Go to the menu toolbar and *click* "File", then "Print Preview". The workbook will be displayed along with a toolbar. Changes cannot be made in this mode. If the chart is active when "Print Preview" is selected, only the chart will be previewed.

- a. **Click** “Zoom” to gain a magnifying-glass view of the workbook. **Click** “Zoom” again to return to the entire page.
 - b. **Click** “Close” to return to the active workbook.
 - c. Use Print Preview to look carefully at the Exercise 1 workbook. Make any desired formatting changes to the workbook. To resize the chart, place the cursor on it and **click** . Eight small black rectangles will appear around the perimeter of the graph. Place the cursor on one of these boxes until the cursor becomes a double-headed arrow (↔) and **drag** the box to the desired size.
2. To print a workbook, go to the menu toolbar and **click** “Print”. The Print dialog box will appear.



- a. The “Name” box indicates where the workbook will be printed. Locate this printer to retrieve the printed document.
 - b. **Click** “OK” to begin printing one copy of the workbook for Exercise 1. Use a photocopier to make more copies, if necessary.
3. Print a copy of the spreadsheet and graph for Exercise 1.

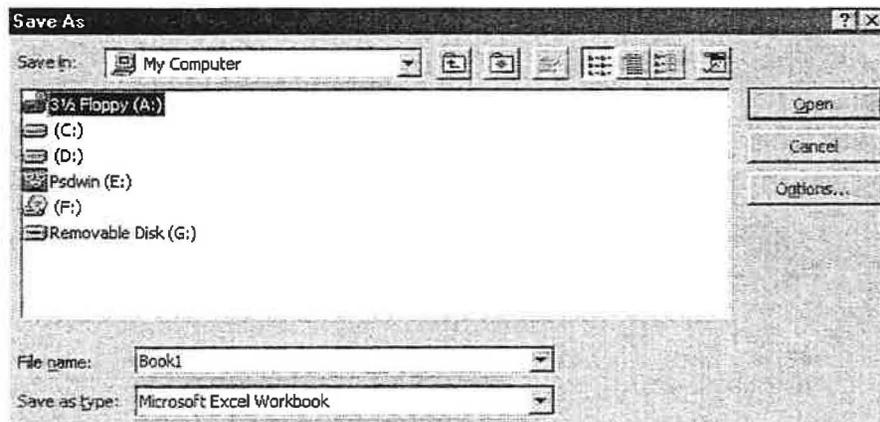
G. Editing Data on the Spreadsheet

1. A spreadsheet can be edited to change or remove data entries. **Click** the cell, row or column to be edited to activate it and then choose the following options.
 - a. An incorrect data entry can be corrected by simply typing the correct entry. The entry will appear after the equal sign on the formula bar.
 - b. A data entry may be deleted by pressing the “delete” key. This choice will leave the cell active.

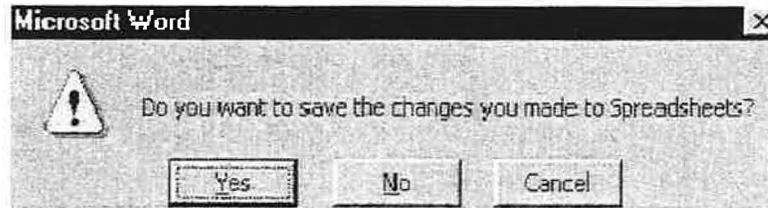
- c. A cell or row of cells may be deleted by *clicking* “Edit” on the menu bar and then *clicking* “delete”. The highlighted cells will disappear.
 - d. If a data entry was accidentally deleted, immediately click “Edit” and then “undo delete”. The deleted data will reappear.
2. Looking closely at the graph, you will see that the fifth data point seems to fall much further away from the line than the other data points. Examine how the graph would look if this data point were eliminated.
- a. Highlight the row where the data for the fifth data point appears.
 - b. Delete the data.
 - c. Place a linear regression trendline, R-squared value and equation line on the graph.
 - d. Print a copy of the edited spreadsheet and graph.

H. Saving, Closing and Opening a Data File

1. To save a file, go to the menu toolbar and *click* “File”. *Click* “Save As” and a dialog box will appear.
 - a. The workbook for Exercise 1 will be saved on the student disk. Place the formatted student disk into the floppy (A:) drive of the computer.
 - b. Use “Save in” to select “Floppy (A:)” if it is not automatically selected. Type in an appropriate title for the workbook in the “File name” space and then *enter*. The small light under the “A:” drive will come on as the workbook is saved.



2. To close a workbook, go to “File” and *click* “Close” on the menu toolbar. The following dialog box appears. If the file has just been save, click “No”. If data has been entered or changed since last saving the file, click “Yes”. The workbook will disappear, but the Excel program is still active.



3. To open a saved file, go to the menu toolbar and *click* “File”, then “Open”. The Open dialog box, very similar to the Save As dialog box, will appear.
 - a. Use the “Look in” box to select “Floppy (A:)”. A list of files on the disk will appear.
 - b. Highlight the file to be opened (Exercise 1). *Click*. The Exercise 1 workbook will appear.

Part 3 Analysis Plotting of Exercise 2 Data Using Excel

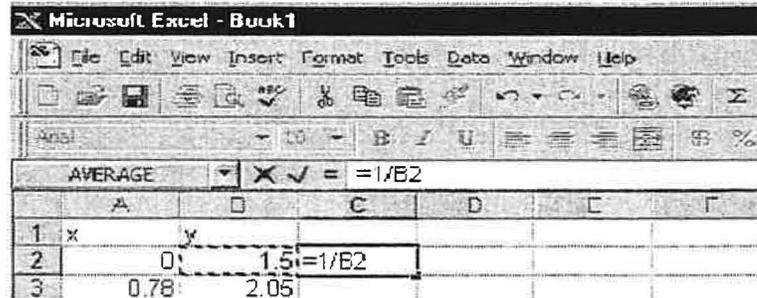
A. Entering Formulas and Performing Calculations

1. Instructions for Entering Formulas and Performing Calculations will be given using the data in Exercise 2 after it has been entered into a new workbook.
 - a. Exercise 2 data:

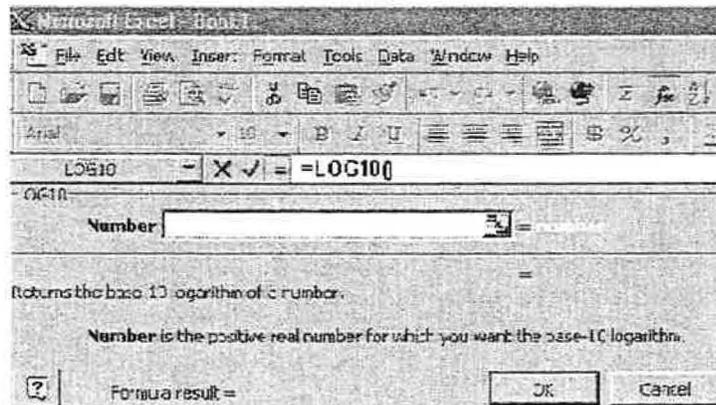
<u>X</u>	<u>Y</u>	<u>X</u>	<u>Y</u>
0.0	1.50	3.86	26.00
0.78	2.50	4.11	35.00
1.51	4.00	4.50	46.50
2.33	9.00	4.98	70.00
3.09	14.00	5.39	90.00

- b. Go to “File” on the menu toolbar and *click* “New”. *Click* “Workbook”.
- c. Use the information in Part 2 to enter the first column of data into cells A1–A11. Enter the second column of data into cells B1 – B11. Format the cells.

2. The formula toolbar will be used to calculate the inverse of the numbers in the Y column (1/Y). The result of the calculation will be placed in the C column.
 - a. Activate cell C2.
 - b. Type an equal (=) sign to begin the formula. This indicates that what follows is a formula, not text or a number.
 - c. Type in the formula for the inverse of the first value in the Y column, but instead of typing in the value, *click* on the cell. 1/B2 will appear in the large white box on the formula bar.



- d. Press *Enter*. The result of the calculation will appear in cell C2.
 - e. To calculate the inverse of the rest of the column, the Excel AutoFill feature will be used. Move the cursor along the bottom right corner of cell C2 until it turns into a black cross (+). Click and drag the cursor down the column, to cell C11. When the mouse button is released, Excel will calculate the result for each value in the Y column and place it in the appropriate cell in the C column.
3. The Excel Formula Palette will be used to calculate the log of the values in the Y column. The results will be placed in the D column.
 - a. Activate cell D 2 and type = .
 - b. Open the Formula Palette by clicking *fx* on the standard toolbar. Highlight “Math & Trig” in the function category to bring up the function names. Scroll down the function names to “LOG10” and *click*. *Click* “OK” The log (base ten) function has been selected.



- c. The number that will be used in the calculation needs to be entered. Place the cursor on the red arrow and *click* to return to the spreadsheet. Select cell B2, *click* and then *enter*. B2 will appear in the number box. Click "OK". The result will appear in cell D2.

The number (1.5) can also be typed in, but the AutoFill feature will not be able to calculate the function for the entire column unless a cell reference is entered.

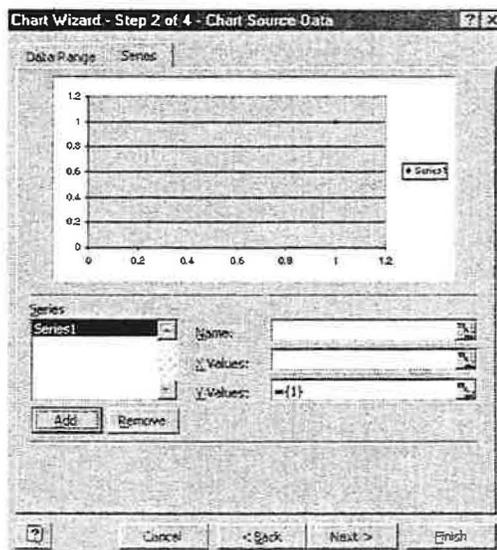
- d. Use AutoFill and the black cross cursor to calculate the log of the remaining values in the Y column. The results will be placed cells D2 to D11.
4. The Formula Palette will also be used to calculate the square root of the numbers in the Y column ($Y^{1/2}$). The result of the calculation will be placed in the E column.
 - a. Activate cell E2 and type = .
 - b. Open the Formula Palette by clicking *fx* on the standard toolbar. Highlight "Math & Trig" in the function category to bring up the function names. Scroll down the function names to "SQRT" and *click*. *Click* "OK" The square root function has been selected.
 - c. The number that will be used in the calculation needs to be entered. Place the cursor on the red arrow and *click* to return to the spreadsheet. Select cell B2, *click* and then *Enter*. B2 will appear in the number box. Click "OK". The result will appear in cell E2.
 - d. Use AutoFill and the black cross cursor to calculate the log of the remaining values in the Y column. The results will be placed cells E2 to E11.

5. The Exercise 2 workbook now consists of five columns of data. Be sure that all columns are appropriately formatted and titled. Save the workbook.

B. Adding Series to Charts

1. Open the Exercise 2 workbook. An XY Scatter graph of $1/Y$ versus X will be plotted. Since there are several choices of rows and columns on the spreadsheet, the computer does not know which data to plot.
 - a. *Click* "Chart Wizard" and proceed as before to step 2.

- b. **Click** the “Series” tab to select the data series that will be plotted. The series dialog box will open. If the computer has automatically plotted data, use “Remove” to clear the series. Highlight the series and **click** “Remove”.



- c. **Click** “Add”. The X and Y values can now be selected.
- d. **Click** the red arrow to the right of “X Values”. The spreadsheet will reappear. Highlight the X values by **clicking** cell A2 and **dragging** the cursor to cell A11. The data in the A column will be highlighted. **Enter**.
- e. Follow step d above to select the data in cells C2 to C11 as the Y values. **Enter**.
- f. Complete the graph by continuing through the remaining steps in Chart Wizard. Include titles, major X and Y grid lines.
- g. Add a linear regression trendline to the graph. Display the R-squared value.
2. Plot XY Scatter graphs of Y versus X, log Y versus X and square root Y versus X, one at a time. If a previously plotted chart is active when Chart Wizard is selected, the new chart will replace the previous one.
- Place these three graphs on the same worksheet as the data table and the previous graph or as instructed by the teacher.
 - Add a linear regression trendline to each graph. Display the R-squared value.
 - Convert the plot area from gray to white.
 - Print one copy of the worksheet and graphs. Be sure to use Print Preview to optimize the appearance of the sheet.

QUESTIONS

1. Consider the three graphs that were plotted for Exercise 1; the hand – plotted graph, the computer graph and the edited – computer graph.
 - a. Compare your slope from the hand – plotted graph with computer calculated slope. How close are the values?
 - b. Compare your slope from the hand - plotted graph with another student's s hand - plotted slope. How close are the values?
 - c. Compare your slope from the computer graph with another student's computer calculated slope. How close are the values?
 - d. Which method, hand – plotting or computer plotting, provides the more consistent interpretation of the data?
 - e. Compare your R-squared value from the computer graph with your R-squared value from the edited – computer graph. What does this information tell you about the graphs?

2. Consider the 4 graphs plotted for Exercise 2.
 - a. Which graph appears more linear?
 - b. What information does the linear regression coefficient tell you? Explain, using information from Exercise 2.

3. Are you comfortable using Excel? If not, what do you need to become more comfortable?

#5 - Acid/Base Phenomena

PURPOSE:

The pH of various solutions will be determined to see if water has reacted with a species in solution. If hydrolysis has occurred, then equilibrium constants for the hydrolysis reactions will be calculated.

Volumetric analysis of a weak acid with a strong base will be performed using computer-assisted data acquisition. Computer-aided volumetric analysis provides an opportunity to study acid/base phenomena in greater depth than standard volumetric analysis - where chemical activity ends at the titration endpoint. A real-time full pH curve extending well past the endpoint will be generated during titration, and then subsequently analyzed.

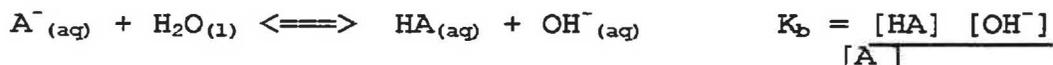
DISCUSSION:

Like water, aqueous acids and bases are characterized by the presence of H^+ or OH^- ions in solution. Measurements of the concentrations of acids or bases in solution are routinely accomplished by use of the pH scale. The addition of strong acid to water results in an increase in the concentration of hydrogen ions, and consequently a lower pH (less than 7), while the addition of strong base to solution results in a pH greater than 7. Strong acids and bases that dissociate completely in solution do not establish equilibria. A **weak acid** is defined as an acid that **dissociates slightly in aqueous solution**. Weak acids establish an equilibrium that lies to the left when dissolved in water and have an associated equilibrium constant, K_a .



The strength of a weak acid is expressed through the K_a value. Very weak acids have very small K_a values. The K_a value increases with the strength of the acid.

Weak acids may be titrated with strong bases in a manner similar to a titration of a strong acid. The equivalence point of any titration occurs when the number of moles of base added is equal to the number of moles of acid present originally. At the equivalence point of a titration of a weak acid with a strong base, the reaction has essentially gone to completion and the conjugate base, A^- , and H_2O are the major species in solution. A new equilibrium will be established as the conjugate base A^- reacts with H_2O in a hydrolysis reaction, with a new equilibrium constant, K_b , now prevailing. K_b is expressed like the K_a :



Computer-aided measurement of the pH during titration allows the real-time graph to be displayed during titration, and it also allows a titration curve of pH versus volume of base to be

plotted later. The shape of the pH curve of a weak acid titration is somewhat different from the pH curve of a strong acid titration. The pH curve of the weak acid rises slowly at first in a **buffering effect**, then rises sharply through the equivalence point, and then levels off again.

The buffering effect in the early portion of the titration occurs because of the **ability of a weak acid to absorb OH^- ions while the pH of the solution remains constant**. The added OH^- ions remove H^+ from solution to form water thereby causing the equilibrium in equation (1) to shift to the right. The remaining HA in solution dissociates into H^+ and A^- , which replaces the H^+ which has been neutralized.

The equivalence point of a weak acid does not occur at pH 7.00 as it does when a strong acid is titrated. In fact, the equivalence point of a weak acid is always greater than 7.00. This occurs because the anion, A^- , that remains in solution after the hydronium ion is neutralized acts as a base.

The **negative log of the K_a ($-\log K_a$)** results in a value called the **$\text{p}K_a$** . The $\text{p}K_a$ scale is the same as the pH scale, with values ranging from 0 to 14. Weak acids that have small K_a values have large $\text{p}K_a$ values. The $\text{p}K_a$ of a weak acid is equal to the pH of solution at the point of fifty percent neutralization, in the middle of the buffering region of the titration pH curve.

