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† These labs require formal reports.
Acknowledgements

The 2012/14 version makes only a few minor corrections to the manual. In the previous 2011/12 version of the CHEM217 lab manual incorporates a major positive change to the home lab kit, the SpectroVis spectrophotometer. The lead author wishes to thank Neil Sexton, Elaine Goth-Birkigt, and Rob Carmichael for their work on the implementation of this improvement.

The authors’ gratefully acknowledges all the contributions and suggestions for improvement to the 2003/04, first version of this manual by Christy Tkachuk and Robert Carmichael (Summer 2002). Special thanks to Elaine Goth-Birkigt and Roberta Franchuk for checking all the B1–B4 home-lab experiments (Fall 1995), and again to Elaine Goth-Birkigt, with the help of Neil Sexton and Richie Golonka, for the construction and testing of the simple spectrophotometers (2002). Thanks again to Neil Sexton and Rob Carmichael for their to incorporate the inclusion of the 10 mL short burette in 2009. The author also wishes to thank Blaise MacMullin for all his photography work (Summer 2002). Finally, the candle image photos reprinted on page 51 are courtesy of the NASA John H. Glenn Research Center. Thanks are very warmly extended to Dr. Daniel L. Dietrich, of NASA’s John H. Glenn Research Center, Cleveland, OH, for all the additional information he provided about the candle flames in microgravity.

The experiments described in this laboratory manual are mainly variations of similar experiments that may be found described in the laboratory manuals of other universities or in commercially produced lab texts. Each experiment has been modified and rewritten, keeping the particular needs of Athabasca University students in mind. The author acknowledges the following sources:


Frantsi, Susan A. *Chemistry Experiments: A Student Manual for Off Campus Use*. Indianapolis, IN: IUPUI. (Exp. B1 and Safety Pledge)


*Laboratory Manual, Chemistry 209*, Athabasca University. (Exp. A3)


Laboratory Manual, Chemistry 320, Athabasca University. (Exp. A2)
General Information
Introduction

Athabasca University is very pleased to offer and welcome you to the home laboratory component of Chemistry 217. Although the laboratory component of this course will involve a lot of work, we hope that you will find the experience both intellectually stimulating and enjoyable. One of the benefits of having an all-home laboratory component in a course such as ours is that it gives students the ultimate in flexibility in planning and performing your experiments.

If you were to take a course such as Chemistry 217 in a traditional college or university, you would probably be expected to attend a three-hour laboratory session every week for 10–12 weeks. During this time you would complete 10–12 experiments, and would receive somewhere in the order of 30 hours of laboratory instruction. In our course, you will spend 30-40 hours of laboratory time, spread over a time frame of your choosing, and will complete 10 at-home experiments. This includes one introductory “kitchen chemistry” experiment, and 9 home lab experiments using the home-study laboratory kit.

Your preparation and organization before beginning your home laboratory experiments is vital!

At this time we would like to bring to your attention several points that will enhance your laboratory experience and minimize any potential problems.

1. **Home-study lab work.** Before doing any experiments, you will be required to do some “kitchen chemistry” (Candle Experiment) at home, and submit a short lab report. This will allow you to sharpen your observation and report writing skills without having the pressure of the home lab experiments. In addition, this is an excellent opportunity for you to receive feedback from your laboratory tutor before diving into the bulk of your experimental work.

2. **Hours of work.** Each experiment may take a full evening to complete.

3. **Home-labs.** The home lab kit can be requested from the Athabasca University science lab any day of the week (see page 6). Kits can be returned by mail (postage prepaid) to the Athabasca University science lab.

4. **Feedback.** In many freshman laboratory courses, students submit their laboratory reports shortly after having completed an experiment and the reports are returned a few days later, before the students attempt the next experiment. In the Athabasca system this is clearly not possible. After completing your first home laboratory experiment, you will have to write up the report, submit it by mail to your tutor, and then wait for some feedback. We hope that the response can be
provided before you begin your second home laboratory experiment, but if you decide to do your experiments close together, or if it takes you a long time to write your reports, this may not be possible. 
*Remember, if you have difficulty in writing your laboratory report, contact your tutor.* Also remember to keep a duplicate copy of all your experimental results—we will suggest a method for doing this in the section of this manual titled “Writing Laboratory Reports.”

**Note:**
Return of lab reports is up to each individual tutor. Your tutor may or may not return corrected lab reports for security/course integrity reasons. However, you will be given feedback by phone or mail.

5. **Preparation.** Before you begin each home lab experiment, prepare yourself by reading the entire experiment, finding and labelling all the equipment and reagents you will require, view the video clips for that particular experiment (view the safety/intro clip first), and finally contact your tutor to find out any “hints for success” in the experiment.

**Note:** As of May 2009, a new ~10 mL ‘short’ burette is being used in Exp. A3 and Project C. The video clips for these experiments has not been updated yet and still shows the use of the old ‘syringe/glass bead/serological pipette / burette’ apparatus. And as of February 2011, the new SpectoVis Plus spectrophotometer replaces the old simple spectrophotometer in Exp. A2. The video clips for Experiment A2 has not been updated yet and still shows the use of the old home-made apparatus. All Videos are now found in Moodle: http://science.lms.athabascau.ca/mod/book/view.php?id=2504

**Organization**

The laboratory component of *Chemistry 217* comprises 30-40 hours of home-lab experiments and assignments. In the past, all this work would be completed in two day-long sessions in a supervised lab. In the all-home lab format, the experiments have been grouped into three “blocks.” Please refer to the contents page of this manual for a list of the experiments that you will be doing.

**Block A**

The experiments in Block A are microlab experiments designed to introduce you to some of the basic techniques used by chemists: the use of burettes and pipettes, spectrophotometers, gravimetric analysis and volumetric analysis. We hope that you have been exposed to at least some of these techniques in your previous chemistry courses; however we realize that such exposure may have occurred several years ago. In addition to providing you with an introduction to basic laboratory techniques, the experiments in Block A are designed to reinforce some of the theoretical concepts that you will have studied in Units #’s of *Chemistry 217*. Thus, for example, Experiment A4 is
concerned with the ideal gas law. Note that Experiment A2, A4, and Project C require formal reports.

**Block B**

The experiments in Block B are also designed to reinforce concepts learned in the course, such as enthalpy, gravimetry and colligative properties. All that is required for a lab report is to fill out the observation sheet at the end of each experiment.

**Block C**

The instructions for completing the Project in Block C are intentionally much vaguer than those given for the experiments in Blocks A and B. The experiment requires students to use some of the techniques that have encountered earlier in the course. Students are expected to have deduced their own detailed procedure for this experiment. Your tutor will ask that written procedures for the Project in Block C be submitted before you begin the final experiment. Note that this Project is to be written up as a formal report.

All students will complete a short online quiz at the end of Block C. The lab quiz can be found in Moodle and is open book. This quiz will cover laboratory safety, techniques and procedures, and the basic principles upon which some of the experiments are based. If necessary, the quiz can be completed by telephone.

As you can see, a total of 10 home-study experiments are described, and others may be added as we find it necessary to modify the course. You will be required to complete a short report for A1 and A3 experiments. Formal reports are required for Experiments A2, A4 and C. Finally, the candle experiment and each of the microlab experiments in Block B require that you fill out the accompanying report form. Thus, each student will complete a total of 10 experiments.

We suggest that you work through each block of experiments in the order they are presented.
Home Lab Kit Request

If you are unsure about any of the following instructions on how to receive your Chemistry 217 home lab kit, please contact the Science Lab Coordinator at:

1-800-788-9041, ext. 6729 (North America),
780-675-6729 (Athabasca), or
780-481-3704 (Edmonton), or
by e-mail at robertc@athabascau.ca.

Note that in 2012 there is a $40.00 Chemistry 217 lab fee included in your course tuition that helps to pay for the shipping of the kit.

If you think you might be eligible for a lab exemption, please contact the Lab Coordinator or consult the Web page:

http://science.athabascau.ca/Labs/exemptions/chemistry.php

The following procedure should be observed in obtaining a Chemistry 217 all-home lab kit for home study.

1. Complete your Safety Pledge (see next page).
2. Complete your TME1 and wait for it to be marked.
3. Then fill out the online lab kit request form at:

   http://science.athabascau.ca/Labs/resources/chemistry.php

   CHEM 217 Kit Request
Safety Pledge for the Home Laboratory

This Safety Pledge can also be completed online when making the home lab kit request.

Safety Rules

1. Wear approved eye protection at all times doing your lab activities.

2. Confine long hair when doing your lab activities.

3. Perform no unauthorized experiments.

4. Select a safe site for lab activities in your home that is well ventilated, and protected from spills, children and pets.

5. Keep all chemicals and equipment out of the reach of children and pets.

6. If possible, keep all unauthorized people out of your selected site when chemicals are in use to avoid any unforeseen accidents. If anyone is allowed to observe you or participate in experiments, follow all of the proper safety rules.

7. In case of a chemical spill, clean up thoroughly with paper towels and dispose of chemicals out of the reach of children and pets.

8. If chemical spills occur on people or clothing, rinse thoroughly with lots of running water, and seek medical attention if necessary.

9. Have no food or drink in the lab area. Be sure to thoroughly clean up lab site and all utensils used after working on labs.

I have read the above rules and will observe them at all times during my chemistry course.

_________________________________________________________________  _____________________________
Print name         Student ID No.

_________________________________________________________________
Signature         Date
Receiving and Returning your Home Lab Kit

Your CHEM217 Home Lab Kit is shipped to you via courier (ground transportation). Typically you will receive your kit 2-3 weeks after you make your lab kit request.

1. After you receive your kit, ensure that all the materials are included in the kit (a check-in list of materials will be provided). Contact the Science Lab if any replacements are required at 1-800-788-9041, ext. 6277.

2. After completing the experiments at home, return the kit to the Science Lab at Athabasca University. It would be wise to insure the kit for at least $800.00.