In Part 2 of this experiment, a weak acid, acetic acid, will be titrated with a standardized strong base, NaOH. The titration will continue to a pH well past the endpoint. The Labworks software and the computer, equipped with a pH electrode, will be used to monitor the real-time progression of the titration. The neutralization point (equivalence point) will be determined through calculation of the inflection point of the pH curve.

In Part 3, an unknown weak acid will be titrated with the same strong base used in Part 1. The unknown acid may be mono, di, or triprotic. The $\text{p}K_a$ ('s) of the weak acid and the point(s) of neutralization will be determined from a graph of the pH curve. The identity of the unknown acid will be determined from a list of possible unknowns.

Salts also dissociate in an aqueous solution to form cations and anions. The ions are surrounded by a cage of polar water molecules, in a process called hydration. The oxygen end of the water molecule (slightly negative) is attracted to the cation, while the hydrogen end of the water molecule (slightly positive) is attracted to the anion. Two factors are involved in this hydration of the ion: the charge of the ion and the size of the ion. Combined, they are referred to as the charge density of the ion. In general, the larger the charge density, the more strongly hydrated the salt becomes. For example Ba^{2+} and Ca^{2+} are weakly hydrated, while Fe^{3+} and Al^{3+} are strongly hydrated.

A strongly hydrated cation attracts the negative end of the water molecule and weakens the O-H bond. This results in H^+

being released, producing an acidic solution. The hydroxide ion produced remains associated with the cation. The water has been broken apart by the salt in a process called **hydrolysis**. The pH of this solution would be less than 7 and the salt would be called an acid salt. On the other hand, a strongly hydrated anion would attract the positive end of the water molecule. Hydrolysis occurs, but in this case the H^+ remains with the anion and the OH^- is released into solution. The pH increases. This is known as a basic salt.

Salts that produce ions that have a very weak attraction for water molecules do not affect the pH of the solution. Some examples of ions not affecting pH are: Group IA, Group IIA, all other metals in +1 oxidation state, and halides. These ions all have relatively low charge density.

Hydrolysis can also be caused by ions that are classified as the conjugate bases of weak acids, or as conjugate acids of weak bases. Salts can be considered to be the result of the reaction between an acid and a base. If both the acid and base are strong, the salt formed will produce a neutral solution when dissolved in water. If, on the other hand, either the acid or the base is weak, the resulting salt can cause hydrolysis. Consider the salt sodium acetate. It contains the acetate ion which is the conjugate base of acetic acid. It will react with water according to the following equation:



This equilibrium lies to the right. When sodium acetate is dissolved in water, the acetate ion reacts with water to form the unionized form of acetic acid. This process releases hydroxide ions into solution, thereby causing the pH to increase. The equilibrium constant for this type of reaction is known as the hydrolysis constant, K_h . The K_h expression is written as a K_a expression or a K_b expression for a given equilibrium reaction, depending on the pH of the solution. When the pH is less than 7, a K_a expression is written for the K_h expression, and vice versa. Since the pH of the reaction equilibrium shown in equation (4) is greater than 7, the K_h expression for this hydrolysis reaction will be written like a K_b expression ($[OH^-]$ in the numerator):

$$K_h = \frac{[HC_2H_3O_2] [OH^-]}{[C_2H_3O_2]}$$

The pH of several salt solutions will be measured in Part 1 of this experiment. K_h expressions will be written and the value of K_h for each salt will be calculated.

MATERIALS and EQUIPMENT:

Solutions

phenolphthalein
0.10 M sodium chloride (NaCl)
0.10 M sodium acetate ($\text{NaC}_2\text{H}_3\text{O}_2$)
0.10 M copper nitrate ($\text{Cu}(\text{NO}_3)_2$)
0.10 M ammonium chloride (NH_4Cl)
0.10 M zinc chloride (ZnCl_2)
0.10 M sodium bicarbonate (NaHCO_3)
0.10 M potassium aluminum sulfate ($\text{KAl}(\text{SO}_4)_2$)
0.20 M sodium hydroxide (NaOH)
liquid weak acid unknowns
pH 7.00 buffer
0.10 M sodium bisulfite (NaHSO_3)
0.10 M sodium monophosphate (Na_2HPO_4)
0.10 M sodium carbonate (Na_2CO_3)
0.20 M sodium hydroxide (NaOH)

Stockroom equipment

pH electrode	computer/Labworks
drop counter/photogates	Labworks interface
pipet pump	5.00 mL pipet

Locker equipment

10.00 mL pipet	150 mL beaker
large beaker	test tube rack
150 mm test tubes	

PROCEDURE:

Part 1: Determination of K_h

1. Prepare a data table with the following columns:

Substance	1	2	3	4	5
	$\frac{1}{H}$	$\frac{2}{[H^+]}$	$\frac{3}{[OH^-]}$	$\frac{4}{K_h \text{ Expression}}$	$\frac{5}{K_h}$

2. Label 10 test tubes as follows:

#1. 0.10 M NaCl	#6. 0.10 M $\text{KAl}(\text{SO}_4)_2$
#2. 0.10 M $\text{Cu}(\text{NO}_3)_2$	#7. 0.10 M NaHCO_3
#3. 0.10 M NH_4Cl	#8. 0.10 M Na_2HPO_4
#4. 0.10 M ZnCl_2	#9. 0.10 M $\text{NaC}_2\text{H}_3\text{O}_2$
#5. 0.10 M NaHSO_3	#10. 0.10 M Na_2CO_3

3. Measure approximately 5 mL of the appropriate substance into the 10 labelled test tubes.
4. Connect a pH electrode to the "pH1" outlet on the interface. Handle the electrode with care!
5. Load Labworks and calibrate the pH electrode (see Appendix A if necessary) using a buffer solution of known pH.
6. Measure the pH of the ten solutions using the **pHmeter** program in Labworks. Record these values in the data table.

Part 2: Titration of a Weak Acid with a Strong Base

1. Pipet 10.00 mL of 0.100 M. $\text{HC}_2\text{H}_3\text{O}_2$ into a clean, dry and labelled 150 mL beaker.
2. Add 25 mL of distilled water and 2 drops of phenolphthalein to the solution and note the color of solution.
3. Scan each student disk for viruses (Appendix E) and then load Labworks. Calibrate the pH electrode using a buffer solution of known pH.
4. Connect a photogate or drop counter to the "counter" connection on the interface panel. Align the drop counter or photogate so that titrant will drop directly through the center and will be detected. Do not touch after alignment.
5. In the **PERFORM EXPERIMENT** mode, load the program **ACIDBASE** from the network (f:). Label the output file (to drive b:) as **ACETIC**. Choose **INTERFACE** and **GRAPH RIGHT** modes.
6. Titrate the solution in the beaker by following the instructions on the screen. Gently and consistently stir the solution with the electrode while titrating.
7. Add titrant until the solution pH is about 12. Note the pH when the solution changes color.

Part 3: Identification of an Unknown Weak Acid

1. Obtain an unknown weak acid solution from the instructor. Record the number of the unknown.
2. Repeat steps 1 - 7 of Part 2, using the weak acid solution unknown this time. Label the output file (to drive b:) as **UNKNOWN**.

DATA ANALYSIS:

Part 1: Determination of K_h

1. From the pH values recorded in the data table, calculate and record the values of $[\text{H}^+]$ and $[\text{OH}^-]$ for each substance.
2. For each salt tested, write the hydrolysis expression in column 4.
3. For each salt tested, calculate and record the value of each hydrolysis constant, K_h .

Part 2: Titration of a Weak Acid with Strong Base

1. In the **SPREADSHEET** mode of Labworks, load the **ACETIC** file, and plot a **Line Graph, No points** graph of drops of titrant on the X axis and pH on the Y axis.

- Use Column E to determine the derivative of the pH curve, by entering the formula **deriv(B,A)**.
- The largest value of the derivative is indicative of the inflection point of the curve, which is equal to the equivalence point of the titration. On the computer screen, find the largest value of the derivative in Column E, and then press the Print Screen key. Note the corresponding value, on the same line, in Column A (which is the number of drops of titrant needed to exactly neutralize the acid in solution, i.e. the equivalence point).
- On the computer screen, also locate the spreadsheet value in Column A which is exactly 1/2 of the Column A value determined in step 3, and then press the Print Screen key again. This new value corresponds to the volume of titrant at halfway to the equivalence point.
- Using the value in Column A from step 4, find the pH value in Column B which is directly across from it. This new value is the pH of the solution at exactly halfway to the equivalence point, and should also be equal to the pK_a of acetic acid.

Part 3: Identification of An Unknown Weak Acid

- Repeat steps 1 - 5 of Part 2, using the **UNKNOWN** file.
- From the pK_a value obtained in step 5, determine the K_a of the unknown weak acid.
- Determine the identity of the unknown weak acid.

<u>Possible Unknowns</u>			
<u>ACID</u>	<u>K_a</u>	<u>ACID</u>	<u>K_a</u>
Boric acid	7.3×10^{-10}	K_2HPO_4	4.8×10^{-13}
	1.8×10^{-13}	KH_2PO_4	6.2×10^{-8}
	1.6×10^{-14}		
Methanoic acid	1.77×10^{-4}	$NaHCO_3$	5.61×10^{-11}
Ethanoic acid	1.76×10^{-5}	H_2SO_3	1.54×10^{-2}
Propanoic acid	1.34×10^{-5}		1.02×10^{-7}
Butanoic acid	1.54×10^{-5}	Oxalic acid	5.6×10^{-2}
Lactic acid	8.4×10^{-4}		5.1×10^{-5}

Titration of a Weak Acid with a Strong Base

Experiment

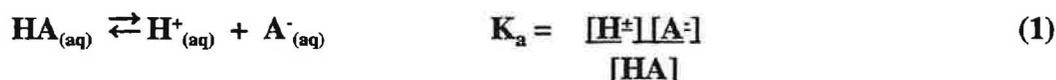
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PURPOSE:

The purpose of this experiment is to perform a volumetric analysis of a weak acid with a strong base using a CBL system for data acquisition. A real-time full pH curve extending well past the endpoint will be generated during titration, and then subsequently analyzed.

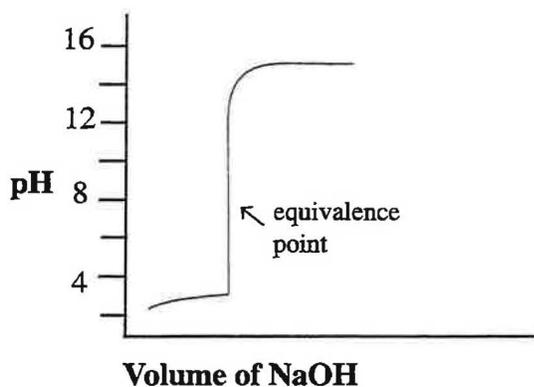
DISCUSSION:

Aqueous acid and base solutions are characterized by the presence of H^+ or OH^- ions in solution. Measurements of the concentrations of acids or bases in solution are routinely accomplished by use of a pH meter. The addition of strong acid to water results in an increase in the concentration of hydrogen ions, and consequently a lower pH (less than 7), while the addition of strong base to solution results in a pH greater than 7. Strong acids and bases that dissociate completely in solution do not establish equilibria. A **weak acid** is defined as an acid that **dissociates slightly in aqueous solution**. Weak acids establish an equilibrium that lies to the left when dissolved in water and have an associated equilibrium constant, K_a .

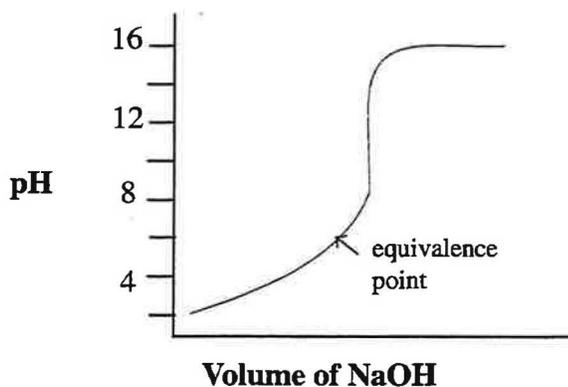


The strength of a weak acid is expressed through the K_a value. Very weak acids have very small K_a values. The K_a value increases with the strength of the acid.

Weak acids may be titrated with strong bases in a manner similar to a titration of a strong acid. Studying an entire titration curve provides an opportunity to study acid/base phenomena in greater depth than standard volumetric analysis - where chemical activity ends at the titration endpoint. The shape of the pH curve of a weak acid titration is somewhat different from the pH curve of a strong acid titration. Significant highlights noted on the titration curve such as the equivalence point, half-equivalence point and buffering region can give information about the weak acid. The pH curve of the weak acid rises slowly at first in a buffering effect, then rises sharply through the equivalence point, and then levels off again:



A. pH curve of a strong acid/strong base titration.



B. pH curve of a weak acid/strong base titration.

The equivalence point of any titration occurs when the number of moles of base added is equal to the number of moles of acid present originally. At the equivalence point of the titration of a weak acid with a strong base, the reaction has essentially gone to completion. The conjugate base, A^- , and H_2O are the major species in solution. A new equilibrium will be established as the conjugate base, A^- , reacts with H_2O in a hydrolysis reaction. The equivalence point of a weak acid titrated with a strong base will always be greater than 7 because of this hydrolysis reaction. A new equilibrium constant, K_b , is established:



The **negative log of the K_a ($-\log K_a$)** results in a value called the **pK_a** . The pK_a scale is the same as the pH scale, with values ranging from 0 to 14. Weak acids with very small K_a have large pK_a values. The pK_a of a weak acid can be determined from a titration curve. It is equal to the pH of the solution at the point of half-neutralization. The half-neutralization point occurs at where the volume of base added is exactly half the value of the equivalence point.

The **buffering effect** in the early portion of the titration of a weak acid with a strong base occurs because of the **ability of a weak acid to absorb OH^- ions, causing the pH of the solution to remain constant**. The half-equivalence point occurs in the middle of the buffering region. The added OH^- ions react with H^+ in solution to form water in a neutralization reaction, thereby reducing the concentration of H^+ in solution. The equilibrium shifts to the right. The remaining HA molecules in solution dissociate into H^+ and A^- , replacing the H^+ ions that have reacted with the added OH^- .



reacts with hydroxide

In Part 1 of this experiment, a weak acid, acetic acid, will be titrated with a standardized strong base, $NaOH$. The titration will continue to a pH well past the equivalence point. The CBL

system equipped with a pH electrode will be used to monitor the real-time progression of the titration. The printed titration curve will be used to determine the equivalence point and the pK_a of the acid.

In Part 2, an unknown weak acid will be titrated with the same strong base used in Part 1. The unknown acid may be mono, di, or triprotic. The pK_a value(s) of the weak acid and the point(s) of neutralization will be determined from a graph of the titration curve. The identity of the unknown acid will be determined from a list of possible unknowns.

MATERIALS and EQUIPMENT:

Solutions

phenolphthalein	aqueous weak acid unknowns
buffers pH 4, 7 and 10	0.20 M sodium hydroxide (NaOH)
0.10 M acetic acid (HC ₂ H ₃ O ₂)	

Stockroom equipment

pH electrode	CBL system with amplifier
TI-86 calculator	pipet pump

Locker equipment

10.00 mL pipet	beakers - 50 mL and 400 mL
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Laboratory waste disposal:

Laboratory waste container labeled with tracking # 1015B

PROCEDURE:

Part 1: Titration of Acetic Acid with a Strong Base

For laboratory waste disposal, use container labeled with tracking # 1015B.

1. Check out a CBL system with an amplifier and pH electrode and a 25.00 mL buret.
2. Clean and rinse the buret. Fasten the buret to a ring stand with a buret clamp.
3. Fill the buret with 0.2 M NaOH. Adjust the level in the buret so the initial reading is 0.00 mL.
4. Pipet 10.00 mL of 0.100 M HC₂H₃O₂ into a clean, dry and labeled 50 mL beaker.
5. Add 2 drops of phenolphthalein to the solution and note the color of the solution.
6. Set up the calculator and CBL unit for pH measurement.
 - a. Select SET UP PROBES (F1) from the main menu.

- b. Enter **1** as the number of probes.
 - c. Select **pH (F2)** from the **SELECT PROBE** menu.
 - d. Enter **1** as the channel number.
 - e. Select **Perform calibration (F2)**.
7. Calibrate the electrode (allow the system to warm up for 30 seconds, first):
- a. Rinse the electrode with a stream of distilled water from the wash bottle and gently blot dry with a paper towel.
 - b. Place the electrode in **pH 4.00** buffer. Stir gently until the reading on the CBL unit is stable and then press **[TRIGGER]**. (Note: the reading is a voltage reading, not a pH reading.)
 - c. Enter the pH value of the buffer into the TI-86.
 - d. Rinse the electrode with a stream of distilled water from the wash bottle and gently blot dry with a paper towel.
 - e. Place in electrode in **pH 7.00** buffer. Stir gently until the reading on the CBL unit is stable. Press **[TRIGGER]**.
 - f. Enter the pH value of the buffer into the TI-86.
8. Prepare the calculator to collect data:
- a. Select **COLLECT DATA (F2)** from the main menu.
 - b. Select **PROMPT TRIGGER (F3)** from the **DATA COLLECTION** menu.
 - c. Press **ENTER** after the system has warmed up for 30 seconds. The pH reading is displayed on the screen of the CBL, but on reading will be stored until **[TRIGGER]** is pressed on the CBL unit.
9. Titrate the acid.
- a. Rinse the electrode with a stream of distilled water from the wash bottle and gently blot dry with a paper towel.
 - b. Place the electrode in the beaker of acid.
 - c. Position the tip of the buret just inside of the beaker.

- d. Press [TRIGGER]. This is the initial pH reading. Enter the initial buret reading into the TI-86 (should be 0.00 ml).
- e. Immediately press **F1** to prepare for another data point.
- f. Add 2 to 4 drops of titrant from the buret into the acid solution in the 50 mL beaker. Stir gently with the electrode for about 20 seconds until the pH reading on the CBL stabilizes.
- g. When the pH has been stable for about 5 seconds, press [TRIGGER] to store the pH value. Enter the corresponding buret reading into the TI-86.
- h. Immediately press **F1** to prepare for another data point.
- i. Repeat steps f – h to continue the titration. Note the pH value when the solution changes color. Be consistent with timing between readings. When approaching the equivalence point of the titration, reduce additions of base to one drop at a time (this should only be 3 to 4 points).
- j. After the equivalence point, resume adding 2 – 4 drops of base until the pH reaches approximately 12.0. Press **F2** to end the titration.
- k. If the message “repeat trial” appears, answer no (**F1**). If you answer yes, all of the stored data for the previous run will be cleared!

10. Transfer the data to Excel.

- a. The pH values and buret readings are stored as L1 and L2 in the **LIST** menu of the TI-86. Transfer both lists to two columns on a new Excel spreadsheet.
- b. Title and format the columns.
- c. Label the Excel spreadsheet “ACETIC ACID”. Save the file.

11. Dispose of laboratory waste by pouring solutions into laboratory waste container labeled with tracking # 1015B.

Part 2: Identification of An Unknown Weak Acid

For laboratory waste disposal, use container labeled with tracking # 1015B.

1. Obtain an unknown weak acid solution from the instructor. Record the number.
2. Repeat steps 1 - 11 of Part 2, using the unknown weak acid solution this time. Label the Excel spreadsheet “UNKNOWN”. Save the file.

3. Dispose of laboratory waste by pouring solutions into laboratory waste container labeled with tracking # 1015B.

DATA ANALYSIS:

Part 1: Titration of a Weak Acid with Strong Base

1. In Excel, load the "ACETIC ACID" file. Plot an XY(Scatter) graph with data points connected by smoothed lines.
 - a. The buret readings should be on the X axis and pH on the Y axis.
 - b. Convert the plot area from gray to white.
 - c. Locate the graph as a new sheet, Chart 1.
 - d. Print one copy of the graph.
2. Locate the equivalence point on the curve. Draw a line from the equivalence point down to the X axis. This is the volume of titrant needed to neutralize the acid in solution. Record the volume of titrant on the X axis.
3. On the X axis, locate the point that is exactly half of the volume recorded in step 2. This value corresponds to the volume of titrant needed to neutralize half of the acid. Draw a line from this point on the X axis to where it intersects the curve (the half – equivalence point).
4. Draw a line from the half – equivalence point to the Y axis. Read the pH value that corresponds to the half-equivalence point. This new value is the pH of the solution at exactly halfway to the equivalence point, and is equal to the pK_a of acetic acid.
5. Calculate the experimental value of K_a for acetic acid.
6. Compare the experimental value of K_a to the accepted value by calculating the percent error.

Part 2: Identification of An Unknown Weak Acid

1. Repeat steps 1 - 4 of Part 2, using the "UNKNOWN" file.
2. From the pK_a value obtained in step 4, determine the K_a of the unknown weak acid.
3. Determine the identity of the unknown weak acid from the following list.

<u>ACID</u>	<u>K_a</u>	<u>ACID</u>	<u>K_a</u>
Boric acid	7.3 x 10 ⁻¹⁰ 1.8 x 10 ⁻¹³ 1.6 x 10 ⁻¹⁴	K ₂ HPO ₄	4.8 x 10 ⁻¹³
Methanoic acid	1.77 x 10 ⁻⁴	KH ₂ PO ₄	6.2 x 10 ⁻⁸
Ethanoic acid	1.76 x 10 ⁻⁵	NaHCO ₃	5.61 x 10 ⁻¹¹
Propanoic acid	1.34 x 10 ⁻⁵	H ₂ SO ₃	1.54 x 10 ⁻² 1.02 x 10 ⁻⁷
Oxalic acid	5.6 x 10 ⁻² 5.1 x 10 ⁻⁵		
Butanoic acid	1.54 x 10 ⁻⁵		
Lactic acid	8.4 x 10 ⁻⁴		

Appendix B

Examples of New Technology Based Experiments

CHEM 1A

Introduction to the Calculator Based Laboratory System

ChemR₂Y Solutions

CHEM 1B

Kinetics of the Crystal Violet Reaction Using Initial Rates of Reaction

Kinetics of the Crystal Violet Reaction Using Integrated Rate Laws

Chemical Kinetics

Buffers

Introduction to the Calculator Based Laboratory System

Experiment

13

PURPOSE:

The purpose of this experiment is to provide the student with an introduction to the operation of one type of instrumentation, the Calculator Based Laboratory System, used in the Mt. SAC CHEM 1A and CHEM 1B courses.

DISCUSSION:

Many experiments conducted in chemistry laboratory require some sort of instrumentation data collection. A Calculator Based Laboratory (CBL) System provides a user-friendly method of collecting data. A CBL system can perform the functions of a multitude of instruments commonly used in chemistry laboratories such as thermometers, pH meters, manometers and spectrophotometers. Unlike these instruments, data can be automatically stored by a CBL as an experiment is performed. The data can then be downloaded into a computer spreadsheet program for analysis. The CBL system used by the Mt. SAC Chemistry Department consists of a **Texas Instrument - 86 (TI-86)** calculator, a CBL interface unit, and several choices of sensors and probes. **Microsoft Excel (Excel)** is the spreadsheet program that will be used to process and analyze data.

The TI-86 calculator connects interactively with the CBL system using a program called CHEMBIO. The program provides commands to the CBL unit that corresponds to the type(s) of probe being used, calibration of probes, what kind of data to collect and how often to collect it. CHEMBIO issues a series of prompts on the TI-86 screen that allows the user to easily enter choices for data collection choices.

This experiment will help students to develop familiarity with the CBL system. The thermistor sensor will be used to collect temperature data. Acquiring competence with any type of equipment requires time and patience. Read the entire procedure carefully **before** beginning to set up anything. Boldfaced words in the procedure section direct the user to press those keys on the calculator, i.e., **ENTER** means to press the ENTER key on the calculator. Words that appear in square brackets direct the user to press that key on the CBL unit. For example, [trigger] means to press the TRIGGER key on the CBL unit.

Words that appear *italicized* direct the user to press that key on the computer or mouse. For example, *ENTER* means to press the Enter key on the computer. Words that appear in quotation

marks are choices on a menu or dialog box. For example, "File" is found on the Excel menu toolbar.

The mouse has two buttons. *Click* directs the user to use the mouse to place the cursor on an icon or selected word on a menu and quickly press the left button. This is the most common use of the mouse. *Double-click* refers to pressing the left mouse button twice in rapid succession. *Right-click* refers to pressing the right mouse button. This is the least common use of the mouse. *Drag* refers to holding down the left mouse button until the cursor is moved to a desired location.

MATERIALS & EQUIPMENT:

Solids

Ice

Computer equipment

IBM compatible computer with TI Graph Link cable connect to serial port 2
Graphical Analysis for Windows program
Microsoft Excel spreadsheet program

Stockroom equipment

CBL system

Thermistor

Locker equipment

250 mL beaker
formatted computer disk

400 mL beaker

PROCEDURE:

Part 1: Setting up and using the CBL system

1. Check out a TI-86 calculator, CBL unit and thermistor.
2. Fill a 250 mL beaker half full with hot water.
3. Fill a 400 mL beaker half full with tap water. Add ice and allow it to melt while setting up the CBL system.
4. Connect one end of the black link cable to the port on the bottom of the TI-86 calculator and the other end to the bottom of the CBL unit. **Firmly** press the connectors into the ports.
5. Connect the thermistor to the DIN adapter. Connect the DIN adapter to the channel 1 port on the top of the CBL system.

6. Turn on both the TI-86 and the CBL unit.
7. Load the CHEMBIO program on the calculator.
 - a. Press **PRGM**. The program menu will appear on the bottom of the screen.
 - b. Press the key under NAMES (**F1**). A line of program names will appear under the program menu.
 - c. Select CHEMB by pressing **F1** and then **ENTER**. The Vernier CHEMBIO welcome screen will appear. Press **ENTER** again to get to the main menu.
 - d. Press **F1** to select SETUP. You will be asked to enter:
 - (i) the number of probes. Press **1** and then **ENTER**.
 - (ii) the type of probe(s). Press **F1** to select TEMP as the type of probe for this experiment.
 - (iii) the lowest channel number (the channel that the probe is connected to). Press the **1** key and then **ENTER** to select channel 1. The calibration menu will appear.
 - (iv) Press **F2** to perform a new 2-point calibration. Directions will appear on the TI-86 screen. Place the thermistor and a thermometer into the cold water (pt. 1). When the voltage readings on the CBL unit are stable press [TRIGGER]. Enter the thermometer reading into the calculator as the reference and then **ENTER**. Place the thermistor and thermometer into the hot water (pt. 2). When the voltage readings on the CBL unit are stable press [TRIGGER]. Enter the thermometer reading into the calculator and then **ENTER**.
 - e. Press **F2** to select COLLECT DATA. You will be asked to choose:
 - (i) the type of input. Press **F2** to select TIME GRAPH, which display data from this experiment in a real time graph on the calculator screen. Time will be on the X-axis and temperature on the Y-axis
 - (ii) the time, in seconds, between samples (temperature measurements). Enter **2**, but this can be changed as needed for other experiments.
 - (iii) the number of samples. Choose **75** and then **ENTER**, but this can be changed as needed for other experiments. The screen will recap the choices. Press **ENTER** again.

- (iv) The next screen allows changes to be made to the original choices. If everything is correct, choose **USE T (F1)**. If not, choose **MODIF (F2)** and reselect the time and number of samples.
 - (v) Since a real time graph will be displayed on the TI-86 screen during data collection, the temperature values and scale for the Y-axis need to be chosen. Set the minimum y (yMin) value at **10**, the maximum y(yMax) value as **60** and the scale (yScl) as **5**. Press **ENTER** after each selection.
- f. The CBL system is now ready to collect data. The temperature measurements are displayed on the CBL unit screen as the program runs. Record the constant temperature value at each plateau.
- (i) Place the thermistor in the cold water and press **ENTER** to start data collection. Stir gently until the temperature values become constant (after ~10 samples).
 - (ii) Move the thermistor into the hot water. Continue stirring gently until the temperature value becomes constant.
 - (iii) Move the thermistor back into the cold water until the program is done.
- (i) When the program is complete, **DONE** will be displayed on the left side of the CBL screen. The temperature and time data has been stored as lists in the TI-86. The names of the data files will be displayed on the calculator screen. Record these names.
- (ii) Press **ENTER** to see a graph of the data.
 - (iii) Press **ON** to end the program and then choose **QUIT (F5)** to return to the calculator functions.
- g. The data now needs to be renamed so that it will not be written over during subsequent trials.
- (i) Select the **LIST** option on the TI-86 by pressing **2ND** (orange key) and then **LIST**. The **LIST** menu will appear on the bottom of the screen.
 - (ii) Select **NAMES (F3)**. The names of stored data lists will appear under the **LIST** menu.
 - (iii) Press the **F** key under the list containing the time data and then **ENTER**. The first few time intervals will appear.

- (iv) Press **STOP?**. The cursor should now contain a blinking "A". The alphabet option (in blue above each key) is now active. Type in a name for the time data. Press **ENTER** when finished. Be sure to record the new name of the data file.
- (v) Repeat steps (i) through (ii) to rename the temperature data file.
- h. Repeat Part 1 until you can comfortably use the CBL system to collect and store temperature data.

Part 2 Downloading The Data

1. Both the calculator and computer need to be prepared for the transfer of data. Connect the gray TI-Graph Link cable to the bottom of the TI-86 calculator.
2. The data will be analyzed using the Microsoft Excel program after it has been transferred to the computer. Open Microsoft Excel. **Click** the minimize button. The name of the program will appear on the status bar on the bottom of the screen since the program is still open.
3. Open the Graphical Analysis for Windows program from the "Vernier Software" folder of the Program menu.
 - a. **Click** on the column to where the first data list will be imported.
 - b. Go to the **FILE** menu and **click** on "Import from TI Calculator". A dialog box will open. Select "COM2" as the serial port. The dialog box will remain on the screen until the data is transmitted.
4. Turn on the TI-86 and activate the **LINK** feature (**2nd**, then **LINK**). The **LINK** menu will appear on the bottom of the screen.
 - a. Select **SND85 (F3)** and then **LIST (F2)**. A catalog of stored data lists will appear on the screen.
 - b. Scan the list, using the **▲** and **▼** keys until the cursor is pointing to the name of the data file where "time" was stored. Select this file by pressing the **F2** key.
5. Transmit the data from the TI-86 to Graphical Analysis for Windows by pressing the **XMIT (F1)** key on the calculator. The data will be copied onto the computer screen in the preselected column.
6. The data file will now be copied from Graphical Analysis to Excel .
 - a. **Click** on the top of the data column so that the entire column of data is highlighted.

- b. Go to the Edit menu and *click* on "Copy Data".
 - c. Reactivate Excel by *clicking* on the Excel icon at the bottom of the screen. A blank Excel workbook will appear.
 - d. *Click* on the letter at the top of a column in the Excel workbook so that an entire empty column is highlighted.
 - e. *Right click*. An edit menu will appear. *Click* "Paste". The data will be copied into the selected column in the Excel workbook.
 - f. Save the Excel workbook on the student disk.
7. Repeat steps 3 through 6 to transfer the temperature data from the TI-86 to Excel. Copy the data into a column in the same Excel workbook as the time data.
 8. Close the Graphical Analysis program. It is not necessary to save the files with this program since they have been saved as an Excel workbook.

DATA ANALYSIS

1. Open the Excel workbook containing the time and temperature data files.
2. Label and format the columns.
3. Use the Chart Wizard to plot an XY scatter graph of the data.
 - a. Time should be on the X-axis and temperature should be on the Y-axis. Rescale the plot as necessary so that the data fills the graph area.
 - b. Include all appropriate labels and titles as well as major X and Y gridlines.
 - c. Convert the plot area from gray to white. Print one copy of the graph. Use a photocopy machine to make more copies, if needed.
4. Use the printed graph to identify the plateaus where the temperature values are constant.
 - a. Calculate the initial change in the temperature when the thermistor was moved from the cold water to the hot water.
 - b. Calculate the final temperature change when the thermistor was moved back into the cold water.

ChER₂Y Solution

Experiment

17

PURPOSE:

The purpose of this experiment is to familiarize the student with some techniques and equipment used to make and analyze solutions. A stock solution of known molarity will be prepared. A series of dilutions will then be made from the stock solution. The amount of light absorbed by the diluted solutions will be measured using a calculator-based laboratory (CBL) system. A Beer's law plot will be prepared by graphing absorbance data. The concentrations of unknown solutions will then be determined.

DISCUSSION:

A **solution** is defined as a homogenous mixture of a solute or solutes in a solvent. Bottles containing solutions are common in chemistry laboratories. Proper labels on solution bottles reflect name and the concentration of the solute, i.e., 1.5 *m* C₆H₁₂O₆, 10% NaOH, 0.10M NaCl. The **concentration** of a solution expresses the amount of solute in a given quantity of solvent or solution. The solvent is assumed to be water (aqueous solution), unless otherwise indicated.