Your Chemistry 217 home lab kit may be returned to location 1 below via prepaid ground courier. Please indicate on the waybill you are returning ‘Educational Materials’. For students living in Edmonton or Calgary the kit can also be dropped off at locations 2-4 listed below:

1. **AU in Athabasca:** AU Science Lab, Athabasca University, 1 University Drive, Athabasca, AB T9S 3A3. (Use Prepaid Waybill)

2. **AU-Edmonton Learning Centre:** 12th floor, 10030-107 St., N. Tower, Edmonton, AB T5J 3E4. (drop off location only)

3. **AU-Calgary Learning Centre:** 1040-7th Ave. SW, Calgary, AB T2P 3G9. (drop off location only)—location will change in 2013

4. **NAIT:** only when AU labs are in progress—Check lab schedule, 11762 - 106 St., Rm G-207. (drop off location only)

**Note:** The contents of the home lab kit may be changed from time to time or may vary from one location to another. Your tutor will advise you if any additional items should be included or if any items should be deleted from the list.

Once back at Athabasca University, the lab manager or technician will check the kit over for damage.*

If you are unsure about any of the following instructions on how to receive your Chemistry 217 home lab kit, please contact, Mr. Neil Sexton, the Science Lab Kit Manager, at:

1-800-788-9041, ext. 6277 (North America), 780-675-6277 (Athabasca), or by e-mail at neils@athabascau.ca.

* You will not be charged for general disposable items. However, you will be charged for replacing any broken or lost non-disposable items.
Materials to Be Provided by the Student

When working on your Chemistry 217 laboratory experiments, each student must provide himself or herself with the following items:

- a lab coat
- an electronic calculator
- a lab notebook
- a pen, a pencil and a ruler
- a supply of metric graph paper (i.e., 1 mm \times 1 \text{mm squares}).

Notes:

1. Lab coats can usually be purchased at college or university bookstores, army surplus stores, or similar establishments. In case of difficulty, see “Uniforms—Retail” in the “yellow pages” of your telephone directory.

2. A lab notebook should be bound. The preferred size is approximately 23 cm \times 29 cm.

Some other items need to be supplied for specific experiments.
## Evaluation of Students’ Work

All students will work individually. Pairing up and the pooling of data, solutions, etc., is not permitted, unless the laboratory tutor or professor specifically asks you to do so. Note that the penalties for plagiarizing laboratory reports are identical to those incurred for other types of plagiarism. You must attain an average of 60% for laboratory work in order to pass the course. The grade for laboratory work is determined as follows:

<table>
<thead>
<tr>
<th></th>
<th>Activity</th>
<th>Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Candle Experiment</strong></td>
<td>Short Report</td>
<td>5%</td>
</tr>
<tr>
<td><strong>Block A</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Experiment A1</td>
<td>Short Report</td>
<td>5%</td>
</tr>
<tr>
<td>Experiment A2</td>
<td>Formal Report</td>
<td>15%</td>
</tr>
<tr>
<td>Experiment A3</td>
<td>Short Report</td>
<td>5%</td>
</tr>
<tr>
<td>Experiment A4</td>
<td>Formal Report</td>
<td>15%</td>
</tr>
<tr>
<td><strong>Block B</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Experiment B1</td>
<td>Report Sheet</td>
<td>5%</td>
</tr>
<tr>
<td>Experiment B2</td>
<td>Report Sheet</td>
<td>5%</td>
</tr>
<tr>
<td>Experiment B3</td>
<td>Report Sheet</td>
<td>5%</td>
</tr>
<tr>
<td>Experiment B4</td>
<td>Report Sheet</td>
<td>5%</td>
</tr>
<tr>
<td><strong>Block C</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Project C</td>
<td>Formal Report</td>
<td>20%</td>
</tr>
<tr>
<td>Lab Quiz (online)</td>
<td></td>
<td>15%</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td></td>
<td>100%</td>
</tr>
</tbody>
</table>
Writing Laboratory Reports

In Chemistry 217 you will be expected to produce two distinctly different types of laboratory report: “short reports” and “formal reports.” Some hints designed to assist you in writing each type of report are given below. Note: Good laboratory report writing begins with preparing your lab notebook before the experiment, taking thorough lab notes, and writing down your observations as they happen.

Short Report

In a short report, we do not require that you provide a detailed description of how the experiment was carried out (see procedure instructions below), or give a detailed discussion of the results obtained. In general, the following format must be used.

1. Title and Date
   Experiment title and date performed as well as your name and ID number.

2. Purpose of Experiment (note there is no introduction or theory section required)
   Clearly state the what scientific principle is being tested, determined or verified. Do not rewrite what is in the lab manual. Note: There may be more than one major objective as well as a few minor objectives. This is a good place to write out the relevant chemical reactions/equations.
   Example: To determine the purity of a given solution of iron (II) sulfate heptahydrate by titration with a standard solution of sodium dichromate. A minor objective is to learn how to properly clean, calibrate and operate a buret.

3. Procedure
   In the short report, reference the appropriate pages in your lab manual, noting only any changes or modifications to the procedure.
   Example: The experiment was carried out as described in Experiment X3 of the Chemistry 217 Laboratory Manual pp. ____, except that ammonium iron(II) sulfate hexahydrate was used instead of iron(II) sulfate heptahydrate.

4. Observations
   Example: When the sodium hydroxide/sodium sulfite mixture was added to the potassium permanganate solution, the solution initially turned green, and then a brown precipitate of manganese(IV) oxide was produced.
5. **Results**

In general, the instructions for each experiment include a suggested format for presenting your results. The numerical results should be listed in the order they were obtained and are best placed into a neat, carefully labelled table. Use more than one table if necessary; i.e., do not mix unrelated data sets.

6. **Calculations**

All calculations should be presented clearly (i.e., titled, answer underlined) and should be carried out using the appropriate number of significant figures. By clearly setting out your results and calculations you make it easier for your instructor to grade your report. Any error analysis and/or discussion can be included at this point.

7. **Answers to Questions**

Don’t forget that the questions pertaining to the experiments are sometimes provided separately. **Hint:** Always rewrite the question on the lab report page and provide your answer below. Show all your work, as part marks are given.

8. **Conclusion**

You would usually include a sentence or short paragraph that summarizes your results and puts them into some kind of context, i.e., the results have some meaning or importance. If you’ve identified an unknown, make sure you clearly state it here. If you’ve made a product, make a final comment on its quality and quantity.

Example: *In this experiment, the solubility of potassium nitrate in water was determined at several temperatures, and a solubility curve was constructed. In common with those of many ionic compounds, the solubility of potassium nitrate was found to increase dramatically with an increase in temperature.*

**Formal Report**

The main difference between a formal report and the short report described above is that in the formal report we expect much more detail, particularly in the areas of the procedure used and the discussion of results. If you wish, you may think of the “short report” as being written for a person who is familiar with the experiment in question. In contrast, a formal report may be regarded as being written for a person who, while having an adequate background in chemistry, is not at all familiar with these experiments. A formal report should consist of the following elements:

1. **Title and Date**

   Title of experiment and date performed, your name and ID number
2. **Purpose of Experiment**
   As for the short report.

3. **Introduction**
   Give a brief introduction to the problem to be solved and the approach to be used in the experiment. Do not copy directly from the laboratory manual. Usually, one or two paragraphs will be adequate. Remember to clearly state the aim of the experiment, and comment on the experiment’s usefulness or importance.

4. **Procedure**
   Your account should be sufficiently detailed that another student could repeat the experiment based on your report. Do not simply regurgitate the laboratory manual, and keep the following points in mind.
   
   a. Use the third person, the passive voice, and the past tense.
      
      Correct: *The solution was heated on a hot-plate for 30 minutes.*
      Incorrect: *I heated the solution on a hot-plate for 30 minutes.*
      Incorrect: *The solution is heated on a hot plate for 30 minutes.*
   
   b. Avoid the “recipe format.”
      
      Incorrect: *Heat the solution on a hot-plate for 30 minutes.*
   
   c. Incorporate your observations into the procedure.
      
      Example: *The solution was heated on a hot-plate for 30 minutes, during which time the colour of the solution changed from red to green.*
   
   d. Avoid unnecessary detail.
      
      Acceptable: *20 mL of hydrochloric acid was added to the solution with constant stirring.*
      
      Unacceptable: *20 mL of hydrochloric acid was poured from a graduated cylinder into a 100-mL beaker containing the solution. During this process the solution in the beaker was stirred with a 15-cm long glass rod having a diameter of 5 mm.*

5. **Results and Calculations**
   As for the short report. Sample calculations can be used if repetitive calculations are involved.

6. **Discussion**
   This section gives you an opportunity to discuss the significance of your results, to assess the validity of the method, to indicate possible sources of error, and so on.
7. **Questions**

8. **Conclusion**

   As for the short report.

   Note that in some cases neither of the above formats is entirely appropriate. In such situations you will be advised as to the most suitable form in which to submit your report.

In most laboratory courses, a student is expected to submit his or her laboratory reports in a bound notebook. With the Athabasca University system this is not practical—mailing costs would be too high, and there might be a problem with getting notebooks returned before the you want to begin your next experiment. Thus the following procedure should be adopted.

9. All your results, observations, etc. should be recorded directly in a bound laboratory notebook (preferred size 23.5 cm $\times$ 18.4 cm). This notebook is your permanent record of work carried out in the laboratory. How you choose to organize this notebook is up to you, as it will not normally be submitted to your instructor. However, in the event of some future discrepancy, you may be asked to produce the notebook for inspection.