There are many types of concentration units. There are three different types of percents. **Percent by mass** is defined as the mass of solute per 100 grams of solution. **Percent by volume** refers to the volume of (liquid) solute per 100 mL of solution. **Mass/volume percent** is defined as the mass of solute per 100 mL of solution. **Molality (*m*)** is the number of moles of solute dissolved per one kilogram of solvent. **Molarity (*M*)**, one of the most commonly used units in chemistry, is the number of moles of solute dissolved in one liter of solution. This experiment will focus on molarity:

$$M = \text{Molarity} = \frac{\text{moles of solute}}{\text{liters of solution}} \quad (1)$$

Suppose that a chemist wishes to prepare 500 mL of an aqueous solution with a solid solute. A series of steps will be followed to complete the task. (A) The amount of solute to be dissolved is calculated by rearranging equation (1):

$$\text{Moles of solute} = (\text{Molarity of solution}) (\text{liters of solution}) \quad (2)$$

The moles of solute is converted to mass (usually in units of grams) using the molar mass of the solute. A balance can then be used to obtain the number of grams of solute needed. (B) Once the solid has been weighed, it is completely transferred to a piece of volumetric glassware such as a graduated cylinder or volumetric flask with a mark indicating 500 mL. (C) The glassware is filled about halfway with distilled water and then shaken to dissolve the solute. Additional small quantities of water are added as needed until the solute dissolves. (D) Once the solute has dissolved, distilled water is carefully added until the meniscus reaches the 500 mL calibration mark. Notice that the exact amount of water added is not known, only that enough was added to prepare 500 mL of solution. (E) The solution is transferred to a container and labeled with the correct name and concentration of the solute. In Part 1, 250 mL of 0.135 M ChER_2Y solution will be prepared in a volumetric flask. ChER_2Y is a red powder with a molar mass of 15.74 g/mol. This solution will be used as a stock solution to prepare diluted standard solutions in Part 2.

Fertilizers and pesticides are sometimes packaged as concentrated solutions to lower shipping costs. They must be carefully diluted to lower concentrations to avoid damaging the plants to which they will be applied. Many medications are also packaged as concentrated solutions and then diluted before administration to patients. Incorrect dilution can cause the patient to receive the wrong dosage. **Dilution** is the process of preparing a less concentrated solution from a more concentrated (stock) solution by adding additional solvent. The following equation is used to calculate the quantities needed to prepare dilutions:

$$(M_{\text{initial}})(V_{\text{initial}}) = (M_{\text{final}})(V_{\text{final}}) \quad (3)$$

M_{initial} is the molarity of the stock solution, before dilution. M_{final} is the molarity of the solution after dilution. V_{initial} is the volume of the stock solution used to make the dilution. V_{final} is the final volume after the dilution is complete. Equation (3) will be used in Part 2 to calculate the amount of stock solution (from Part 1) needed to prepare a series of diluted standard solutions ranging from 0.0090 M ChER_2Y to 0.045 M ChER_2Y .

Companies whose products are solutions must assure that the solutions are the correct concentration before they are delivered to consumers. These companies employ quality control chemists to analyze the solutions. Solutions that contained colored species absorb specific wavelengths of light and can be analyzed spectrophotometrically. The amount of light transmitted through a colored solution decreases as the concentration of the colored species increases.

An instrument that measures the amount of light that is transmitted through a solution is called a **spectrophotometer**. A spectrophotometer separates light into its component wavelengths and is able to selectively measure the intensity of a specific wavelength after it passes through a solution. The basic components of a spectrophotometer include a light source, a device to hold samples, a prism or grating to separate light into component wavelengths, a photoelectric cell to measure light intensity and a readout that displays data. A Vernier **colorimeter**, a very simple spectrophotometer, is used in this experiment. The colorimeter has diodes that produce three specific wavelengths (470, 565, and 650 nm) of light, instead of a prism or grating. ChER_2Y solutions absorbs light at 470 nm.

Transmittance, T, is defined as the ratio of the intensity of light emerging from the solution (I), and the intensity of the light entering the solution (I₀). **Percent transmittance, %T**, is defined as T x 100:

$$T = \frac{I}{I_0} \qquad \%T = \frac{I}{I_0} \times 100$$

Absorbance, A, is defined as a function of transmittance:

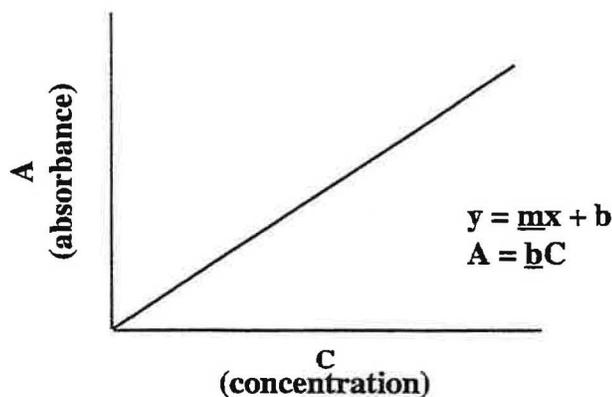
$$A = -\log T \qquad \text{or} \qquad A = -\log \frac{I}{I_0}$$

The relation between A and %T is simple. When no light is absorbed by a solution, 100 % of the light is transmitted. When all of the light is absorbed by the solution, 0% is transmitted.

Beer's Law states that the absorbance A of a colored species in solution is proportional to the concentration, C, according to the equation:

$$A = bC$$

B is a **proportionality constant** called **Beer's constant**, and it is for a given substance at a particular wavelength of light. The units of b are M⁻¹ if C is in units of molarity. The equation of Beer's Law is in the form of a straight line. A **standard absorbance curve** can be prepared by measuring the absorbance of a series of solutions of varying concentrations. A plot of absorbance versus concentration is made.



The line should pass through the origin. The slope of the graph is equal to **b, Beer's constant**. A linear regression trendline can be used to calculate the value of the slope. The absorbances of the diluted standards prepared in Part 2 will be measured. The data will be transferred to a Microsoft Excel workbook and a scatter graph will be plotted. The resulting graph will be used to determine the concentrations of two ChER₂Y solutions of unknown concentration.

MATERIALS & EQUIPMENT:

Solids

ChER₂Y powder

Stockroom equipment

CBL system with colorimeter

5 mL graduated pipet

Kimwipes

cuvette

pipet pump

volumetric flasks (25 & 250 mL)

Locker equipment

Spatula

250 mL beaker

test tubes and rack

Florence flask (500 mL or 1000 mL)

wash bottle filled with distilled water

PROCEDURE:

Part 1 Preparing stock solution

1. Check out a 250 mL volumetric flask from the instructor. Clean the flask and then rinse with distilled water. It does not need to be dry.
2. Calculate the number of grams of ChER₂Y powder needed to prepare 250 mL of a 0.135 M solution.
3. Use an analytical balance to weigh the amount of solid calculated in step 2 into a clean, dry 250 mL beaker. Get as close as possible to the calculated amount, to the highest precision of the balance. Record the amount of solid.
4. Add approximately 100 mL of distilled water to the powder in the 250 mL beaker. Swirl the beaker to mix. Pour the mixture into the volumetric flask.
5. Rinse any ChER₂Y remaining powder the beaker with a small portion of distilled water, pouring the rinse solution into the volumetric flask. Continue rinsing until all red residue from the powder has been transferred from the beaker into the volumetric flask.
6. Add distilled water to the flask until the level reaches the base of the neck. Cap or stopper the flask, invert and shake the flask until all solid is dissolved.
7. Use a wash bottle containing distilled water to carefully fill the volumetric flask to the mark on the neck. Use a dropper to add the last amount of distilled water. Cap the flask again, invert and shake the flask for at least a minute to mix thoroughly.
8. Transfer the solution to a clean, dry Florence flask and stopper it. Label the flask "Stock 0.135 M ChER₂Y powder Solution". Save the solution for Part 2.
9. Clean the volumetric flask and return it to the instructor.

Part 2 Preparing Diluted Standards and Unknown ChER₂Y Solutions

1. Check out a 5 mL graduated pipet and a 25 mL volumetric flask from the instructor.
2. Label 8 clean, dry test tubes #0 to #7.
3. Use equation (3) to calculate the amounts of stock ChER₂Y solution needed to prepare a series of diluted standard solutions from the stock solution. The final volume of each diluted solution is 25 mL. The concentrations of the dilutions are:

<u>Test tube #</u>	<u>Molarity (mol/L) of diluted ChER₂Y solution</u>
0	0.0000
1	0.0090
2	0.0180
3	0.0270
4	0.0360
5	0.0450

4. Test tube #0 will be the blank, used to set the colorimeter to 100% transmittance. Fill it with distilled water.
5. Pipet the amount of ChER₂Y stock solution calculated for test tube #1 into a 25 mL clean volumetric flask that has been rinsed with distilled water.
6. Add distilled water to the flask up to the base of the neck. Stopper and shake thoroughly.
7. Use a dropper to add the last amount of distilled water to the flask. Cap the flask again, invert and shake the flask for at least a minute to mix thoroughly.
8. Pour 15-18 ml of the solution into test tube #1. (Not all of the solution will fit.)
9. Thoroughly rinse the flask with distilled water until all traces of red are removed.
10. Repeat steps 5 through 9 for test tubes #2 - #5. Clean the volumetric flask and return it to the instructor.
11. Take test tubes #6 & #7 to the instructor to obtain two unknown ChER₂Y solutions. Record the numbers.

Part 3 Measuring The Absorbance Of ChER₂Y Solutions

1. Checkout a CBL system with colorimeter and a cuvette from the instructor.
2. Connect one end of the black link cable to the port on the bottom of the TI-86 calculator and the other end to the bottom of the CBL unit. **Firmly** press the connectors into the ports.

3. Connect the colorimeter to the DIN adapter. Connect the DIN adapter to the channel 1 port on the top of the CBL system.
4. Turn on both the TI-86 and the CBL unit.
5. Load the CHEMBIO program on the calculator:
 - a. Press **PRGM**. The program menu will appear on the bottom of the screen.
 - b. Press the key under NAMES (**F1**). A line of program names will appear under the program menu.
 - c. Select CHEMB by pressing **F1** and then **ENTER**. The Vernier CHEMBIO welcome screen will appear. Press **ENTER** again to get to the main menu.
6. **SETUP** the CBL system by pressing **F1**. You will be asked to select:
 - a. the number of probes. Press **1** and then **ENTER**.
 - b. the type of probe(s). Press **F4** to select COLOR (colorimeter) as the type of probe.
 - c. the lowest channel number (the channel that the probe is connected to). Press **1** and then **ENTER** to select channel 1. The calibration screen will appear.
 - d. A 2-point calibration is required when the colorimeter is used.
 - i. Turn the knob on the colorimeter to 0% transmittance (point 1). When the knob is in this position, no light is transmitted to the photocell. When the voltage readings on the CBL unit are stable press [TRIGGER]. Enter **0** (for 0% transmittance) into the calculator as the reference and then **ENTER**.
 - ii. Turn the knob on the colorimeter to 470 nm. Fill the cuvette with the blank solution (test tube # 0). Wipe the outside of the cuvette with a Kimwipe. Place the cuvette into the colorimeter with the black edge facing forward. When the voltage readings on the CBL unit are stable press [TRIGGER]. Enter **100** (for 100% transmittance) into the calculator and then **ENTER**. The intercept and slope of the calibration curve will appear on the screen. Record these values. Press **ENTER** to return to the Main Menu.
7. **COLLECT DATA** by pressing **F2**:
 - a. You will be asked to choose the type of input. Press **F4** to select TRIGGER. The CBL unit will read and store absorbance and transmittance data when [TRIGGER] is pressed. The CBL system is now ready to collect data.

8. Measure the absorbance of each solution using the colorimeter.
 - a. Remove the cuvette from the colorimeter and discard the solution into a 400 mL beaker that will be used to collect waste.
 - b. Rinse the cuvette twice with 2 mL portions of the next solution to be read. Discard these rinse solutions into the waste beaker.
 - c. Fill the cuvette H full with the solution.
 - d. Wipe of the outside of the cuvette with a Kimwipe.
 - e. Insert the cuvette into the colorimeter with the black edge forward and close the cover.
 - f. Press [TRIGGER] on the CBL unit to record and store data.
 - g. Select MORE (F1) if more samples will be measured and repeat steps a through f. Select STOP (F2) after the last sample has been read. Be sure to immediately remove the cuvette from the colorimeter after the last sample has been read.
 - h. Record the names of the lists where the TI-86 has stored the data files.
9. Discard of the ChER_2Y solutions down the sink. Clean the cuvette until all traces of red are gone.

Part 4 Downloading The Data

1. Both the calculator and computer need to be prepared for the transfer of data. Connect the gray TI-Graph Link cable, connected to the back of the computer, to the bottom of the TI-86 calculator.
2. The data will be analyzed using the Microsoft Excel program after it has been transferred to the computer. Open Microsoft Excel. *Click* the minimize button. The name of the program will appear on the status bar on the bottom of the screen since the program is still open.
3. Open the Graphical Analysis for Windows program from the "Vernier Software" folder of the Program menu.
 - a. *Click* the column to where the first data list will be imported.
 - b. Go to the file menu and *click* on "Import from TI Calculator". A dialog box will open. Select "COM2" as the serial port. The dialog box will remain on the screen until the data is transmitted.

4. Turn on the TI-86 and activate the LINK feature (2nd, then LINK). The LINK menu will appear on the bottom of the screen.
 - a. Select SND85 (F3) and then LIST (F2). A catalog of stored data lists will appear on the screen.
 - b. Scan the list, using the \blacktriangle and \blacktriangledown keys until the cursor is pointing to the name of the data file where "absorbance" was stored. Select this file by pressing the F2 key.
5. Transmit the data from the TI-86 to Graphical Analysis for Windows by pressing the XMIT (F1) key on the calculator. The data will be copied onto the computer screen in the preselected column.
6. The data file will now be copied from Graphical Analysis to Excel .
 - a. *Click* the top of the data column so that the entire column of data is highlighted.
 - b. Go to the Edit menu and *click* "Copy Data".
 - c. Reactivate Excel by *clicking* the Excel icon at the bottom of the screen. A blank Excel workbook will appear.
 - d. *Click* the letter at the top of a column in the Excel workbook so that an entire empty column is highlighted.
 - e. *Right click*. An edit menu will appear. *Click* "Paste". The data will be copied into the selected column in the Excel workbook.
 - f. Save the Excel workbook on the student disk.
7. Close the Graphical Analysis program. It is not necessary to save the files with this program since they have been saved as an Excel workbook.

DATA ANALYSIS:

1. Open the Excel workbook containing the absorbance data file.
2. Label and format the columns.
 - a. Label the first column containing the absorbance data. Format the absorbance data to four places past the decimal point.
 - b. Label the next column "Test Tube #". Enter the values 0-7, corresponding to the absorbance data.

- c. Label the third column "Solution Concentration". Enter the solution concentration values (Part 2, step 3) that correspond to the test tube # 0-7. Note that there are no solution concentration values for test tubes #6 and #7 (the unknowns). Format the data to the appropriate number of significant figures.
 3. Use the Chart Wizard to plot a standard absorbance curve from the **concentration** and **absorbance** data.
 - a. *Click* the Chart Wizard icon.
 - b. Step 1 – Select XY (Scatter) graph.
 - c. Step 2 – *Click* the "Series" tab to select "Solution concentration" as the X-axis and "absorbance" as the Y-axis. Do not include the absorbance data of the two unknowns (tubes #6 & # 7) on this plot.
 - d. Step 3 – Include all appropriate labels and titles as well as major X and Y gridlines.
 - e. Step 4 – locate the chart as an object in sheet 1
 4. Add a linear regression trendline, equation and regression coefficient to the standard absorbance curve.
 - a. *Click* "Chart" on the Excel Menu Bar. *Click* "Add Trendline" and then "Linear".
 - b. *Click* the "Options" tab. *Click* "Display equation on chart" and then "Display R-squared value on chart". A check mark will appear in the boxes next to these two choices.
 - c. *Click* "OK". The computer will draw the best straight line that fits the data points on the chart. The equation of this line and the regression coefficient (R) will also appear on the chart.
 5. Convert the plot area from gray to white. Print one copy of the graph. Use a photocopy machine to make more copies, if needed.
 6. Use the printed graph to identify the concentrations of the two unknown solutions.
 - a. Locate the absorbance value of the first unknown solution (in test tube #6) on the Y axis. Draw a straight line, parallel to the X axis, from the Y axis to the linear regression trendline. At that point draw a straight line, parallel to the Y axis, down to the X axis. Read the concentration value from the X-axis.
 - b. Repeat the process for the second unknown (test tube #7).

- c. Obtain the true concentration values of the unknown ChER_2Y solutions from the instructor. Calculate the percent error of the two experimental values.

QUESTIONS:

1. Convert the molarity of the diluted standard solutions made in Part 2 to % mass/volume.
2. What information is needed to convert the molarity of the stock solution to molality?
3. Review how the stock ChER_2Y solution was prepared in Part 1. Would it be correct to weigh the ChER_2Y powder and then add 250 mL of water to the powder? Explain your reasoning.
4. The package of a medication states that the concentration is 5.00% mass/volume. A doctor orders a patient to receive 0.080 grams of the medication in a 10 mL injection. How should you prepare the patient's medication?
5. A curious student determines that the density of the ChER_2Y stock solution is 1.023 g/mL. At what temperature should the ChER_2Y solution freeze?

Kinetics of the Crystal Violet Reaction using Initial Rates of Reaction

Experiment

2

PURPOSE

The purpose of this experiment is to determine the order for the reaction of crystal violet with sodium hydroxide. The initial rate of reaction will be determined for several trials, using two different concentrations of sodium hydroxide. The data will then be compared to determine the order of reaction.

DISCUSSION

Kinetics is the area of chemistry that concerns rates of reactions. The rate at which a chemical reaction takes place depends on the following factors: (1) the inherent properties of the reacting materials, (2) the temperature, (3) the concentration of the reactants, and (4) the presence of catalyst(s). In this experiment, the effects of concentration in relation to reaction rate will be studied.

Reaction Order

To introduce the concept of the rate or speed of a reaction, let's consider the following hypothetical reaction where D and G are reactants, P is a product, and d, b and p are numerical coefficients.



The rate law represented by equation (2) expresses rate as a function of concentration and is called the **differential rate law**.

$$\text{Rate} = k [D]^n [G]^m \quad (2)$$

Such an expression, which shows how the rate depends on the concentration of reactants, is called a **rate law**. The proportionality constant, k, is called the **rate constant**. The exponents, n and m are called the **order** of the reactants with respect to D and G. They must both be determined **experimentally**. The orders of simple reactions are often positive integers and zero. Note that the concentration of the product does not appear in the rate law because the reverse reaction is considered to be negligible. Again, the orders n and m must be determined by experiment, they cannot be written from coefficients of the balanced equation.

Simple chemical reactions are classified according to their orders. The order of reaction can be determined by adding the exponents. A reaction is considered to be **zero order** if $n = m = 0$. A reaction is considered to be **first order** if $n = 1$ and $m = 0$, or vice versa. A reaction is considered

to be **second order** if $n = 2$ and $m = 0$ (or vice versa) or if $n = 1$ and $m = 1$ (*simple* second order for the former and *mixed* second order for the latter case). One way to determine the order of reaction is using the method of initial rates. The values of the exponents, n and m are determined experimentally.

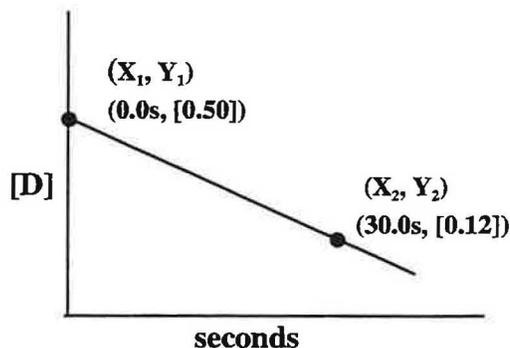
Method of Initial Rates

Chemical reactions, in general, are reversible. The forward and reverse reactions can occur simultaneously. This potentially complicates the kinetics of a chemical reaction. To avoid this complication, experimental conditions are chosen so that the reverse reaction makes only a negligible contribution to the rate. Under these conditions, the reaction rate will depend only on the initial concentrations of the reactants.

The reaction rate is defined as the change in concentration of a reactant or product per unit of time. The Greek symbol delta (Δ) is used to indicate a change in a given quantity. The negative sign in front of the rate of disappearance of a reactant is to define the rate of appearance of a product as a positive quantity. The square brackets ($[]$) are used to indicate the concentration of a solution in units of molarity. A lower case t (t) is used to define time.

$$\text{Rate} = -\frac{\Delta[\text{D}]}{\Delta t} = \frac{\Delta[\text{G}]}{\Delta t} = \frac{\Delta[\text{P}]}{\Delta t} \quad (2)$$

The initial rate of a chemical reaction can be determined experimentally by monitoring the concentration of a reactant or product as the reaction begins, over a very short time interval as the reaction progresses. Let's assume that the concentration of reactant D is monitored as the hypothetical reaction above (1) proceeds. The order with respect to D (the value of the exponent n in equation (2)) will be determined. The data is plotted with time on the X axis and concentration on the Y axis:



The numerical value for the rate of reaction is calculated by determining the slope of the line. Slope is defined as the change in Y divided by the change in X. It can be calculated by selecting two points on the line and calculating the differences in the Y values, divided by the differences in the X values. Since concentration is on the Y axis and time is on the X axis, the equation for the slope of the line is the same as the equation for rate of reaction. The sign will be negative, since the concentration of D is decreasing:

$$\text{Slope} = \frac{\Delta Y}{\Delta X} = \frac{Y_2 - Y_1}{X_2 - X_1} = \frac{\Delta[\text{D}]}{\Delta t} = \frac{[0.12] - [0.50]}{30.0 \text{ s} - 0.0 \text{ s}} = \frac{-[0.38]}{30.0 \text{ s}} = \frac{-0.013 \text{ M}}{\text{second}} \quad (3)$$

The effect of concentration on the reaction can be determined by performing a second trial of the experiment, with reactant D at twice the concentration as in the first trial. The concentration of reactant G is kept constant with the first trial. The data for the second trial is plotted and the rate of reaction determined.

	<u>initial [D]</u>	<u>initial [OH-]</u>	<u>rate (molarity/sec)</u>
Trial 1	0.50	0.50	- 0.013
Trial 2	1.0	0.50	- 0.052

Now, let's compare the effect that doubling the concentration of reactant D has on the rate of reaction. Substitute the data for each trial into equation 2 and compare the rates of reaction:

$$\frac{\text{Rate}_2}{\text{Rate}_1} = \frac{-0.052}{-0.013} = \frac{k [1.0]^n [G]^m}{k [0.50]^n [G]^m}$$

The constant, k, will cancel. Since the concentration of reactant G was kept constant in both trials, [G]^m will also cancel. The equation can then be solved for the value of n:

$$\frac{-0.052}{-0.013} = \frac{[1.0]^n}{[0.50]^n}$$

$$4 = [2]^n$$

$$n = 2$$

The reaction is second order with respect to reactant D. The value of n is substituted into equation 2.

$$\text{Rate} = k [D]^2 [G]^m$$

The order of reaction with respect to reactant G can be determined in a similar manner. Another trial of the experiment is performed, altering the concentration of reactant G while holding the concentration of reactant D constant with Trial 1.

	<u>initial [D]</u>	<u>initial [G]</u>	<u>rate (molarity/sec)</u>
Trial 3	0.50	1.0	- 0.026

The value of exponent m can then be calculated:

$$\frac{\text{Rate}_3}{\text{Rate}_1} = \frac{-0.026}{-0.013} = \frac{k [0.50]^n [1.0]^m}{k [0.50]^n [0.50]^m}$$

$$\frac{-0.026}{-0.013} = \frac{[1.0]^m}{[0.50]^m}$$

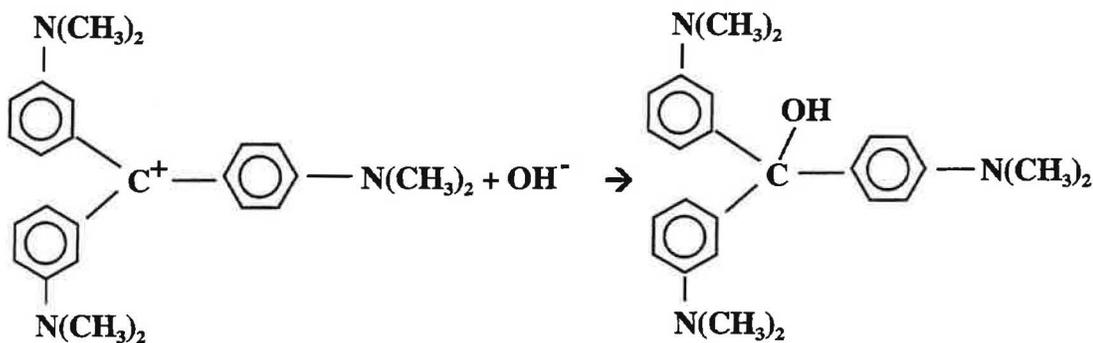
$$2 = [2]^m$$

$$m = 1$$

The orders of both reactants D and G have both been experimentally determined. The reaction is second order with respect to D, first order with respect to G and third order overall.

$$\text{Rate} = k [\text{D}]^2 [\text{G}]^1$$

The system under investigation in this experiment is the reaction of crystal violet, a purple biological stain with sodium hydroxide (colorless) to produce a colorless product.



Beer's Law

Colored species in solution absorb specific wavelengths of light. Crystal violet is a violet solution that absorbs light in the visible region. Its concentration can be easily monitored using a colorimeter. A **colorimeter** is an instrument that measures the amount of light that is transmitted through a solution. A colorimeter emits light of a specific wavelength. It is then able to measure the intensity of the light after it passes through the solution. The basic components of a colorimeter include:

1. a light source (specific wavelengths)
2. a device to hold samples
3. a photoelectric cell to measure intensity
4. a readout that displays data

The colorimeter used in this experiment is a Texas Instrument Calculator Based Laboratory Unit(see Figure 1).

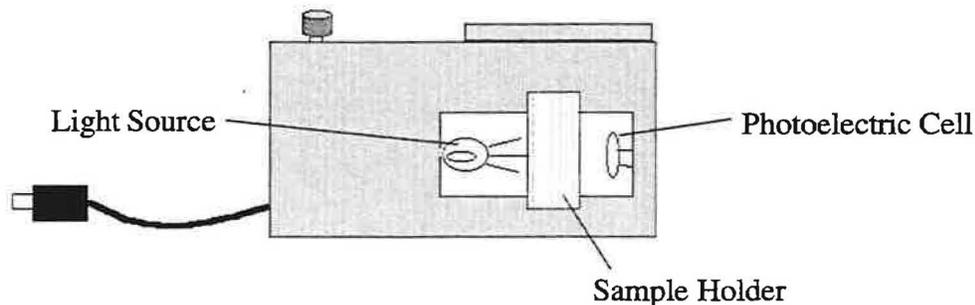
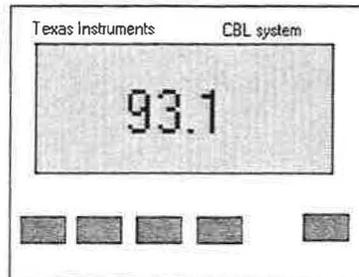


Figure 1 Texas Instruments Colorimeter

Figure 2 Texas Instruments CBL screen

The display on the front of the CBL unit reads percent transmittance.



Transmittance, T, is defined as the ratio of the intensity of light emerging from the solution (I) and the intensity of the light entering the solution (I_0). The ratio is multiplied by 100 to calculate %T.

$$T = \frac{I}{I_0} \quad \% T = \frac{I}{I_0} \times 100$$

Absorbance, A, is defined as the inverse of the log of transmittance:

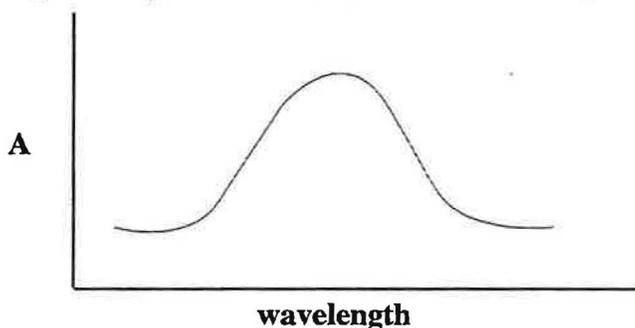
$$A = -\log T \quad \text{or} \quad A = -\log \frac{I}{I_0}$$

Beer's Law states that the absorbance (A) of a colored species in solution is proportional to the concentration (C) where b is a **proportionality constant** called **Beer's constant**:

$$A = bC$$

The value of Beer's constant is specific for a given substance at a particular wavelength of light. The units of b are generally M^{-1} . The amount of light absorbed by a solution is directly proportional to the concentration of the colored species.

The optimum wavelength to measure absorbance of a given substance in solution can be determined by measuring the absorbance of the solution at a series of wavelengths. These absorbance values are plotted against wavelength to produce an optimum absorbance curve:



The peak of the graph, the **maximum absorbance wavelength**, is the most effective wavelength to measure the absorbance of the solution.

Crystal violet is very advantageous for this experiment, because its maximum absorbance wavelength is 565 nm and because the reaction with sodium hydroxide occurs very quickly. For this experiment, the reactants are mixed and then immediately placed in a cuvette, which is promptly inserted into a colorimeter. The absorbance of the solution, which is proportional to the concentration of crystal violet present, is read automatically at preset time intervals. The absorbance and time values are stored in the memory of the TI-86 calculator. Several trials are performed using different concentrations of crystal violet. The rates of the different trials are compared to determine the order of reaction with respect to crystal violet.

MATERIALS & EQUIPMENT

Solids

ice

Solutions

2.0 x 10⁻⁵ M crystal violet

1.0 M NaOH (stock solution)

Stockroom equipment

CBL system with colorimeter

Cuvettes with one edge marked (2)

25 mL Vol flask (2)

Calibrated 10 mL pipet

pipet pump

Locker equipment

10 mL graduated cylinders (2)

100 mL beaker

400 mL beaker

Laboratory waste container

None needed

PROCEDURE

For laboratory waste, solution may be poured into sink with a lot of water.

Part 1 Preparation of 0.50 M NaOH Solution

1. Check out a 25 ml volumetric flask from the instructor.
2. Calculate the amount of stock sodium hydroxide solution needed to prepared 25 mL of 0.50 M sodium hydroxide.
 - a. Measure the amount of stock solution into the volumetric flask.

- b. Add distilled water to the base of the neck.
- c. Stopper the flask.
- d. Hold the stopper and invert the flask. Mix the contents thoroughly.
- e. Add distilled water to the calibration mark.
- f. Hold the stopper and invert the flask. Mix the contents thoroughly

Part 2 Reaction of Crystal Violet with Sodium Hydroxide

1. Check out a CBL system, colorimeter and 2 cuvettes from the stockroom.
2. Obtain reactant solutions:
 - a. Measure 5 mL of 0.50 M NaOH in a 10 mL graduated cylinder
 - b. Measure 5 mL of 2.0×10^{-5} M crystal violet solution into another 10 mL graduated cylinder.
3. Prepare a blank cuvette for calibrating the colorimeter. Fill a clean dry cuvette with distilled water. Carefully dry the outside with a Kimwipe.
4. Load the CHEMBIO program on the calculator:
 - a. Press **PRGM**. The program menu will appear on the bottom of the screen.
 - b. Press the key under NAMES (**F1**). A line of program names will appear under the program menu.
 - c. Select CHEMB by pressing **FI** and then **ENTER**. The Vernier CHEMBIO welcome screen will appear. Press **ENTER** again to get to the main menu.
5. **SETUP** the CBL system by pressing **F1**. You will be asked to select:
 - a. the number of probes. Press **1** and then **ENTER**.
 - b. the type of probe(s). Press **F4** to select COLOR (colorimeter) as the type of probe.
 - c. the lowest channel number (the channel that the probe is connected to). Press **1** and then **ENTER** to select channel 1. The calibration screen will appear.
 - d. A 2-point calibration is required when the colorimeter is used.