10. Your reports should be written on loose-leaf paper (21.5 cm $\times$ 28 cm) and be submitted by mail to your instructor. Be sure to number the pages, and write your name and the number of the experiment on each page. Should your report get lost in the mail, you will still have your results recorded in your notebook and a photocopy of the submitted report. In this way the photocopy of the report can be re-submitted. Please include your address, e-mail address, and telephone number with your reports.
Sample Chemistry 217 Laboratory Reports

Short Report Format

Title: Acid Base Titrations – The Percentage of Acetic Acid in Vinegar
By: A. Student, #990001

dd/mm/yy

Purpose: To use a quantitative titration method to determine the % of acetic acid in vinegar. 
Also to learn the concept of neutralization, moles, dilution, and stoichiometry as it applies to titration data. This was accomplished by first determining what volume of standard hydrochloric acid (of known concentration) will neutralize a known volume of sodium hydroxide of unknown concentration, using titration with the aid of a pH indicator (see Equation 1). Once the concentration of sodium hydroxide was standardized, it was used to determine the concentration of acetic acid in a diluted unknown vinegar solution (see Equation 2).

A minor objective of the experiment was to learn how to properly clean, operate and read a burette.

Equation 1: \( \text{HCl (aq)} + \text{NaOH (aq)} \rightarrow \text{NaCl (aq)} + \text{H}_2\text{O (l)} + \text{heat} \)

Equation 2: \( \text{CH}_3\text{COOH (aq)} + \text{NaOH (aq)} \rightarrow \text{CH}_3\text{COO}^-\text{Na}^+\text{(aq)} + \text{H}_2\text{O (l)} + \text{heat} \)

Procedure: The experiment was carried out as outlined in Experiment AX of the Chemistry 217 Home Lab Manual, pp. xx-yy. Note: 1.60 g of NaOH was dissolved in ~400 mL water in Part A step 1. Otherwise no changes or modification were made.

Observations: Part A. During Titration of 0.1000 N HCl with Unknown [NaOH]:
1. Cresol red pH indicator in HCl initially reddish-orange.
2. After addition of NaOH, at the endpoint solution was pink in colour.

Part B. During Titration of CH\(_3\)COOH with Standardized NaOH:
1. Phenolphthalein pH indicator in CH\(_3\)COOH initially was clear and colourless.
2. After addition of NaOH, at the endpoint, the solution was pale pink in colour.

Results: Part A. Standardization of Sodium Hydroxide Solution

Given: Standard hydrochloric acid, [HCl] = 0.1000 N or M

Volume 0.1000 N HCl used = 25.00 mL

Indicator Used = cresol red

Table 1. Part A. Titration Data for 0.1000 N HCl vs. Unknown [NaOH] Titrant

<table>
<thead>
<tr>
<th>Burette Reading</th>
<th>Trial 1</th>
<th>Trial 2</th>
<th>Trial 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final NaOH Volume (mL)</td>
<td>25.00</td>
<td>49.98</td>
<td>25.02</td>
</tr>
<tr>
<td>Initial NaOH Volume (mL)</td>
<td>0.00</td>
<td>25.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Volume NaOH Used (mL)*</td>
<td>25.00</td>
<td>24.98</td>
<td>25.02</td>
</tr>
<tr>
<td>Avg. Vol. NaOH Used (mL)^</td>
<td></td>
<td></td>
<td>25.00</td>
</tr>
</tbody>
</table>
Results (cont.): Sample Calculations for Part A

* Volume Used = Final Vol. – Initial Vol.

^ Average of all three trials = x

\[ x = \frac{\sum \text{Trials 1, 2, and 3}}{n} \]

\[ \frac{25.00 \text{ mL} + 24.98 \text{ mL} + 25.02 \text{ mL}}{3} \]

= 25.00 mL

Amount in moles of 25.00 mL of 0.1000 M HCl present in flask:

Given: \( \text{moles} = M \times L \), therefore, \( 0.1000 \text{ N} \times 0.02500 \text{ L} = 2.50 \times 10^{-3} \text{ moles of HCl} \)

Number of moles of NaOH reacted at titration endpoint:

Given: the stoichiometric relationship between HCl and NaOH is 1:1 (see rxn equation 1 in Purpose)

Thus: moles NaOH = moles HCl = \( 2.50 \times 10^{-3} \text{ moles of NaOH} \)

Concentration of Standardized NaOH Solution

Given: \( M = \text{moles/L} \), therefore, \( 2.50 \times 10^{-3} \text{ moles NaOH/0.02500 L} \)

\( \text{NaOH} = 0.1000 \text{ M NaOH} \)

Part B. Determination of Unknown [Acetic Acid] Using Standardized NaOH Solution

Given: Standardized, \([\text{NaOH}] = 0.1000 \text{ M} \) (from Part A)

Volume diluted \( \text{CH}_3\text{COOH} \) used = 10.00 mL

Indicator Used = phenolphthalein

Table 2. Part B. Titration Data for Unknown [Acetic Acid vs. Standardized NaOH

<table>
<thead>
<tr>
<th>Burette Reading</th>
<th>Trial 1</th>
<th>Trial 2</th>
<th>Trial 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final Vol. NaOH (mL)</td>
<td>24.30</td>
<td>47.76</td>
<td>24.16</td>
</tr>
<tr>
<td>Initial Vol. NaOH (mL)</td>
<td>1.02</td>
<td>24.35</td>
<td>0.83</td>
</tr>
<tr>
<td>Volume NaOH Used (mL)*</td>
<td>23.28</td>
<td>23.31</td>
<td>23.33</td>
</tr>
<tr>
<td>Avg. Vol. NaOH Used (mL)^</td>
<td>23.31</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Sample Calculations for Part B

* Volume Used = Final Vol. – Initial Vol.

^ Average of all three trials = x

\[ x = \frac{\sum \text{Trials 1, 2, and 3}}{n} \]

\[ \frac{23.28 \text{ mL} + 23.31 \text{ mL} + 23.33 \text{ mL}}{3} \]

= 23.31 mL
Amount in moles of 0.1000 N NaOH consumed in neutralization reaction:
Given: moles = M × L, therefore, 0.1000 N × 0.02331 L = 2.331 × 10⁻³ moles of NaOH

Results (cont.): Number of moles of CH₃COOH reacted at titration endpoint:
Given: the stoichiometry between CH₃COOH and NaOH is 1:1 (see equation 2 in Purpose)
Thus: moles NaOH = moles CH₃COOH = 2.331 × 10⁻³ moles of CH₃COOH

Concentration of Diluted Acetic Acid Solution
Given: M = moles/L, therefore, 2.331 × 10⁻³ moles of CH₃COOH/0.01000 L
CH₃COOH = 2.331 × 10⁻¹ M CH₃COOH

Concentration of Undiluted Acetic Acid Solution
Given: [Initial = [Diluted Unknown] × D.F., (D.F. = dilution factor = 1/dilution)
Therefore, 2.331 × 10⁻¹ M CH₃COOH × 100.00 mL/25.00 mL = 9.324 × 10⁻¹ M CH₃COOH

Mass of CH₃COOH in 1L of Undiluted Vinegar
Given: Molecular Weight of acetic acid = 60.06 g/mol and g/L = M × Mwt.
Therefore, 9.324 × 10⁻¹ M CH₃COOH × 60.06 g/mol = 60.00 g/L CH₃COOH

Mass Percentage of Acetic Acid in Vinegar
Given: Density of vinegar = 1.005 g/mL = 1.005 g/mL × 1000 mL/L = 1005 g/L
Therefore, 60.06 g/L CH₃COOH/1005 g/L) × 100% = 5.57% CH₃COOH in vinegar

Answers to Questions:

1. Write the net ionic equation for:
   a. HCl + NaOH

   \[
   \text{HCl (aq)} + \text{NaOH (aq)} \rightarrow \text{NaCl (aq)} + \text{H}_2\text{O (l)}
   \]

   \[
   \text{H}^+(aq) + \text{Cl}^- (aq) + \text{Na}^+ (aq) + \text{OH}^- (aq) \rightarrow \text{Na}^+ (aq) + \text{Cl}^- (aq) + \text{H}_2\text{O (l)}
   \]

   \[
   \text{H}^+(aq) + \text{OH}^- (aq) \rightarrow \text{H}_2\text{O (l)}
   \]

   b. Acetic acid + NaOH

   \[
   \text{CH}_3\text{COOH (aq)} + \text{NaOH (aq)} \rightarrow \text{CH}_3\text{COO}^- \text{Na}^+ (aq) + \text{H}_2\text{O (l)}
   \]

   \[
   \text{CH}_3\text{COOH (aq)} + \text{Na}^+ (aq) + \text{OH}^- (aq) \rightarrow \text{CH}_3\text{COO}^- \text{Na}^+ (aq) + \text{H}_2\text{O (l)}
   \]

   \[
   \text{CH}_3\text{COOH(aq)} + \text{OH}^- (aq) \rightarrow \text{CH}_3\text{COO}^- (aq) + \text{H}_2\text{O (l)}
   \]

Conclusion: The percentage of acetic acid in vinegar was determined to be 5.57% (w/w). The concentration of the sodium hydroxide solution used to titrate the acetic acid was determined to be 0.1000 M. The titration method proved to be easy to perform and very precise in measuring concentrations of acids and bases. It is worthy of note that error was introduced into the percentage of acetic acid measurement because of the use of uncalibrated pipettes and burettes.
Purpose: To learn the application and use of a simple spectrophotometer by determining the absorbance of light by a number of dilute solutions of chromium (III) nitrate solutions (see equation 1). The \( \lambda_{\text{max}} \) for chromium (III) ions is to be determined, then a calibration curve for several solutions of known concentration, and finally, the concentration of an unknown chromium (III) nitrate solution. A minor objective of the experiment is to learn the proper steps for the preparation of a stock solution as well as how to make dilutions.

Introduction: In this experiment, a simple spectrophotometer is used to measure the light-absorbing ability of chromium (III) nitrate solutions. A simple spectrophotometer is useful in quantitative assays because it operates on the principle of the Beer-Lambert Law, \( A = \varepsilon cl \), where \( A = \) absorbance, \( \varepsilon = \) molar extinction coefficient, \( c = \) concentration, and \( l = \) path length. This law states that the absorbance is directly proportional to the concentration, i.e., the concentration of a liquid can be determined by its absorbance.

\[
\text{Equation 1: } \text{Cr(NO}_3\text{)}_3\text{(aq)} + \text{H}_2\text{O(l)} \rightarrow \text{Cr}^{3+}\text{(aq)} + 3\text{NO}_3\text{(aq)}
\]

To achieve this, first we must determine the best wavelength of light to use to detect the chromium (III) ions. This is called determining the adsorption spectrum and its wavelength maximum or \( \lambda_{\text{max}} \). Second we determine the absorbance of six different known concentrations of chromium (III) nitrate solutions at the optimum wavelength, and then plot a graph of the absorbance versus concentration. Once a plot is obtained, it can be used to determine the concentration of an unknown concentration solution of chromium (III) nitrate by simply measuring its absorbance.


Part A. Preparation of a Stock Solution of Chromium (III) Nitrate

1. A 4.0 g mass of \( \text{Cr(NO}_3\text{)}_3\cdot9\text{H}_2\text{O} \), chromium (III) nitrate nonahydrate was weighed on a general balance and placed into a clean dry glass vial.
2. The mass of \( \text{Cr(NO}_3\text{)}_3\cdot9\text{H}_2\text{O} \) plus vial, weighed on an analytical balance, was 12.6086 g.
3. The \( \text{Cr(NO}_3\text{)}_3\cdot9\text{H}_2\text{O} \) was then transferred to a clean 100 mL volumetric flask, using a funnel. The mass of the empty vial, weighed on an analytical balance, was 9.2068 g.
4. Water was used to dissolve the solid in the 100 mL vol. flask and bring the solution to a final volume of 100.00 mL.

Part B. Dilution of the Stock Solution

1. A 25 mL volumetric pipette was used to transfer a 25.00 mL aliquot of stock solution to a 50 mL clean dry volumetric pipette. The solution in the 50 mL vol. flask was filled to the graduation mark with water, then sealed, and mixed thoroughly by inversions (= Solution #1).

(Note: The rest of the procedure has been omitted here, for the sake of brevity. Of course, in your lab report, you would continue to report on the full procedure in a similar manner to the above.)
Results and Calculations

Observations: Part A. Preparation of the Stock Solution
1. The chromate solution was ____________ in colour.

Part B. Preparation of Dilutions of Stock Solution
1. No observations to report other than diluted solutions made were progressively lighter in colour.

Part A. Preparation of the Chromium (III) Nitrate Stock Solution
1. Mass of chromium (III) nitrate required to prepare 100.00 mL of 0.1 M solution:
   Given: Mwt. of Cr(NO₃)₃ · 9 H₂O = 400.26 g/mol
   Since mass (g) = Concentration (M) × Mwt. (g/ mol) × Volume (L)
   mass (g) = 0.1 M × 400.26 g/ mol × 0.100 (L) = 4.0 g Cr(NO₃)₃ · 9H₂O

2. Actual mass of chromium (III) nitrate used to prepare 0.1 M stock solution:
   Mass of Cr(NO₃)₃ · 9H₂O + vial used = 12.6086 g
   Mass of 'empty vial' used = 9.2068 g
   Mass of Cr(NO₃)₃ · 9H₂O used = 3.4018 g

3. Actual concentration (M) of chromium (III) nitrate stock solution:
   Given: Mwt. of Cr(NO₃)₃ · 9H₂O = 400.26 g/mol
   Since Mwt. = g/mole, therefore, moles = g/Mwt., and since M = moles/L,
   Therefore M = (3.4018/400.26 g/mol)/0.100 (L) = 8.499 × 10⁻² M Cr(NO₃)₃ · 9 H₂O

Part B. Preparation of Dilutions of the Chromium (III) Nitrate Stock Solution
3. To calculate the final [Cr(NO₃)₃] for each diluted solution, use the formula:
   Given: M_i × V_i = M_f × V_f, or M_f = (M_i × V_i)/V_f
   e.g., Solution #1 M_f = (8.499 × 10⁻² M_i × 25.00 mL)/50.00 mL = 4.25 × 10⁻² M and for the calculation of the other 5 solutions, see Table 1 below.

   Table 1. Part B. Calculation of Concentration of Six Diluted Chromium (III) Nitrate Solutions
<table>
<thead>
<tr>
<th>Sol’n#</th>
<th>[Initial] M_i</th>
<th>Initial Volume (V_i)</th>
<th>Final Volume (V_f)</th>
<th>[Final] (M_f)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8.499 × 10⁻²</td>
<td>25.00</td>
<td>50.00</td>
<td>4.25 × 10⁻²</td>
</tr>
<tr>
<td>2</td>
<td>8.499 × 10⁻²</td>
<td>10.00</td>
<td>25.00</td>
<td>3.40 × 10⁻²</td>
</tr>
<tr>
<td>3</td>
<td>8.499 × 10⁻²</td>
<td>25.00</td>
<td>100.00</td>
<td>2.12 × 10⁻²</td>
</tr>
<tr>
<td>4</td>
<td>8.499 × 10⁻²</td>
<td>5.00</td>
<td>25.00</td>
<td>1.70 × 10⁻²</td>
</tr>
<tr>
<td>5</td>
<td>8.499 × 10⁻²</td>
<td>5.00</td>
<td>50.00</td>
<td>8.50 × 10⁻³</td>
</tr>
<tr>
<td>6</td>
<td>8.499 × 10⁻²</td>
<td>5.00</td>
<td>100.00</td>
<td>4.25 × 10⁻³</td>
</tr>
</tbody>
</table>

Part C. Determination of λ_max for Chromium (III) Nitrate Using a Spectronic 20
Water used as blank.
The 4.25 × 10⁻² M Solution #1 was used for all readings in wavelength scan.
Table 2. Part C. Wavelength Scan* and Determination of $\lambda_{\text{max}}$ for Chromium (III) Nitrate

<table>
<thead>
<tr>
<th>Wavelength, $\lambda$ in nm</th>
<th>Absorbance</th>
<th>Wavelength, $\lambda$ in nm</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td>375</td>
<td>0.582</td>
<td>575</td>
<td>0.740</td>
</tr>
<tr>
<td>380</td>
<td>0.670</td>
<td>580</td>
<td>0.731</td>
</tr>
<tr>
<td>385</td>
<td>0.750</td>
<td>585</td>
<td>0.720</td>
</tr>
<tr>
<td>390</td>
<td>0.800</td>
<td>590</td>
<td>0.700</td>
</tr>
<tr>
<td>395</td>
<td>0.850</td>
<td>595</td>
<td>0.682</td>
</tr>
<tr>
<td>400</td>
<td>0.885</td>
<td>600</td>
<td>0.650</td>
</tr>
<tr>
<td>405</td>
<td>0.875</td>
<td>605</td>
<td>0.620</td>
</tr>
<tr>
<td>410</td>
<td>0.835</td>
<td>610</td>
<td>0.589</td>
</tr>
<tr>
<td>415</td>
<td>0.790</td>
<td>615</td>
<td>0.544</td>
</tr>
<tr>
<td>420</td>
<td>0.740</td>
<td>620</td>
<td>0.505</td>
</tr>
<tr>
<td>425</td>
<td>0.680</td>
<td>625</td>
<td>0.468</td>
</tr>
</tbody>
</table>

*For the graph of the wavelength scan, please see page 1 of the appendix.

Part D. Absorbances of Diluted Chromium (III) Nitrate Solutions at $\lambda_{\text{max}}$ and Graphing of Calibration Curve†

Water used as blank. Solutions were read at 400 nm and 575 nm wavelengths.

Table 3. Part D. Absorbance of Six Diluted Chromium (III) Nitrate Solutions

<table>
<thead>
<tr>
<th>Sol’n #</th>
<th>[Cr(NO$_3$)$_3$]</th>
<th>Abs 400 nm</th>
<th>Abs 575 nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>$4.25 \times 10^{-2}$</td>
<td>0.885</td>
<td>0.682</td>
</tr>
<tr>
<td>2</td>
<td>$3.40 \times 10^{-2}$</td>
<td>0.710</td>
<td>0.548</td>
</tr>
<tr>
<td>3</td>
<td>$2.12 \times 10^{-2}$</td>
<td>0.470</td>
<td>0.348</td>
</tr>
<tr>
<td>4</td>
<td>$1.70 \times 10^{-2}$</td>
<td>0.355</td>
<td>0.277</td>
</tr>
<tr>
<td>5</td>
<td>$8.50 \times 10^{-3}$</td>
<td>0.181</td>
<td>0.173</td>
</tr>
<tr>
<td>6</td>
<td>$4.25 \times 10^{-3}$</td>
<td>0.105</td>
<td>0.083</td>
</tr>
</tbody>
</table>

†See Graphs section, pp. 23–27.

Part E. Determination of the Concentration of an Unknown Diluted Chromium (III) Nitrate Solution

Water used as blank. Solutions were read at 400 nm and 575 nm wavelengths.

<table>
<thead>
<tr>
<th>Sol’n #</th>
<th>[Cr(NO$_3$)$_3$]</th>
<th>Abs 400 nm</th>
<th>Abs 575 nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>X</td>
<td>?</td>
<td>0.226</td>
<td>N/D</td>
</tr>
</tbody>
</table>

N/D = not determined

From the calibration graph plotted in Part D, Solution X (absorbance of 0.226 units), which corresponds with a concentration of Cr(NO$_3$)$_3$ equal to $1.50 \times 10^{-2}$ M.