- i. Turn the knob on the colorimeter to 0% transmittance (point 1). When the knob is in this position, no light is transmitted to the photocell. When the voltage readings on the CBL unit are stable press the TRIGGER button. Enter 0 (for 0% transmittance) into the calculator as the reference and then **ENTER**.
 - ii. Turn the knob on the colorimeter to 470 nm. Fill the cuvette with the blank solution (test tube # 0). Wipe the outside of the cuvette with a Kimwipe. Place the cuvette into the colorimeter with the black edge facing forward. When the voltage readings on the CBL unit are stable press the TRIGGER button. Enter 100 (for 100% transmittance) into the calculator and then **ENTER**. The intercept and slope of the calibration curve will appear on the screen. Record these values. Press **ENTER** to return to the Main Menu
6. Prepare the CBL system to collect data by pressing **F2**.
 - a. Select the following:

Data Collection – time (**F2**)
Time between samples – 2 sec.
Number of samples – 10

Press **F1** to use the program with the above selections. Press **F2** to modify the choices if necessary.
 - b. Format the TI-86 screen. Enter the following:

Y min – 0 Y max – 1 Yscl – 0.1
7. Remove the blank cuvette from the colorimeter. Keep the blank. It will be used for subsequent calibrations.
8. To begin collecting data, this process must be done quickly, but in a controlled manner.
 - a. Pour the 5 mL portions of crystal violet and sodium hydroxide into the clean, dry 100 mL beaker and swirl to mix.
 - b. Fill the clean, dry cuvette $\frac{3}{4}$ full with the reaction mixture. Wipe the outside of the cuvette with a Kimwipe, if needed.
 - c. Place the cuvette in the cuvette slot of the colorimeter and close the cover.
 - d. Press **ENTER** to begin collecting data.
 - e. When data collection is complete, DONE will appear on the CBL screen.

- f. Record the names of the data files where time, absorbance and transmittance data are stored from the screen of the TI – 86.
 - g. Press **ENTER** to display a graph on the calculator screen.
 - h. If a prompt to repeat the trial choose appears on the screen, choose **NO (F1)**. Choosing **YES (F2)** will overwrite the trial and erase all data.
 - i. Press **ON** to exit the CHEMBIO program.
9. Go to the list menu function on the TI-86 and choose the EDIT function. Record the time and absorbance data from the TI-86 into the lab notebook.
 10. Dispose of the reaction solution by pouring into the sink with a lot of water.
 11. Repeat steps 2 – 10 twice for a total of three trials at the same concentration of sodium hydroxide.
 12. Perform three more trials, using 1.0 M NaOH. A total of six trials will be performed: 3 with two different concentrations of sodium hydroxide.
 13. Delete the data lists of TI – 86:
 - a. Press **2nd, MEM** to open the delete menu.
 - b. Press **F2** to open the delete program.
 - c. Press **F4** to delete lists. A menu of lists will appear. Use the up and down arrow keys to place the cursor next to data lists collected in this experiment. Press **ENTER** to delete.

DATA ANALYSIS

Part 2 Reaction of Crystal Violet with Sodium Hydroxide

1. Graph the data for each trial on a piece of graph paper. Place time on the X axis and molarity on the Y axis. Graph all trials for one concentration on the same axes.
 - a. Draw the best straight line that represents the data for each trial.
 - b. Calculate the slope of the line (rate of reaction) for each trial.

2. Calculate the average rate of reaction for each concentration of sodium hydroxide.
3. Enter the time and absorbance data into an Excel spreadsheet. Place the data from all trials of a given concentration of sodium hydroxide on the same spreadsheet.
 - a. Open the Excel program. A new spreadsheet will appear.
 - b. Activate cell A1. Label the column. Enter the time data for the first trial into the A column.
 - c. Activate cell B1. Label the column. Enter absorbance data for the first trial into the B column.
 - d. Activate cell C1. Label the column. Enter the time data for the second trial into the C column.
 - e. Activate cell D1. Label the column. Enter absorbance data for the second trial into the D column.
 - f. Activate cell E1. Label the column. Enter the time data for the third trial into the E column.
 - g. Activate cell F1. Label the column. Enter absorbance data for the third trial into the F column.
 - h. Save the data on the student disk.
 - i. Open a new spreadsheet. Enter the data for the second concentration of sodium hydroxide on the new spreadsheet.
4. Use Chart Wizard to plot graphs of the data. Absorbance vs. time will be plotted:
 - a. Highlight the time and the absorbance data for the first trial. Don't include the column labels.
 - b. Plot an XY(scatter) graph. Label the graph and axes. Add major Y gridlines.
 - c. Place the graph on the same sheet as the data.
 - d. Add a linear regression trendline. Display the equation and R-squared value on the graph.
 - e. Convert the plot area from gray to white.
 - f. Repeat steps a – e for each trial.

5. Print a copy of the spreadsheet and graphs.
6. Use the Excel graphs to determine the rate of reaction for each trial.
7. Use the Excel graph to calculate the average rate of reaction for each concentration of crystal violet.
8. Use the average rates of reaction to determine the order of reaction with respect to sodium hydroxide.

QUESTIONS

1. What is the rate law for the reaction of crystal violet with sodium hydroxide?
2. Compare the average rate of reaction from the hand drawn graphs with the values from the Excel graphs.

Kinetics of the Crystal Violet Reaction using Integrated Rate Laws

Experiment

3

PURPOSE:

The purpose of this experiment is to use the integrated rate method to determine the kinetics of a reaction. Reaction order, specific rate constant, and activation energy will be determined for the reaction of crystal violet with sodium hydroxide, using experimental data acquired at different reaction temperatures.

DISCUSSION:

Kinetics is the area of chemistry that concerns rates of reactions. The rate at which a chemical reaction takes place depends on the following factors: (1) the inherent properties of the reacting materials, (2) the temperature, (3) the concentration of the reactants, and (4) the presence of catalyst(s). In this experiment, the effect temperature will be studied in relation to reaction rate.

Reaction Order

To introduce the concept of the rate or speed of a reaction, let's consider the following hypothetical reaction where D and G are reactants, P is a product, and d, b and p are numerical coefficient



Chemical reactions, in general, are reversible. The forward and reverse reactions can occur simultaneously. This potentially complicates the kinetics of a chemical reaction. To avoid this complication, experimental conditions are chosen so that the reverse reaction makes only a negligible contribution to the rate. Under these conditions, the reaction rate will depend only on the concentrations of the reactants. For our reaction, we can write

$$\text{Rate} = k [D]^n [G]^m \quad (2)$$

Such an expression, which shows how the rate depends on the concentration of reactants, is called a **rate law**. The proportionality constant, **k**, is called the **rate constant**. The exponents, **n** and **m** are called the **order** of the reactants with respect to D and G. They must both be **determined experimentally**. The orders of simple reactions are often positive integers including zero. Note that the concentration of the product does not appear in the rate law because the reverse reaction is considered to be negligible. Again, the orders **n** and **m** must be determined by experiment, they cannot be written from coefficients of the balanced equation.

Simple chemical reactions are classified according to their orders. The overall order of a reaction can be determined by adding the exponents. A reaction is considered to be **zero order** if $n = m = 0$. A reaction is considered to be **first order** if $n = 1$ and $m = 0$, or vice versa. A reaction is considered to be **second order** if $n = 2$ and $m = 0$ (or vice versa) or if $n = 1$ and $m = 1$ (*simple* second order for the former and *mixed* second order for the latter case.)

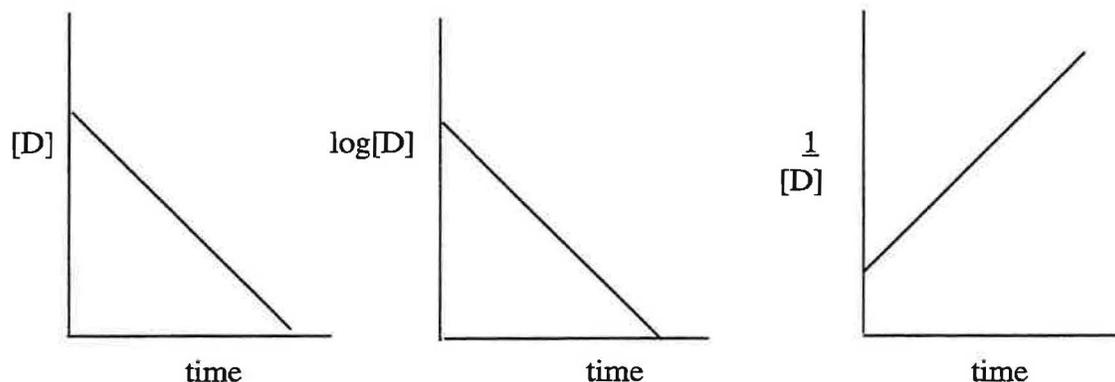
The rate law represented by equation (2) expresses rate as a function of concentration and is called the **differential rate law**. A second kind of rate law, which is easier to manipulate, is called the **integrated rate law**. The integrated rate law expresses how the concentration of a reactant or product depends on time. The differential and integrated rate laws for a chemical reaction are related in a well-defined way. Ignoring the details of the mathematical conversions between the two laws, the integrated rate laws can be written as follows for reactant D in equation (1). The concentration of reactant G is in large excess, so that it does not affect the kinetics. $[D]_0$ represents the concentration of reactant D at the beginning of the reaction (time zero). $[D]_t$ represents the concentration, in units of molarity, of reactant D at any given time (t).

$$[D]_t = -kt + [D]_0 \quad (3) \quad (\text{Zero order})$$

$$\ln [D]_t = -kt + \ln [D]_0 \quad (4) \quad (\text{First order})$$

$$\frac{1}{[D]_t} = kt + \frac{1}{[D]_0} \quad (5) \quad (\text{Second order})$$

Equations (3), (4) and (5) are all in the form of a straight line, with the slopes equal to the rate constant (k) or the negative rate constant (-k). These equations provide a useful tool to determine the order of a reaction with respect to one reactant. Let's continue to explore the reaction represented by equation (1). Assume that reactant D is a substance whose concentration can easily be monitored. The reaction is carried out in a manner that allows the concentration of D to be determined at specific time intervals. Three graphs of the data, corresponding to equations (3), (4), and (5), are then plotted:



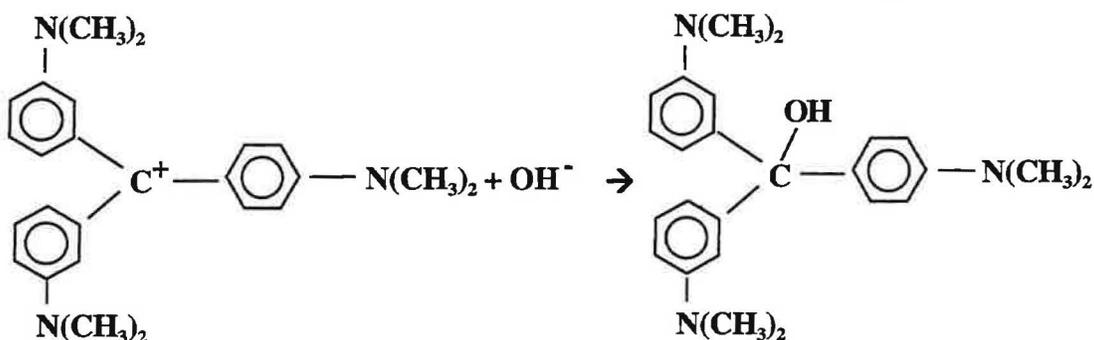
The graphs are then evaluated using linear regression (see page XVII). The plot of data that is closest to a straight line using the values of the linear regression coefficients (R) represents the order of reaction with respect reactant D. Examining the graphs reveals that the plot in the third

graph, time vs. $1/[D]$, has the correlation coefficient closest to 1. Therefore, the value of n is 2, since the reaction is second order with respect to reactant D. The value of the rate constant is determined from the slope of the line. The differential rate law for the reaction (equation 2) can now be rewritten:

$$\text{Rate} = \text{slope}[D]^2 [G]^m \quad (6)$$

To determine the value of m , the experimental procedure would be repeated, monitoring the concentration of reactant G and manipulating the concentration of D so that it had no effect on the kinetics of the reaction.

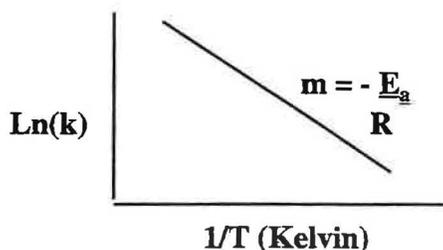
The reaction under investigation in this experiment is the reaction of crystal violet, a purple biological stain with sodium hydroxide (colorless) to produce a colorless product.



Crystal violet absorbs light at 565 nm. Its concentration can be easily monitored using a colorimeter. Crystal violet is very advantageous for this experiment, because the reaction with sodium hydroxide occurs very quickly. The reactants are mixed and then quickly placed in a cuvette, which is immediately placed in a colorimeter. The absorbance of the solution is proportional to the concentration of crystal violet present. The absorbance is monitored over a period of time. Three graphs of the data are plotted. The order of reaction with respect to crystal violet and the value of the rate constant are determined.

Energy of Activation

The activation energy (E_a) is defined as the minimum amount of energy necessary for a chemical reaction to proceed.



The rate constant that is determined for reactant D in this experiment is specific for the conditions under which the reaction is carried out. Changing reaction conditions such as temperature will

change the value of the rate constant. Knowing the specific rate constants at different reaction temperatures permits the activation energy to be calculated for the reaction. The **Arrhenius equation** expresses the relation between the rate constant and temperature of a reaction in the form of a linear equation:

$$\ln k = \frac{-E_a}{R} \frac{1}{T} + \ln A \quad (7)$$

R is the universal gas constant with a value of 8.3145 J/K·mole, T is absolute temperature (Kelvin) and A a the collision frequency factor. A plot of $\ln k$ versus $1/T$ results in a straight line with a **slope** = $-E_a/R$. The value of E_a can then be calculated from the value of R and the slope of the line.

This experiment uses the equation relating the temperature and the specific reaction rate constant to determine the activation energy for the crystal violet reaction. The reaction will be performed at three different temperatures and the values of the corresponding rate constants determined. A graph of the natural log of the rate constant (k) versus the inverse of the absolute temperature ($1/T$) will be plotted and the value of the activation energy will be calculated.

Review the Beer's law and spectrophotometry in Experiment 2. Handle the cuvette by the frosted sides only. Wipe the cuvette with a Kimwipe. Always align the black edge to the front of the colorimeter.

MATERIALS & EQUIPMENT:

Solids

ice

Solutions

4.0 x 10⁻⁵ M crystal violet

1.0 M NaOH

Stockroom equipment

CBL system with colorimeter

cuvettes with one edge marked (2)

Kimwipes

Locker equipment

10 mL graduated cylinders (2)

beakers (100 mL, 800 mL)

stirring rod

Laboratory waste container

none needed

PROCEDURE:

Caution: Crystal violet is a biological stain. Do not allow it to come into contact with your skin or clothing.

For disposal of laboratory waste, solutions may be poured into sink with a lot of water.

1. Check out a CBL system with a colorimeter and 2 cuvettes from the stockroom.
2. Obtain temperature assignments from the instructor.
3. Use an 800 mL beaker to prepare a water bath at the lowest of the three assigned temperatures. Add enough water so that a 10 mL graduated cylinder can be submerged to the top of the graduation marks. Use ice as needed to maintain the temperature.
4. Obtain reactant solutions:
 - a. Measure 5 mL of 1.0 M NaOH into a 10 mL graduated cylinder
 - b. Measure 5 mL of 2.0×10^{-5} M crystal violet solution into another 10 mL graduated cylinder.
5. Place both 10 mL graduated cylinders into the prepared water bath. Incubate both reactant solutions in the water bath for a minimum of 10 minutes.
6. Prepare a blank cuvette for calibrating the colorimeter. Fill a clean dry cuvette with distilled water. Carefully dry the outside with a Kimwipe.
7. Activate the Chembio program by pressing **PRGM**, and selecting **NAMES (F1)**.
 - a. Select **CHEMB** by pressing **F1** and **ENTER**. The Vernier **CHEMBIO** welcome screen will appear. Press **ENTER** again to get to the main menu.
8. Setup the CBL system by selecting **SET UP (F1)**
 - a. Make the following selections:
Number of probes – **1** Probe – colorimeter (**F4**) Channel number – **1**
 - b. Calibrate the colorimeter at 0% and 100 % transmittance:

Insert the blank into the cuvette slot in the colorimeter with the black edge facing the front. Turn the knob to 0% transmittance. When the voltage reading on the CBL has stabilized, press "Trigger". Enter **0** (for 0% transmittance) at the prompt.

Set the wavelength to 565 nm. When the CBL unit has stabilized, press "Trigger". Enter **100** (for 100% transmittance) at the prompt.