Discussion: In this experiment we learned how light can be used and measured and that all atoms and molecules absorb light of certain wavelengths. This was accomplished by testing the absorbance of chromium (III) nitrate solutions using a spectrophotometer. The adsorption spectrum of a $8.499 \times 10^{-2}$ M Cr(NO$_3$)$_3$ solution was found to have two $\lambda_{\text{max}}$, at 400 nm and 575 nm as seen in graph 1 (Appendix, page 1). The series of diluted stock solutions were found to have a linear relationship between absorbance and concentration as seen in graph 2 (Appendix page 2). Since the maximal value of absorbance occurs at the 400 nm $\lambda_{\text{max}}$, this wavelength was chosen to measure the absorbance of the unknown solution. Reading from the graph, we found that its concentration was $1.50 \times 10^{-2}$ M.
The major source of error in the experiment was obtaining an accurate reading for the higher absorbance levels (0.6–1.0). At the higher levels the meter had a narrowly spread scale with intervals that were not easily seen (as to be expected on a logarithmic scale). Other sources of error would include flaws in the glass cuvette, the manual graphing process, and the use of uncalibrated pipettes and volumetric flasks. Also only one set of dilution series were made and only single readings were done in the spectrophotometer. It would have been better to have done all dilution series and readings in triplicate in order to achieve a greater degree of precision and accuracy in the determination of the concentration, $1.50 \times 10^{-2} \text{ M}$, of the unknown chromium (III) nitrate solution.

**Answers to Questions:**

1. What coloured light is associated with electromagnetic radiation at $\lambda = 400 \text{ nm}$ and $575 \text{ nm}$?

   - $\lambda = 400 \text{ nm} =$ yellow transmitted, violet absorbed
   - $\lambda = 575 \text{ nm} =$ purple transmitted, green absorbed

   Thus the transmitted light is a mixture of the yellow and purple radiation, which gives the chromium (III) ion solution a greenish/blue colour.

**Conclusion:** Two $\lambda_{\text{max}}$ for chromium (III) nitrate were found at 400 nm and 575 nm. There was a linear relationship between absorbance and concentration as shown in the calibration curves, thus proving that the absorbance of a solution is directly proportional to concentration of the absorbing species, therefore quantitative analysis of coloured solutions could take place. By learning to use a Spectronic 20 spectrophotometer, we were then able to determine with a reasonable degree of accuracy, the concentration of an unknown chromium (III) nitrate solution to be $1.50 \times 10^{-2} \text{ M}$. 
Graphs

In both the practical and theoretical components of this course you will be required to plot and interpret a number of graphs. Experience has shown that this is one area in which many students are unaware of the standards required for university-level work. Thus, these notes have been included in this manual in order to alert you to some of the criteria, which your laboratory instructor will use when marking a graph that is submitted as part of a laboratory report.

In order to draw a graph you will require 1) some data, 2) a pencil, pen and ruler, and 3) some graph paper.

1. Data

When plotting a graph as part of a laboratory report, you will normally have obtained the data during the experiment. It is essential that you organize your data in such a way that your instructor can check your experimental results in the event that you are unable to obtain a satisfactory graph. Usually, the best way to present your data is in the form of a table.

2. Pencil, Pen and Ruler

To many of you reading this manual, this section may seem to be superfluous. However, you would be amazed to see some of the work that has been submitted to us in the past.

A pen should be used for writing the title of the graph across the top of the page, for labelling the axes, for marking the scales on the axes, and for writing your name on the paper. A pencil is used to mark the data points and to draw the actual graph. A ruler is used to assist you in drawing the best straight line through the data points; that is, if the points look as if they lie on a straight line rather than a curve. The ruler should also be used for drawing your axes.

3. Paper

With regard to paper, most chemistry instructors have seen an incredible variety of paper used by students to prepare graphs: blank paper on which students have drawn their own lines, regular lined writing paper with vertical lines added by hand, and so-called “graph paper” on which the squares are so large that the paper would more appropriately be used as a surface on which to play chess or checkers. The only graph paper accepted for use in this course is the type, which is popularly known as “metric graph paper” (i.e., 1 mm × 1 mm squares). The use of this particular type of paper will enable you to plot very precise graphs, and will give your graphs a suitably professional appearance. Graphs submitted on any other type of paper will be returned to the student, unmarked.
4. **Computer-generated Graphs**

   Although we encourage students to use a personal computer to produce easily read laboratory reports, we wish to caution you in regard to including computer-generated graphs in your reports. Such graphs rarely meet the requirements of a scientific report and should not be used. Students who feel that their software is capable of generating graphs of the required standard should discuss the matter with their laboratory instructor before investing any time in such an endeavour.

**Getting Started**

Let us imagine that you have some data and you are ready to begin plotting your graph. By convention, the independent variable (that is, the controlled factor) is marked off along the horizontal (x) axis, and the dependent variable (the varying factor) is marked off along the vertical (y) axis.

Thus, if we were to plot a graph of the concentration of a solution against time, time would be the independent variable and would be placed on the horizontal (x) axis. Similarly, in a plot of volume of gas against temperature, temperature would be regarded as the independent variable. In each case you must decide for yourself whether the short side of your paper is to be the x axis, or whether the paper should be turned around so that the longer side forms the bottom of the page and represents the x axis.

An important consideration when plotting a graph is the choice of a suitable scale. All too frequently a poor choice of scale makes a graph difficult to interpret, hides an important trend or deviation, or is simply inappropriate. A useful guideline is to select the scale in such a way that the graph fills as much of the available space as possible. It is possible to choose your scale so that the data appear to be “better” than they really are. This is a ruse which has been used by generations of chemistry students (and their instructors), and is also used by governments and private business in their publicity materials.

Let us consider an example to illustrate exactly what we mean by choosing an appropriate scale. Suppose that you have measured some physical property of a reaction mixture (e.g., concentration of a particular species) as a function of time. As indicated above, time is the independent variable and should be placed on the horizontal (x) axis. Whether you choose the short or long side of the paper to be your x axis will depend how convenient it is to choose scales that will enable you to make your graph as large as possible. Suppose that you have taken readings of the concentration of a certain species approximately every 10 minutes for two hours (i.e., 120 minutes). Notice that the short side of the graph paper is marked off into eighteen 1-cm blocks, with each block sub-divided into ten 1-mm blocks. (This type of graph paper is often designated as 10 mm/cm.) If you chose to let 1 cm represent 10 minutes—which might be particularly convenient as 1 mm
would then represent 1 minute—by choosing the short side of the paper as your x-axis, only two thirds (i.e., 12/18) of the axis will be used. Notice that if we allow the long side of the paper to be our x axis, we can use a scale of 1 cm = 5 minutes (or 2 mm = 1 minute) and 96% (i.e., 24/25) of the axis is used.

Two matters remain to be mentioned regarding the choice of scale. First, if you refer to the example just described, you should note that your final decision regarding which side of the paper should represent the x-axis cannot be made without considering the choice of scale for the y axis. Although the long side of the paper appears to be the best choice for the x-axis at the moment, this may have to be reviewed after the range of concentrations to be plotted has been considered. Second, do not become over-obsessed with the idea of using the largest possible scale. For example, when plotting the above data with the x-axis on the long side of the paper, you should resist the temptation of allowing 1 cm to represent 4.8 minutes just so the whole of the axis is used. By choosing this scale, 1 minute = 2.08 mm, and it becomes very difficult to plot data points that may have been taken at 11 minutes, 32 minutes, 53 minutes, etc.

Once you have decided on how your axes and scales are to be organized, the next step is to plot the data points. In courses at this level it is generally sufficient for you to mark the point with a dot and to place a small circle around the dot so that can be readily identified as a data point by your instructor.

**Straight-line Graphs**

By far the most useful graphs to chemists are those that result in a straight line. Such graphs are particularly useful in verifying or deriving laws. You may recall that the general form of a relationship that will give rise to a straight-line graph is

$$y = mx + c$$

where, m is the slope of the graph, and c is a constant which corresponds to the value of y when x is equal to zero. Of course, if the value of the constant itself is zero, the relationship simplifies to

$$y = mx$$

and the straight line which relates y and x passes through the origin.

You will encounter many such relationships in your general chemistry course(s). Frequently, the value of y increases as the value of x increases, but sometimes the value of y decreases as x increases. Graphs pertaining to the former type of relationship are said to have positive slopes, whereas graphs in which y decreases as x increases have negative slopes (see Figure I.1).
The slope of graph is sometimes defined as “rise over run,” i.e.,

$$slope = \frac{\text{rise}}{\text{run}}$$

and may be determined as follows:

Select two points that lie on the graph (these points should generally be as far apart as possible and should not be actual data points). Determine the values of x and y for each of these two points; in general terms these values may be expressed as $x_1, y_1$ for point 1, and $x_2, y_2$ for point 2. The slope of the graph may then be calculated as follows:

$$slope = \frac{(y_2 - y_1)}{(x_2 - x_1)}$$

The calculation of the slope of the graph and any other relevant calculations can be carried out at a convenient location on your sheet of graph paper. By doing this you will avoid the situation in which your tutor must flip through several sheets of paper in order to determine to which graph a given calculation belongs.
value of c can be found directly from the graph (see Figure I.2), or through calculation once the value of the slope (m) is known.

From the graphs shown in Figure I.2, it should be apparent that the value of the intercept, c, is equal to the value of y when x = 0.

We have now reviewed the process for obtaining the slope and/or intercept from a straight-line graph. However, some students experience difficulty in drawing the straight line once the data points have been plotted. Remember that when you are dealing with experimentally determined data points, the points themselves are unlikely to fall exactly along a straight line. When you draw the line through the points, draw the best straight line through all the points. For example, if you have five data points, three of which appear to lie “exactly” on the line with the other two points just above or below the line, you are not justified in neglecting the latter points. Instead, you should draw a line that passes as close as possible to all the points (see Figure I.3).

Figure I.3a Incorrectly drawn straight-line graph
Figure I.3b Correctly drawn straight-line graph

Occasionally, one data point may be so far away from what would appear to be the “best straight line” that it should be discarded (see Figure I.4). Sometimes the experimenter makes a genuine error (e.g., misreading a burette, or not mixing a solution adequately) and the resulting data point is not valid. If you feel that there is a good reason for rejecting one of your data points, by all means do so, but include your rationale for doing so within the body of your laboratory report.