9. Prepare the CBL system to collect data by pressing **F2**.

a. Select the following:

Data Collection – time (**F2**) Time between samples - **3** sec.
Number of samples – **100**

Press **F1** to use the program with the above selections. Press **F2** to modify the choices if necessary.

b. Format the TI-86 screen. Enter the following:

Y min – **0** Y max – **1** Yscl – **0.1**

10. Remove the blank cuvette from the colorimeter. Keep the blank. It will be used for subsequent calibrations.

11. To begin collecting data, this process must be done quickly, but in a controlled manner.

- a. Pour the 5 mL portions of crystal violet and sodium hydroxide into the clean, dry 100 mL beaker and swirl to mix.
- b. Fill the clean, dry cuvette $\frac{3}{4}$ full with the reaction mixture. Wipe the outside of the cuvette with a Kimwipe, if needed.
- c. Place the cuvette in the cuvette slot of the colorimeter and close the cover.
- d. Press **ENTER** to begin collecting data.
- e. Measure and record the temperature of the reaction mixture in the beaker.
- f. When data collection is complete, **DONE** will appear on the CBL screen. Measure and record the temperature of the reaction mixture in the beaker.
- g. Record the names of the data files where time, absorbance and transmittance data are stored from the screen of the TI – 86.
- h. Press **ENTER** to display a graph on the calculator screen.
- i. If prompted to repeat the trial choose **NO (F1)**. Choosing **YES (F2)** will overwrite the trial and erase all data.
- j. Press **ON** to exit the program.

12. Rename the time and absorbance data lists on the TI – 86. Record the new names in the lab notebook. Do not use the same name for any other trials. Each time a data list is saved, it will erase any data that was previously saved using that name.
13. Dispose of the reaction solution by pouring it into the sink with a lot of water.
14. Transfer the data lists from the TI – 86 to an Excel spreadsheet. Place the time and absorbance data from the first trial into columns A and B. Save on a floppy disk.
15. Repeat steps 3-14 for a second trial at the same temperature. Transfer the data for subsequent trials to new sheets in the same workbook.
16. Perform two trials for each of the other assigned temperatures by repeating steps 3-14. A total of six trials should be performed: 2 at each temperature.
17. Delete the data lists from the TI – 86:
 - a. Press 2nd, MEM to open the delete menu.
 - b. Press F2 to open the delete program.
 - c. Press F4 to delete lists. A menu of lists will appear. Use the up and down arrow keys to place the cursor next to data lists collected in this experiment. Press ENTER to delete.

DATA ANALYSIS:

A Determination of Order of Reaction

1. Calculate the average temperature over the course of the reaction.
2. Open the Excel Spreadsheet file containing time and absorbance data. For each trial:
 - a. Insert a row at the top of the sheet. Use this row to appropriately title the spreadsheet.
 - b. Insert another row. Use this row to appropriately label the columns.
 - c. Use the appropriate column to determine the half-life of crystal violet.
 - d. Determine three half-lives of crystal violet.
 - e. Calculate the natural log of absorbance values in column C.
 - f. Calculate the inverse of absorbance values in column D.

3. Use Chart Wizard to plot **3 graphs for each trial**. Absorbance vs. time, natural log of absorbance vs. time and inverse of absorbance versus time. Only the data for three half-lives of crystal violet will be plotted:
 - a. Highlight the time and the absorbance data column down to the point for three half-lives of crystal violet.
 - b. Plot an XY(scatter) graph. Label the graph and axes. Add major Y gridlines.
 - c. Place the graph as an object the Sheet.
 - d. Add a linear regression trendline. Display the equation and R-squared value on the graph.
 - e. Convert the plot area from gray to white.
 - f. Resize the graph so that it appropriately displays the data.
 - g. Repeat steps a – f for time and natural log of absorbance.
 - h. Repeat steps a – f for time and inverse of absorbance. A total of three graphs will appear on the sheet for each trial.
4. Print one copy of each file, including the spreadsheet and the graphs. Do not print each graph separately!
5. Review the graphs for all six trials. Determine the order of the reaction with respect to crystal violet.
6. Determine the value of the reaction constant for each trial. Write these values on the appropriate graphs.
7. Calculate the average temperature for the two trials at each assigned temperature.
8. Calculate the average value for the reaction constant at each assigned temperature.

B. Determination of energy of activation

1. Insert a new worksheet into the spreadsheet file.
2. Label the columns as follows:

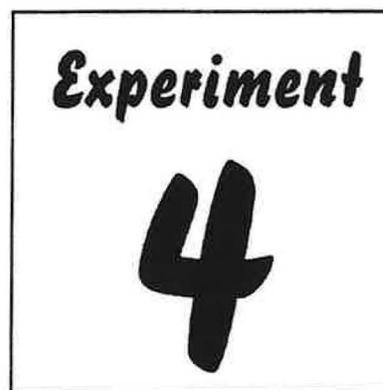
Trial Numbers	Avg. T Celsius	Avg. T Kelvin	$\frac{1}{\text{Kelvin}}$	Avg. reaction constant (k)	Natural log of k
--------------------------	---------------------------	--------------------------	---	---------------------------------------	-----------------------------

3. Enter the trial numbers into column A. More than one trial number will appear since the data was averaged.
4. Enter the average temperature (from Part A, Step 8) into column B.
5. Enter the corresponding value of the reaction constant into column E.
6. Use column C to convert the average temperatures from degrees Celsius to degrees Kelvin.
7. Use column D to calculate the inverse of the average temperature (Kelvin).
8. Use column F to calculate the natural log of the reaction constants.
9. Use Chart Wizard to plot a graph of the natural log of the reaction constant versus the inverse of the absolute temperature.
 - a. Plot an XY(scatter) graph. Label the graph and axes. Add major Y gridlines.
 - b. Place the graph as an object the Sheet.
 - c. Add a linear regression trendline. Display the equation and R-squared value on the graph.
 - d. Convert the plot area from gray to white.
 - e. Resize and position the graph so that it appropriately displays the data.
10. Print a copy of the spreadsheet containing the graph.
11. Use the slope of the line and the value of the gas constant to calculate the activation energy of the reaction.

QUESTIONS:

1. What effect does a ten-degree temperature increase have on the rate of reaction? Use experimental data to explain your answer.
2. Based on your results of this experiment and Experiment 2, what is the rate law for the reaction of crystal violet with sodium hydroxide?

Chemical Kinetics



PURPOSE:

Spectroscopic techniques will be used to study the kinetics of a reaction. Students will design an experiment that will allow them to discover the order of a chemical reaction and the rate constant.

DISCUSSION:

Chemical kinetics is the area of chemistry that concerns reaction rates. To introduce the concept of the rate or speed of a reaction, let's consider the following hypothetical reaction where A and B are reactants and P is a product:



For chemical reactions, the quantity that changes is usually the concentration of a reactant or product. Chemical kinetics deals with the speed at which these changes occur. So the **reaction rate** is defined as the change in concentration of a reactant or product per unit of time.

$$\text{Rate} = -\frac{\Delta[A]}{\Delta t} = -\frac{\Delta[B]}{\Delta t} = \frac{-\Delta[P]}{\Delta t} \quad (1)$$

The Greek symbol delta (Δ) is used to indicate a change in a given quantity. The negative sign in front of the rate of disappearance of a reactant is to always define the rate of appearance of product as a positive quantity.

Rate Laws

Chemical reactions, in general, are reversible. The forward and reverse reactions can occur simultaneously. This potentially complicates the kinetics of a chemical reaction. To avoid this complication, experimental conditions are chosen so that the reverse reaction makes only a negligible contribution to the rate. Under these conditions, the reaction rate will depend only on the concentrations of the reactants. For our reaction, we can write

$$\text{Rate} = k [A]^n [B]^m \quad (2)$$

Such an expression, which shows how the rate depends on the concentration of reactants, is

called a **rate law**. The proportionality constant, k , is called the **rate constant**. The exponents, n and m are called the **order** of the reactants with respect to A and B. They must both be determined by **experiment**. The orders of simple reactions are often positive integers (including zero). Note that the concentration of the product does not appear in the rate law because the reverse reaction is considered to be negligible. Again, the orders n and m must be determined by experiment, they cannot be written from coefficients of the balanced equation.

Simple chemical reactions are classified according to their orders. The order of reaction can be determined by adding the exponents. A reaction is considered to be **zero order** if $n = m = 0$. A reaction is considered to be **first-order** if $n = 1$ and $m = 0$, or vice versa. A reaction is considered to be **second-order** if $n = 2$ and $m = 0$ (or vice versa) or if $n = 1$ and $m = 1$ (*simple second order* for the former and *mixed second order* for the latter case.)

The rate law represented by equation (2) expresses rate as a function of concentration and is called the **differential rate law**. A second kind of rate law, which is easier to manipulate, is called the **integrated rate law**. The integrated rate law expresses how the concentration depends on time. The differential and integrated rate laws for a chemical reaction are related in a well-defined way. Ignoring the details of the mathematical conversions between the two laws, we can write the integrated rate laws involving only one reactant, A, as follows:

$$[A]_t = [A]_0 - kt \quad (3) \quad \text{(Zero-order)}$$

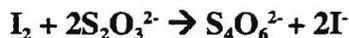
$$\ln [A]_t = \ln [A]_0 - kt \quad (4) \quad \text{(First-order)}$$

$$\frac{1}{[A]_t} = \frac{1}{[A]_0} + kt \quad (5) \quad \text{(Simple second-Order)}$$

$[A]_t$ represents the concentration, in units of molarity, of reactant A at any given time (t). $[A]_0$ represents the concentration of reactant A at the beginning of the reaction (time zero).

Pseudo-First-Order Rate Law

Let's consider the following reaction:



From *experimental* evidence we know that the rate law is

$$\text{Rate} = k[I_2][S_2O_3^{2-}] \quad (6)$$

Suppose we carry out this reaction with initial concentrations of $[I_2]_0 = 0.0010$ M and $[S_2O_3^{2-}]_0 = 0.10$ M. As the reaction proceeds, $[I_2]$ decreases significantly, but because the $S_2O_3^{2-}$ ion concentration is so large initially, relatively little of this reactant is consumed. Thus, $[S_2O_3^{2-}]$ remains approximately constant throughout the reaction. Under such conditions, the rate law (6) can be written as

$$\text{Rate} \approx k[I_2][S_2O_3^{2-}]_0 \approx k_{\text{obs}}[I_2] \quad (7)$$

The observed rate constant, k_{obs} , is equal to the rate constant multiplied by the initial concentration of $\text{S}_2\text{O}_3^{2-}$:

$$k_{\text{obs}} = k[\text{S}_2\text{O}_3^{2-}]_0$$

The new rate law (7) is first order. However, since this law was obtained by simplifying a more complicated one, it is called a **pseudo-first-order** rate law. The proportionality constant, k_{obs} , is called a pseudo-order rate constant and can be determined from experimental data in exactly the same way as a first-order rate constant.

Experimental Determination of Rate Laws

Two common methods are used to experimentally determine the form (zero, 1st, 2nd) of the rate law for a chemical reaction.

(A) Initial – Rate Method

The idea in this method is to determine the rate of the reaction just after the reaction begins and before the initial concentrations of reactants have decreased significantly. Several experiments are carried out using different initial concentrations and the initial rate is determined for each run. The results are then compared to see how the initial rate depends on the initial concentrations (See textbook for examples).

(B) Integrated – Rate Method

In this method the change in concentration of reactant [A] or a property that linearly varies with [A], such as absorbance for a colored solution, is followed for an appropriate length of time. The data, [A] vs. time or absorbance vs. time are analyzed graphically using the integrated rate laws stated earlier (equations 3, 4 and 5). This analysis allows the form of the rate law and the rate constant, k , to be determined.

The reaction between sodium hypochlorite (bleach) and the food colorant FD&C Blue # 1 (see structure on last page of this experiment) will be studied in this experiment. The chemical equation of this reaction is:



And the general rate equation is:

$$\text{Rate} = k [\text{Blue \# 1}]^n [\text{NaOCl}]^m$$

The goal of this experiment is to determine the values of exponents n and m and the value of k at room temperature. Familiarity with Beer's Law ($A = \epsilon bC$) and basic working skills with a CBL system are assumed. Remember that the color of the solution is complementary to the color absorbed by the solution. For example, a solution that absorbs only yellow light appears violet. Likewise, a solution that absorbs only blue light appears orange.

MATERIALS & EQUIPMENT:

Solutions

stock solution of FD&C Blue # 1 Colorant
bleach (containing sodium hypochlorite, NaOCl)
0.67 M NaCl solution (to be used to dilute bleach, if needed)

Stockroom Equipment

2.0 ml graduated pipet CBL system with a colorimeter and cuvettes

Locker Equipment

all items

Laboratory Waste Containers

none needed

PROCEDURE:

Organization and Guiding Questions

As you can see, there is no written procedure for this experiment. Instead, working in groups of three, you will use your practical and thinking skills to design, write a procedure and execute the different parts of the experiment. Use the list of available equipment, materials and the following “guiding questions” to focus on the important aspects of the experimental design.

1. To follow the rate of disappearance of the blue colorant with time you will use a CBL-system with a colorimeter probe. The probe wavelength can be set at 635 nm, 565 nm or 437 nm for an experiment. Which wavelength will you choose for this experiment? Why? (Hint: consider Beer’s Law)
2. The blue colorant concentration (stock solution) is not known. How can you determine this concentration? What data do you need? If you need additional data, ask your instructor.
3. How can you determine the molarity of NaOCl solution? What assumption(s) do you need to make in this calculation? (Hint: see bleach label)
4. How can you determine what volumes of blue colorant and bleach to mix in order to get the reaction to be “nearly complete” in about 10-12 minutes? (Hint: try mixing the reactants in the ratio of colorant: bleach in ratios of 10:1, 20:1 and 30:1. Visually monitor and time the color change. The color does not have to disappear completely.)
5. Once you have determined the appropriate volumes to mix in question 4, what are the initial concentrations of the colorant and the NaOCl when the solutions are mixed? Does this reaction follow pseudo-order kinetics? (Hint: the initial concentrations must be calculated from their initial values.)

6. Do you need to know the change in concentration with time of the blue colorant to determine the order of the reaction or can you use another variable that is directly proportional to this concentration? If the latter, what is the variable in this experiment?
7. What calculation or graphical analysis will you need to do in order to determine the order of the reaction with respect to the blue colorant?
8. How will you determine the value of the observed k , k_{obs} ? Does it depend on the initial concentration of the blue colorant? the NaOCl?
9. After you have determined the value of exponent n , how can you determine the value of exponent m ? Do you need to do another experiment? If yes, what do you need to change? If no, how do you determine m ?
10. How will you determine the value of k , the rate constant?

λ of maximum absorption	Color absorbed by solution	Color of solution
420-440	Violet-blue	Green-yellow
470 - 480	Blue	orange
520-530	Green	Purple
560-580	Yellow-green	Violet
610-630	Orange	Blue
650-680	Red-orange	Blue-green
700-720	Red	Green

Writing the Procedure

Safety Precaution Commercial sodium hypochlorite (bleach) is a strong oxidizing agent that should be handled with care in the laboratory and at home. Do not let it touch your skin or clothing. If some is spilled, immediately wash the area with plenty of water.

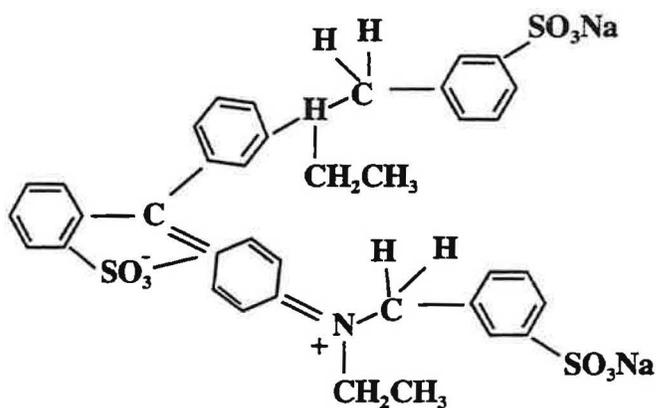
For laboratory waste disposal, all solutions can be poured down the drains with a large amount of water.

After you have successfully answered the guiding questions, write a concise procedure to include:

1. steps on how you will do the experiment. Include which items of equipment to use, volumes to use, rate of data collection, etc. Remember it is very critical to deliver volumes *as accurately as possible* in a kinetic experiment. Use the most accurate equipment available to you for this purpose. Plan to perform duplicate runs to assure data reproducibility.

2. how data analysis will be done. Include what graphical analysis method will be used and how the rate constant, k , and orders, n and m , will be determined.

Show a draft of your experimental design to your instructor for approval and/or modification. When finally approved, go ahead and perform the experiment with your group. Your instructor will inform you on the format of the lab report you need to produce for the experiment and grading.