Figure I.4 Example of a straight-line graph with an obviously erroneous data point
Checklist

You may find the following checklist useful when submitting graphs to your laboratory instructor.

Have I:

1. used the correct type of graph paper?
2. written my name on the graph paper? (Graphs easily become separated from written reports during the marking process.)
3. given my graph a title?
4. labelled the axes appropriately?
5. chosen a scale that is sensible and convenient?
6. marked all data points with a circle?
7. drawn the best straight line? (if appropriate)
8. completed all the required calculations and presented the experimental results clearly?

When you can answer “yes” to each of the above questions, mail in your report(s) and wait to hear the instructor’s glowing comments.

The object of these notes is to provide a brief review of the fundamentals of plotting graphs. Hopefully you were already familiar with most of the points raised, and you are now prepared to plot graphs of the data that you will collect in some of the experiments in the laboratory component of this course. If you would like some practice, some sample problems are given below. If you feel that you need additional information, we suggest that you contact Athabasca University library and request one of the books listed in this manual under the heading “Further Reading.”
Problems

1. The vapour pressure of water at various temperatures is shown in the table below. Plot a graph of temperature (x axis) against vapour pressure (y axis), and from your graph determine the value of the vapour pressure of water at 75.0°C.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Vapour Pressure (kPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>0.61</td>
</tr>
<tr>
<td>20.0</td>
<td>2.34</td>
</tr>
<tr>
<td>40.0</td>
<td>7.37</td>
</tr>
<tr>
<td>50.0</td>
<td>12.3</td>
</tr>
<tr>
<td>60.0</td>
<td>19.9</td>
</tr>
<tr>
<td>70.0</td>
<td>31.1</td>
</tr>
<tr>
<td>80.0</td>
<td>47.3</td>
</tr>
<tr>
<td>90.0</td>
<td>70.1</td>
</tr>
<tr>
<td>95.0</td>
<td>84.5</td>
</tr>
<tr>
<td>100.0</td>
<td>101.3</td>
</tr>
</tbody>
</table>

2. Each of the following mathematical equations represents a relationship which you will encounter in Chemistry 217 or 218. In each case specify

   a. exactly what you would plot in order to obtain a straight-line graph, and
   b. the slope of the graph so obtained.
   c. \( c = \frac{k}{t} \) where \( k \) is a constant, and \( c \) and \( t \) are variables.
   d. \( \log k = -\frac{E_a}{2.303 RT} \) where \( E_a \) and \( R \) are constants, and \( k \) and \( T \) are variables.

3. The volume of a fixed mass of gas at constant pressure is related to its temperature. The table below shows the volume of a sample of helium gas at various temperatures and a pressure of 10.0 kPa. Plot a graph of temperature (°C, x axis) against volume (L, y axis). From your graph

   a. determine the volume of the gas at 0°C, and
   b. determine the temperature at which the volume of the gas would be reduced to zero. (In practice the gas would liquefy before this point was reached.)

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Volume (L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-173</td>
<td>0.0853</td>
</tr>
<tr>
<td>-123</td>
<td>0.128</td>
</tr>
<tr>
<td>-73</td>
<td>0.171</td>
</tr>
<tr>
<td>-23</td>
<td>0.213</td>
</tr>
<tr>
<td>20</td>
<td>0.250</td>
</tr>
<tr>
<td>77</td>
<td>0.299</td>
</tr>
<tr>
<td>123</td>
<td>0.341</td>
</tr>
</tbody>
</table>

Answers to these problems may be found on page 30 of this lab manual.
Further Reading


Both of these books are available from the Athabasca University library.
Answers to Problems

1. At 75.0°C, the vapour pressure of water is 38.5 kPa.

2. a. plot c (y axis) against 1/t (x axis)
   b. slope = k
   c. plot log k (y axis) against 1/T (x axis)
   d. slope = $E_a/(2.303 \, R)$

3. a. From the graph, the volume of the gas at 0°C is 0.233 L.
   b. The temperature at which the volume of the gas reaches zero can be found in two ways:
      i. by extrapolation, or
      ii. by recognizing that the graph represents a relationship of the type
         $V = mT + c$
         where $m$ is the slope of the graph, and $c$ is the volume found in part (a). By measuring the slope, and substituting the values for $m$ and $c$ into the equation when $V = 0.0 \, L$, the corresponding temperature can be found. Whichever method is chosen, the temperature should be $-273°C$. 
Safety

In 1975, a survey carried out by Her Majesty’s Inspectors of Schools showed that of the 70,000 accidents reported in British schools, only two per cent occurred in a science laboratory. Although Athabasca University students are not attending laboratory sessions in Britain, and are more mature than most school children, this statistic is relevant to the laboratory component of Chemistry 217. Of the over 300 Athabasca University students who have recently attended supervised chemistry lab sessions, only two have suffered an accident (an acid splash, and a severe cut). The figures suggest that, although a laboratory is a potentially dangerous place to work, the chances of an injury-causing accident are relatively low. This situation exists because of the strict safety rules that are applied to students working in laboratories, and because of a willingness of both students and instructors to look out for unsafe practices and possible hazards at all times.

Some people will approach the laboratory component of their Athabasca University chemistry course with a certain amount of trepidation. In a sense, this is a good thing—no one can afford to adopt a complacent attitude towards laboratory safety. Most of the hazards that you are likely to face while performing the experiments at home are relatively minor and easily avoided, including:

**minor cuts**—most cuts can be avoided if a student never uses broken or cracked glassware, and is particularly careful when carrying out potentially dangerous operations, such as inserting glass tubing into a rubber stopper.

**burns**—burns usually occur when a student forgets that something which has just been heated on a hot-plate or in a heating mantle may be very hot.

**chemical spills**—spills can usually be avoided if students pay particular attention to the technique used when pouring chemicals from a container. Injury caused by spills can be minimized if students wear the appropriate protective clothing: safety glasses, gloves, and lab coat or apron.

Another possible danger is the presence of hazardous gases or vapours in the air. In this course we have attempted to keep the use (or production) of such materials to a minimum. This will protect you and your co-habitants from exposure to undesirable concentrations of toxic or otherwise unpleasant vapours.

When designing the home laboratory component of this course, we found it necessary to strike a balance between minimizing possible hazards and exposing you to a full range of techniques. By its very nature, chemistry often necessitates the handling of dangerous substances; if chemistry students are never exposed to such situations, we would never have any fully trained chemists. Having said this, perhaps we should reassure you that, provided you follow the safety rules that follow, we do not anticipate that any problems will arise.
Safety Rules

1. **Safety glasses must be worn at all times when performing the home laboratory experiments.** Wearers of prescription glasses may wear their own spectacles, but should be aware of the possibility that chemicals or flying glass could enter the eye through the gap between the temple and the frames of the glasses. Thus, in potentially hazardous situations, wearers of spectacles are advised to wear safety goggles or a safety mask over their prescription glasses.

   **Note 1.**
   Safety glasses will be provided by Athabasca University, and must be worn at all times—even when you are not actively using chemicals and glassware.

   **Note 2.**
   Contact lenses are not permitted for two reasons:
   a. If a chemical is splashed into the eye of a person wearing contact lenses, neither the normal tearing mechanism nor external irrigation (with water) is effective in removing chemicals from under the contact. The contact must first be removed before tearing and irrigation is effective; however, the contact may be difficult to remove because of the tight squeezing shut of the eye that occurs in response to the chemical in the eye. Since time is of the essence with a chemical burn, a delay caused by the necessity of removing a contact lens could have serious consequences.
   b. Soft contact lenses present an additional hazard. Any chemical (including vapours) that comes into contact with such a lens can diffuse into the interior of the lens, which then acts as a reservoir that can create additional exposure, even if the lens is removed and rinsed.

   **Note 3.**
   The correct emergency treatment for chemicals that enter the eye is to wash the injured eye thoroughly with plain water for 15 minutes. Medical attention should be sought for all eye injuries. At home, you must set up a sink, which can serve as an eye-wash fountain.

2. **A lab coat should be worn at all times.** You must purchase a lab coat in order to participate in the laboratory component of this course. A lab coat will not only make you look and feel like a chemist, but will also protect you and your clothes in the event that you inadvertently spill a chemical.

   While we are on the subject of clothes, dress sensibly. It can become very hot in the laboratory and you will not be comfortable working all day with a three-piece suit underneath your lab coat. Similarly, clothes worn while performing laboratory experiments tend to acquire a
chemical odour, and it may be advisable to not wear your better shirts and sweaters.

3. **Protect your feet by wearing sensible shoes.** Bare feet, open-toed sandals, etc., are not permitted. Spilling concentrated hydrochloric acid on your toe or cutting your foot on a piece of broken glass would result in a trip to the hospital. Avoid high-heeled shoes; remember that you will be on your feet for up to four hours on any given lab evening.

4. **Tie back long hair.** Long hair can be a fire hazard. Also, when you bend over to inspect the contents of a beaker containing a chemical, long hair can easily fall into that chemical. Not only could this damage your hair, but it could also ruin your experiment!

5. **Never run with chemicals in hand, and never be tempted to become involved in practical jokes or other horseplay.**

6. **On no account attempt an unauthorized experiment.**

7. **Eating, drinking and smoking are not permitted while performing laboratory experiments.** Toxic substances may contaminate food and drink. Smoking is a fire hazard. When you leave your designated laboratory area, wash your hands, particularly before eating.

8. **In the event of fire:**

   a. do not panic; many small fires can be extinguished without the use of a fire extinguisher, simply by cutting off the air supply. For example, when a flammable liquid catches fire in a beaker, the fire can be quickly put out by covering the beaker.

   b. Most of the fire extinguishers that you will encounter are of the ABC type, which means they are effective on fires involving trash, wood or paper (Class A), liquids and grease (Class B), and electrical equipment (Class C). These extinguishers are not effective on Class D fires, i.e., those involving active metals such as sodium and potassium. Fires involving the latter substances are unlikely to occur during the course of Chemistry 217, but you should be aware of the special problems that these materials can cause. When using a fire extinguisher, aim at the base of the fire and use a sweeping motion. Note that you should never attempt to extinguish a laboratory fire using water. (A possible exception might be to extinguish a burning paper towel by placing it in a sink and turning on the tap.)

   c. if your clothing catches fire, wrap yourself in a fire blanket (or a coat if no fire blanket is available) and roll on the ground.

9. **Report all accidents.** All accidents, however minor, must be reported to your tutor and the details entered in the accident book. If you are
involved in an accident, do not resume work until you have received the appropriate first aid or medical attention. Never work with open cuts on your hands, cover all small cuts and scratches with band aids.

10. **Always dispose of chemical wastes in the correct manner.** In general, most of the reagents you have used in this home lab course may be discarded by diluting them 100–1000 fold and then pouring them down the drain. For example, certain substances, such as dilute acids, solutions of harmless compounds (e.g., sodium chloride), etc., *may* be washed down the drain with copious amounts of water.

   In a supervised lab setting, you would find that you would be instructed to “pour excess reagents into the waste container provided.” In this case, you must ensure that waste chemicals are placed in the correct container—putting the wrong material into a container is potentially dangerous. Another thing that you should never attempt is to return used chemicals to their original containers. Be particularly careful to place any chlorinated hydrocarbons in the waste container designated for such substances.

11. **Clean your sink.** After you have rinsed the reagents by diluting them 100–1000 fold and then pouring them down the drain, clean your sink with a detergent and rinse the sink down thoroughly.

### Some General Advice Regarding Laboratory Work

1. People with clean and tidy work areas are less likely to be involved in accidents. Clean up all spills immediately. Any glassware containing chemicals that is left in a lab area should be clearly labelled with the details of the contents (e.g., concentrated nitric acid). You can use masking tape and a marker to label equipment.

2. Wear your lab coat at all times when working in your lab area, and wear protective nitrile gloves whenever handling corrosives and solvent. Do not store sharp objects (e.g., Pasteur pipettes) in your coat pocket.

3. When assembling apparatus or glassware, always check with the lab manual and video guide before proceeding with the experiment.

4. Handle all organic solvents (e.g., ethanol, methanol) with care. Most are flammable, and many have a long-term, cumulative effect on the body.

5. If a fire starts, extinguish it by covering it with a large pot. Unplug any electrical apparatus involved, and vacate the lab area in an orderly manner.
6. When diluting a concentrated acid, always **add the acid to the water**. Do so slowly, with stirring.

7. If you get acid on your clothing, neutralize it with **dilute ammonia** solution (1 mol · L\(^{-1}\)) and wash well with water.

8. If you get alkali on your clothing, wash it off with large quantities of water.

9. If you get any corrosive chemical on your skin, wash it off immediately and thoroughly with water and consult your tutor. Pay special attention to the safety notes given in bold type in the “Procedure” sections of the lab manual. These notes will inform you of any special precautions that you might need to take, and will also inform you if the “wash well with water” maxim does not apply.

10. If you spill a large quantity of acid on the table or floor, use crude sodium bicarbonate (baking powder) to neutralize the acid and then wash well with water.

11. Always check for any possible hazards associated with using a given chemical. The quickest way of doing so is to make certain that you read the label on the container from which the chemical is removed, and check the provided MSDS. See in Moodle:

12. In the event of a real emergency, it could be important for medical personnel to know certain facts about you, facts that they could not obtain if you were unconscious or in a severe state of shock. On the next page is a copy of a **Medical Information Form**. We advise you to fill out the form that you received, and paste it inside the front cover of your lab notebook. You might regard some of this information as being rather personal. However, keep in mind that normally we do not expect you to show us your lab notebook (see “Writing Laboratory Reports”) so confidentiality of your medical history should be maintained. If you still have doubts, keep in mind that, in the event of an accident, a significant other could be asked to put your lab notebook on your stretcher as they carry you off to the hospital.

13. As mentioned in the safety rules, all accidents that result in injury must be reported and recorded in the accident book. In addition, an “Accident Report Form” must be completed and returned to the course coordinator. A sample form is shown on the page after next.

**Note:** on broken glassware. The thermometer provided does not contain any mercury. It is an alcohol thermometer. Any glassware that is broken should not be returned with the kit. Please discard of it in your own broken-glass garbage system.

**Note:** The **Medical Information Form** on the next page is adapted from one suggested by Ben Ruekberg and David W. Ball, *Journal of Chemical Education*, 63, **A247** (1986).
Medical Information Form
Chemistry 217

Name: A. Student

Social Insurance Number: 123 456 789

Address: 4812, 43rd Street, Small Town, Alberta

Phone: 675-6111

Alberta Health Care Number: 987.65.432.123

Age: 35

Sex: M

Height: 173 cm

Weight: 68 kg

Chronic medical problems: Epilepsy

Current medical problems: None

Do you normally wear contact lenses? No

Physical disabilities: Partially deaf

Allergies to medication: Allergic to penicillin

Current medication being used: None

Personal physician: Dr. V. Rich

In case of emergency, please contact: Susan Student (wife) 675-6111

Special information: My religious beliefs prevent me from accepting a blood transfusion.
Medical Information Form
Chemistry 217

Name:
Social Insurance Number:
Address:
Phone:
Alberta Health Care Number:
Age:
Sex:
Height:
Weight:
Chronic medical problems:
Current medical problems:
Do you normally wear contact lenses?
Physical disabilities:
Allergies to medication:
Current medication being used:
Personal physician:
In case of emergency, please contact:
Special information:
Chemistry Laboratory Accident Form
(Home Labs)

Name of injured student: Alan Student

Date of incident: April 1, 2009

Time of incident: 2:06 p.m.

Course: Chemistry 217

Tutor: Stockwell Solution

Nature of injury: Glass tubing penetrated palm of right hand.

How injury incurred: Student was attempting to insert glass tubing into rubber stopper without using recommended lubricant.

First aid rendered: Wound was washed thoroughly, a piece of glass appeared to be embedded in the hand. Pressure applied around the wound using a ring pad. Covered with built-up dressing.

First aid rendered by: Spouse

Further medical treatment sought? Yes; if yes give details: Patient was driven to outpatients at the nearest hospital, where the wound was examined and the embedded glass removed.

Student’s signature: A. Student

Follow up (course coordinator): Contacted student by phone (April 3), his condition is now being monitored by his family physician.
Chemistry Laboratory Accident Form
(Home Labs)

Name of injured student:

Date of incident:

Time of incident:

Course:

Tutor:

Nature of injury:

How injury incurred:

First aid rendered:

First aid rendered by:

Further medical treatment sought?

Student’s signature: ____________________

Follow up (Course Coordinator):
WHMIS

On October 31, 1988, the Workplace Hazardous Materials Information System (WHMIS) went into effect. This is a national system intended to provide laboratory personnel with uniform information on chemicals used in the workplace. There are three main features of WHMIS:

1. Chemical manufacturers are now obliged to label each container of hazardous material, giving details on the product’s hazards and what action to take in an emergency.

2. The manufacturer must provide the consumer with a Material Safety Data Sheet (MSDS) for each hazardous product. These sheets give complete details on the possible health effects that exposure to the product can produce, preventative measures that should be taken, etc.

   For CHEM217 MSDS information see in Moodle:


3. Employers must provide an appropriate education program for all workers whose work may bring them into contact with hazardous products.

   The WHMIS regulations do not affect you as a student, although if you are involved in a chemistry-related job you should be familiar with them. Most of the chemicals that you will handle in this course are no longer in their original containers. Under the WHMIS regulations, such chemicals do not require detailed labels. However, you should read all labels carefully and pay special attention to the hazard warnings that appear throughout the laboratory manual. The hazard symbols that you may observe on certain chemical containers are reproduced on the following page. MSDS for all the chemicals used in Chemistry 217 are located on the CHEM217 Moodle home page (see link above). Additional information on WHMIS may be obtained from Work Safe Alberta website:

Hazard Symbols

**HAZARD SYMBOLS**

**CLASS A:** Compressed gas

**CLASS B:** Flammable and combustible material

**CLASS C:** Oxidizing material

**CLASS D:**
1. Materials causing immediate and serious toxic effect
2. Materials causing other toxic effects
3. Biohazardous infectious material

**CLASS E:** Corrosive material

**CLASS F:** Dangerously reactive material
Common Apparatus

On the following pages are shown sketches of some common pieces of equipment that are found in your home lab kit and in almost all chemistry laboratories. Familiarize yourself with the name of each piece of equipment before you begin your first experiment.

150 and 50 mL beakers (Experiments A2 and B1)

Erlenmeyer flask

Volumetric flasks (Experiments A2 and A3)

Volumetric pipette and blue pipette bulb (Experiments A2, A3, A4, and Project C)
‘Short’ ~10 mL Burette

Solution dropper
(Experiments B3, A1, A2, A3, A4, Proj. C)

Plastic funnel
(Experiment B4, A3 and Project C)

Alcohol burner
(Experiment B3)

Graduated cylinder
(Experiment A1)
Stir wire  
(Experiment B2)

Spatula  
(Experiments A1, A2, A3, A4, B1, B4, and Project C)  
Conductivity apparatus  
(Experiment B1)

Calorimeter setup  
(Experiment B2)  
Vial is inserted into foam block before use

Glass rod  
(Experiment B4)

Nichrome wire  
(Experiment B3)

Multi-cell row  
(Experiment B3)
Dialysis tubing: 30 cm long, cut into two equal lengths before using (Experiment B1)

Burette clamp, metal

SpectroVis Plus and USB cable (save the box and packaging materials)
Checklist of Equipment Contained in Each Student Kit

Each student is provided with a home lab kit containing the equipment listed below. Inform your tutor if any of this apparatus is missing. Similarly, please let your tutor know if you find that the glassware has been left dirty. Please note that for some experiments, you are expected to supply some of your own materials (e.g., Block B experiments).