Buffers

Experiment

9

PURPOSE:

The purpose of this experiment is to prepare and study buffer solutions. The effect of small additions of acid and base both to water a buffer solution will be explored. A buffer of a designated pH and molarity will then be prepared. The pH of the prepared buffer will be compared to the theoretical pH value.

DISCUSSION:

In many areas of research, scientists need an aqueous solution that will resist a pH change when relatively small amounts of acid or base are added to it. For example, animal cells growing in culture solution may die if the pH changes by more than 0.2 pH units. Growing cells produce carbon dioxide as a waste product. Carbon dioxide reacts with water to form carbonic acid, which can lower the pH of the culture solution. Therefore, culture solutions are prepared with a **buffer**, an aqueous solution that will absorb H^+ and OH^- with very little change in pH. Buffers are excellent examples of chemical equilibria in action. A buffer is a mixture of a weak acid and its conjugate base, or conversely, a weak base and its conjugate acid.

Let's investigate a buffer solution prepared from a weak acid (HA) and the sodium salt of its conjugate base (NaA). An equilibrium equation can be written for the weak acid:



Applying LeChatelier's principle, addition of acid (H^+) to a solution of HA will cause the equilibrium to shift to the left in order to remove the extra H^+ from solution. Addition of OH^- to the buffer solution will cause a neutralization reaction to occur with H^+ , thereby reducing the concentration of H^+ in solution. The system will compensate by shifting to the right, releasing H^+ into solution. In both situations, the pH will remain very close to the original value. In Part 1 of this experiment, the effect of addition of acid and base on the pH of an acetic acid/sodium acetate buffer solution will be studied.

For a solution to be an effective buffer, it must contain relatively large, similar concentrations of both HA and A^- . This allows the buffer solution to absorb the addition of either acid or base. A solution containing only HA (a weak acid) would have a very low concentration of A^- and

therefore would not be able to absorb very much extra H^+ before the concentration of A^- was exhausted. A solution containing mostly A^- (the conjugate base of the weak acid) would have a very low concentration of HA and therefore would not be able to absorb very much extra OH^- before the concentration of HA was completely eliminated.

The Henderson-Hasselbalch equation is a useful tool for preparing buffer solutions:

$$pH = pK_a + \frac{\log [\text{conjugate base}]}{[\text{acid}]} \quad (1)$$

This equation can be used to select a weak acid for the preparation of a buffer at a specific pH. Suppose the concentration of the conjugate base ($[A^-]$) is equal to the concentration of the acid ($[HA]$). The ratio in equation (1) will then be equal to 1. The log of 1 is 0. The pH of the buffer solution will then be equal to the pK_a of the weak acid:

$$pH = pK_a + \frac{\log [A^-]}{[HA]} = pK_a + \log 1$$

$$pH = pK_a$$

A weak acid with a pK_a near the desired pH can then be selected. For example, suppose that a chemical reaction requires a constant pH of 3.75 to proceed. A pK_a table reveals that formic acid has a pK_a of 3.75, the pH value required for the reaction. Formic acid would be a good choice as the acid and sodium formate could be chosen as the salt. The buffer solution would then be prepared by adding equal molar amounts of formic acid and sodium formate.

The Henderson – Hasselbalch equation also allows calculation of the ratio of conjugate base to acid, if the pH of the buffer solution required is different from the pK_a of the weak acid. Suppose that a reaction system requires a constant pH of 3.85. Formic acid/ sodium formate could still be used as a buffer system, but the ratio of conjugate base to acid would not be 1/1:

$$pH = pK_a + \frac{\log [\text{conjugate base}]}{[\text{acid}]}$$

$$3.85 = 3.75 + \frac{\log [\text{formate ion}]}{[\text{formic acid}]}$$

$$0.10 = \frac{\log [\text{formate ion}]}{[\text{formic acid}]}$$

Taking the antilog of both sides will result in the ratio of formate ions to formic acid molecules needed to prepare a buffer of pH 3.85:

$$1.26 = \frac{[\text{formate ion}]}{[\text{formic acid}]}$$

For every mole of formic acid ions in the buffer solution, 1.26 moles of sodium formate should be added to prepare a buffer of pH 3.85.

The concentration of the buffer must also be considered. A 1.00 M formic acid/sodium formate buffer at pH 3.85 should contain a **total** of 1.00 moles of both solutes in one liter of solution. The actual amounts of each solute that should be added can be calculated, assuming 1.00 liter of buffer will be prepared. Let X = moles of formic acid and $1.26 X$ = moles of sodium formate:

$$1.00 \text{ mole total solute} = X \text{ moles formic acid} + 1.26 X \text{ moles sodium formate}$$

$$1.00 \text{ moles of total solute} = 2.26 X \text{ moles of solute}$$

$$\frac{1.00}{2.26} = X$$

$$X = 0.443 \text{ moles of formic acid}$$
$$1.26 X = 0.558 \text{ moles of sodium formate}$$

The 1.00 molar buffer will actually be 0.443 M formic acid and 0.558 M sodium formate. The actual amounts the weak acid and salt needed can be adjusted when different volumes of buffer are prepared.

In Part 2 of this experiment, a buffer of a given pH will be prepared as assigned by the instructor. The quantity of acid and base required to change the pH a designated amount will be then calculated. The calculated amount of acid and base will then be added to the buffer and the pH of the resulting solution measured. The experimental results will be compared to theoretical values.

MATERIALS and EQUIPMENT:

Solids

sodium acetate

salts for buffers

Liquids

distilled water

glacial acetic acid

Solutions

Buffers (pH 4.00, 10.00)

0.100 M acetic acid $\text{HC}_2\text{H}_3\text{O}_2$

0.010 M NaOH

0.100 M sodium acetate ($\text{NaC}_2\text{H}_3\text{O}_2$)

0.010 M HCl

Stockroom equipment

pH electrode

small vial

pipet pump
250 mL volumetric flask

CBL system or pH meter

Locker equipment

150 mm test tubes/rack	250 mL beaker
10.00 mL graduated cylinder	150 mL beaker
10.00 mL pipet	100 mL beaker

Laboratory waste

Laboratory waste containers labeled with tracking # 10025B

PROCEDURE:

Part 1: The Effects of HCl and NaOH on a Buffer Solution

For laboratory waste use container labeled with tracking # 10025B.

1. Check out a CBL system and pH electrode with amplifier.
2. Prepare a data table for the pH values of the buffer solution and water, before and after addition of acid and the addition of base.
3. Boil about 100 mL of distilled water. Cover and cool to room temperature.
4. Pipet 20.00 mL of boiled distilled water into a 150 mL beaker.
5. Pipet 10.0 mL of 0.100 M. $\text{NaC}_2\text{H}_3\text{O}_2$ and 10.0 mL of 0.100 M. $\text{HC}_2\text{H}_3\text{O}_2$ into a 100 mL beaker.
6. Set up the calculator and CBL unit for pH measurement.

Number of probes – 1 Probe – pH (F2) Channel number – 1

7. Calibrate the electrode (allow the system to warm up for 30 seconds, first):
 - a. Rinse the electrode with a stream of distilled water from the wash bottle into a waste beaker. Gently blot dry with a paper towel.
 - b. Place the electrode in pH 4.00 buffer. Stir gently until the reading on the CBL unit is stable and then press [TRIGGER].
 - c. Enter the pH of the buffer into the TI-86.
 - d. Rinse the electrode with a stream of distilled water from the wash bottle into a waste

- beaker. Gently blot dry with a paper towel.
- e. Place in electrode in pH 10.00 buffer. Stir gently until the reading on the CBL unit is stable. Press [TRIGGER].
 - f. Enter the pH of the buffer into the TI-86.
8. Prepare the calculator to collect data.
- a. Select COLLECT DATA (F2) from the main menu.
 - b. Select MONITOR INPUT (F1) from the DATA COLLECTION menu. Press ENTER after the system has warmed up for 30 seconds. The pH reading is displayed on the screen of the CBL. No readings are stored when using the MONITOR INPUT mode.
9. Determine and record the pH of the contents of both beakers.
10. Pipet 10.00 mL of 0.0100 M. HCl into both beakers and stir gently with the pH electrode.
11. Measure and record the pH of both beakers. Be sure to rinse the electrode well with distilled water between measurements.
12. Discard the solutions by pouring into waste container labeled with tracking #10025B.
13. Repeat steps 3-5.
14. Pipet 10.00 mL of 0.0100 M. NaOH into each beaker. Mix thoroughly.
15. Measure and record the pH of both beakers.
16. Discard all solutions by pouring into waste container labeled with tracking #10025B .

Part 2: Preparation of a Buffer

For laboratory waste use container labeled with tracking # 10025B.

1. Check out a CBL system and electrode with amplifier.
2. Obtain a pH assignment and a 250 mL volumetric flask from the instructor.
3. Calculate the amount of acid and salt required to prepare 250 mL of the assigned buffer solution. Have the instructor approve your calculations.
4. Prepare the buffer solution.

5. Set up the CBL system to record pH as in Part 1.
6. Measure and record the pH of the buffer system.
7. Use 0.010 M HCl or 0.010 M NaOH as needed to adjust the pH to the assigned value.
8. Calculate the amount of 0.010 M HCl required to change the pH of 50.0 mL of the buffer solution by 0.17 pH units.
9. Measure 50.0 mL of the buffer into a beaker. Add the calculated amount of 0.010 M HCl to the buffer. Record the pH of the solution.
10. Calculate the amount of 0.010 M NaOH required to change the pH of 50.0 mL of the buffer by solution by 0.25 pH units.
11. Measure another 50.0 mL of the buffer into a beaker. Add the calculated amount of 0.010 M NaOH to the buffer. Record the pH of the solution.
12. Dispose of the solutions used in this part of the experiment by pouring into laboratory waste container labeled with tracking # 10025B.

DATA ANALYSIS:

Part 1: The Effects of HCl and NaOH on a Buffer Solution

1. Calculate the theoretical pH of the acetic acid/sodium acetate buffer system.
2. Compare to the experimental pH of the acetic acid/sodium acetate buffer system to the theoretical value by calculating the percent error.
3. Calculate the theoretical pH of distilled water.
4. Calculate the theoretical pH of the distilled water upon addition of 0.0100 M HCl.
5. Compare to experimental pH of the distilled water after adding 0.0100 M HCl to the theoretical value by calculating the percent error.
6. Calculate the theoretical pH of the buffer system after adding 0.0100 M HCl.
7. Compare to experimental pH of the buffer system after adding 0.0100 M HCl to the theoretical value by calculating the percent error.
8. Calculate the theoretical pH of the distilled water after adding 0.0100 M NaOH.

9. Compare to experimental pH of the distilled water after adding 0.0100 M NaOH to the theoretical value by calculating the percent error.
10. Calculate the theoretical pH of the buffer system after adding 0.0100 M NaOH.
11. Compare to experimental pH of the buffer system after adding 0.0100 M NaOH to the theoretical value by calculating the percent error.

Part 2: Preparation of a Buffer

1. Compare the measured pH of the buffer before adjusting to the theoretical value by calculating the percent error.
2. Compare the measured pH of the buffer system after adding 0.0100 M HCl to the theoretical value by calculating the percent error.
3. Compare the experimental pH of the buffer system after adding 0.0100 M NaOH to the theoretical value by calculating the percent error.

QUESTIONS:

1. Calculate the pH of the buffer prepared in Part 1 if:
 - (a) 7.25 mL of 0.0100 M HCl was added instead of 10.00 mL.
 - (b) 4.50 mL of 0.0100 M NaOH was added instead of 10.00 mL.
2. What substance(s) could be used prepare a buffer solution at a pH of 3.36? Explain your answer.
3. Suppose that you are going to prepare one liter of a 1.00 M buffer solution at pH 5.20.
 - a. What substances could be used to prepare the buffer?
 - b. How much of each substance would be required?

Appendix C
Examples of New Nontechnology- Based Experiments

CHEM 1A

The 10 mL Transfer

CHEM 1B

Analysis of a Sodium Chloride Solution

The 10 ml Transfer

Experiment

2

PURPOSE:

The purpose of this experiment is to familiarize the student with the chemical laboratory and to practice some basic laboratory skills. The student will use the scientific method in a group setting to solve a problem.

DISCUSSION:

Asking and answering questions about nature is the realm of scientists. Most scientists use an organized, repeatable process, known as the **scientific method**, to study and solve problems. The scientist begins by posing a reasonable question or problem about which he/she hopes to learn some useful information. The scientist researches the question to see what facts are already known. Then, he/she proposes a **hypothesis** that summarizes these facts and/or predicts the outcome of exploration. Next, the scientist designs and performs **experiments** to collect data that will test the hypothesis. Data can be either qualitative or quantitative. Non-numerical observations made using the five senses (note that taste is rarely used in a chemistry laboratory!) are considered **qualitative data**. Numerical values such as measurements are considered to be **quantitative data**. The scientist analyzes the data from repeated experiments to find reproducible patterns that will support the hypothesis. If the data supports the hypothesis, the scientist draws a **conclusion** and proposes a **theory** that explains the data and answers the initial question. If the data does not uphold the hypothesis, it can be revised, and the process repeated.

The most common item used by chemists to document their experimental work is a laboratory notebook (see p.18 in the laboratory manual). All procedural steps, data and observations are recorded in a notebook, producing a permanent, accurate record of what occurred. Numerical values are recorded with the correct units and to the proper number of significant figures. Observations should be thorough, detailed and descriptive. Entries should be made in black or blue ink, directly into the notebook as the experiment is performed, not written on scratch paper and copied into the notebook later. A well-written notebook becomes an invaluable tool when preparing a report several days after the experiment has been completed.

MATERIALS & EQUIPMENT:

All items in the student locker, the laboratory and the balance room

PROCEDURE:

The instructor will assign students to groups. Each group will develop a written, detailed protocol that uses the scientific method to determine which piece of calibrated glassware (choices will be provided by the instructor) will most accurately transfer 10 mL of distilled water into a beaker. The group protocol must include all procedural steps as well as instructions for any needed calculations. Once the instructor approves the protocol, each group will perform its experiment. All procedural steps and data will be directly entered into each student's laboratory notebook in black or blue ink. A formal laboratory report (more information to follow) will be written after the experiment is completed.

Analysis of Sodium Chloride Solution

**PURPOSE:**

The purpose of this experiment is for students to gain experience in preparing and analyzing solutions.

DISCUSSION:

Fountain-Of-Youth, Inc. (a food supplement manufacturer) has discovered a new product. This liquid extract of a tree from the Amazon rain forest supposedly reverses the aging process until a person appears to be approximately 21 years of age. Preliminary testing of the product shows that it is most effective when given as a 0.955 M solution.

This product could be the most revolutionary new drug since human insulin was cloned. More testing is needed from independent research labs to verify the preliminary results. Any research lab participating in the study must be able to accurately prepare and analyze solutions. Your research group has applied for a contract to prepare and test the new product.

Fountain-Of-Youth, Inc. has selected your research group as a finalist. The next step in the selection process is a practical exercise. Your group will be given the name and concentration of a solution to prepare. Once the solution has been prepared, your group will develop two methods to analyze the solution. One method of analysis will be based on a chemical property of the solution. The second method of analysis will be based on a colligative property. All procedures, data, calculations and conclusions must be documented and presented in such a way that the auditors of Fountain-Of-Youth can repeat and verify your process. The research group that best meets these goals will be awarded the contract.

MATERIALS & EQUIPMENT:

Solids

sodium chloride rock salt
ice

Solutions

~ 0.1 M AgNO_3 standardized (maximum of 75 mL)
dichlorofluorescein

Stockroom Equipment

250 mL volumetric flask pipet pump
25 mL buret

Locker equipment

all items

Laboratory Waste Disposal

laboratory waste disposal container labeled with tracking #1022B

PROCEDURE:

**For disposal of solutions containing AgNO_3 , use container labeled with tracking # 1022B.
For disposal of solutions containing only NaCl , pour into the sink.**

The instructor will assign students to groups. Each group will develop a clearly written, detailed protocol that uses the scientific method. The group protocol must include all procedural steps as well as instructions for any necessary calculations. Once the instructor approves the protocol, each group will perform its experiment. Carry out the following tasks:

1. Prepare 250 mL of 0.62M NaCl . Be sure your protocol has indicated a method to determine exactly how much water was added when the solution was prepared.
2. Analyze the solution, using a chemical property, to verify that the molarity is correct.
3. Analyze the solution, using a colligative property, to verify that the molarity is correct.

All procedural steps and data will be directly entered into each student's laboratory notebook in black or blue ink. A formal laboratory report (more information to follow) will be written after the experiment is completed.