### Block A Experiments and Project C Equipment List

**Equipment (Quantity)**

<table>
<thead>
<tr>
<th>Item</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>safety glasses—all experiments</td>
<td>1</td>
</tr>
<tr>
<td>general pan balance—A1, A2, A3, A4, B4, C</td>
<td>1</td>
</tr>
<tr>
<td>200 g calibration weight (or 100 g weight if Ohaus balance inside)</td>
<td>1</td>
</tr>
<tr>
<td>spatula—A1, A2, A3, A4, C</td>
<td>1</td>
</tr>
<tr>
<td>thermometer, alcohol filled + rubber stopper—A1, B2</td>
<td>1</td>
</tr>
<tr>
<td>pipette filler bulb, blue—A2, A3, A4, C</td>
<td>1</td>
</tr>
<tr>
<td>SpectroVis spectrophotometer +USB cable—A2</td>
<td>1</td>
</tr>
<tr>
<td>Cuvettes, square shaped—A2</td>
<td>1</td>
</tr>
<tr>
<td>stand and burette clamp—A3, A4, Project C</td>
<td>1</td>
</tr>
<tr>
<td>tweezers—A4</td>
<td>1</td>
</tr>
<tr>
<td>latex tubing + glass bead + syringe—A4</td>
<td>1</td>
</tr>
<tr>
<td>bent straw—A4</td>
<td>1</td>
</tr>
<tr>
<td>latex tubing + tube thru rubber stopper—A4</td>
<td>1</td>
</tr>
</tbody>
</table>

**Glassware/Plasticware (Quantity)**

<table>
<thead>
<tr>
<th>Item</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>vials + lids—A1, A4, C</td>
<td>2</td>
</tr>
<tr>
<td>50 mL graduated cylinder</td>
<td>1</td>
</tr>
<tr>
<td>50 mL beaker</td>
<td>1</td>
</tr>
<tr>
<td>150 mL beaker—A4, B1</td>
<td>1</td>
</tr>
<tr>
<td>1000 mL plastic beaker</td>
<td>1</td>
</tr>
<tr>
<td>1 mL volumetric pipette</td>
<td>1</td>
</tr>
<tr>
<td>5 mL volumetric pipette</td>
<td>1</td>
</tr>
<tr>
<td>10 mL volumetric pipette</td>
<td>1</td>
</tr>
<tr>
<td>10 mL volumetric flask</td>
<td>1</td>
</tr>
<tr>
<td>25 mL volumetric flask</td>
<td>1</td>
</tr>
<tr>
<td>50 mL volumetric flask</td>
<td>1</td>
</tr>
<tr>
<td>100 mL volumetric flask</td>
<td>1</td>
</tr>
<tr>
<td>~10 mL burette with Teflon™ stopcock—A3, Project C</td>
<td>1</td>
</tr>
<tr>
<td>10 mL plastic gas trap pipette</td>
<td>1</td>
</tr>
<tr>
<td>solution droppers</td>
<td>3</td>
</tr>
<tr>
<td>50 mL Erlenmeyer flask</td>
<td>1</td>
</tr>
<tr>
<td>125 mL Erlenmeyer flask—A4, C</td>
<td>1</td>
</tr>
<tr>
<td>small glass vial—A4</td>
<td>1</td>
</tr>
<tr>
<td>plastic cups—A2</td>
<td>10</td>
</tr>
</tbody>
</table>
Chemicals (Quantity)

- (1) carbon powder in vial — A1
- (1) tin powder vial — A1
- (1) recycled carbon powder vial — A1
- (1) recycled tin powder vial — A1
- (1) iron (III) chloride in vial, FeCl₃·₆H₂O (>2.16 g) — A2
- (1) dropper (~2 mL) 1M hydrochloric acid, HCl — A2
- (1) 25 mL 1M hydrochloric acid, HCl — C
- (1) 0.30–0.35 g ASA std. in vial
- (2) 5 mL 1M sodium hydroxide, NaOH — A2
- (10) Aspirin® or ASA tablets — A2, A3
- (1) >1.2 g sodium hydroxide, NaOH pellets — A3
- (1) 60 mL 0.1000 N hydrochloric acid, HCl — A3
- (1) cresol red indicator in small solution dropper — A3
- (1) phenolphthalein indicator in small solution dropper — A3
- (1) sulfamic acid (0.2xx g) — A4
- (1) sodium nitrite, NaNO₂ (>0.30 g) — A4
- (1) potassium iodate, KIO₃ (~0.32xx g) — C
- (1) potassium iodide, KI (>8.3 g) — C
- (1) 50 mL sodium thiosulfate Na₂S₂O₃, 0.1000 N — C
- (2) starch indicator in small solution dropper — C (2)

Block B Contents List

Equipment/Supplies (Quantity)

- (1) conductivity apparatus (9V batt.+leads) — B1
- alcohol thermometer + rubber stopper — B2
- (1) calorimetric vial in foam block — B2
- (1) coffee powder — B1
- (1) dialysis tubing, 30 cm
- (4) filter paper circles, Whatman #1
- (1) pair of nitrile gloves
- (3) plastic multi-cell rows — B3
- (1) rock salt — B1
- (1) Video clips / MSDS — please see CHEM217 Moodle home page
- (1) wire, stir — B2
- (1) wire, nichrome — B3

Glassware/Plasticware (Quantity)

- (1) alcohol burner — B3
- (1) 50-mL graduated cylinder
- (1) 50-mL beaker — B1
- (1) 150-mL beaker — B1
- (1) glass vial plus cap, preweighed — B4
- (1) plastic funnel — B4
- (2) test tubes, small
- (2) test tubes, large
- (1) stir rod, glass
- (1) 125-mL Erlenmeyer flask — B4
Chemicals (Quantity)

0 (1) vial of 4.0 g magnesium sulfate (MgSO$_4$·7H$_2$O) — B4
0 (2) vials of 0.12xx g magnesium metal powder (Mg(s)) — B2
0 (2) vials of 0.25xx g magnesium oxide (MgO(s)) — B2
0 (1) bottle of 10.0 mL 2-propanol
0 (1) bottle of 25.0 mL ammonia (3% solution of NH$_3$)
0 (4) bottles of 20.0 mL hydrochloric acid (1.0 M HCl)
0 (1) vial of >1.2 g plant food, 10-52-10 — B4
0 (1) bottle of 35 mL 95% ethanol

Cation Droppers* (1 each of the following:)

- ammonium — NH$_4^+$ (1.0 M NH$_4$NO$_3$)
- ammonium carbonate — (1.0 M (NH$_4$)$_2$CO$_3$)
- barium — Ba$^{2+}$ (saturated soln Ba(NO$_3$)$_2$)
- calcium — Ca$^{2+}$ (1.0 M Ca(NO$_3$)$_2$ · 4H$_2$O)
- hydrochloric acid — (1.0 M HCl)
- iron — Fe$^{3+}$ (1.0 M Fe(NO$_3$)$_3$ · 9H$_2$O)
- magnesium — Mg$^{2+}$ (1.0 M Mg(NO$_3$)$_2$ · 6H$_2$O)
- potassium salt — K$^+$ (1.0 M KNO$_3$)
- silver — Ag$^+$ (1.0 M AgNO$_3$)
- sodium hydroxide — (1.0 M NaOH)
- sodium salt — Na$^+$ (1.0 M NaNO$_3$)
- dipotassium chromate — (1.0M K$_2$CrO$_4$)
- plus 2 unknown cation solutions (i.e., 2 of the above)

*Individually sealed solution droppers containing ~3 mL of the following 1.0 M cationic salt solutions dissolved in distilled water:

If damage has occurred to the kit during shipping, or if you are missing anything, contact Neil Sexton, Rob Carmichael or Elaine Birkigt in the Athabasca University Central science lab at 1-800-788-9041, ext. 6277. For your information, all the MSDS are also included for the chemicals used in the kit.

Your Chemistry 217 home lab kit may be returned to any one of the four locations listed below:

1. **AU in Athabasca**: AU Science Lab, Athabasca University, 1 University Drive, Athabasca, AB T9S 3A3. (used provided waybill)

2. **AU-Edmonton Learning Centre**: 12th floor, 10030-107 St., N. Tower, Edmonton, AB T5J 3E4. (drop off location only)

3. **AU-Calgary Learning Centre**: 1040-7th Ave. SW, Calgary, AB T2P 3G9. (drop off location only)

4. **NAIT**: only when AU labs are in progress — Check lab schedule, 11762 - 106 St., Rm G-207. (drop off location only)

**Note**: The contents of the home lab kit may be changed from time to time or may vary from one location to another. Your tutor will advise you if any additional items should be included or if any items should be deleted from the list.
Experiments

Note: Students must complete the candle experiment and submit their reports before proceeding to Block A home lab experiments.

This experiment does not require the home lab kit and can be done with simple household materials. We encourage you do this experiment right away, while you arrange for the shipment of your home lab kit.
Candle Experiment

Both images were taken with 35 mm cameras with nearly identical lenses and film, and the scale factor for both images was controlled (i.e., the relative flame sizes are correct). The microgravity candle flame on the right measured 1.5 cm in diameter and the shutter speed for capturing the image was between 20–30 seconds! The normal candle flame on the left was filmed using a shutter speed of less than 1 second. Hence, microgravity flames are very dim, nearly invisible to the eye!

Note: The microgravity flame image was taken by Dr. Shannon Lucid, aboard the MIR OS.

The candle images were reprinted with permission of Dr. Daniel L. Dietrich of NASA’s John H. Glenn Research Center, Cleveland, OH.

For more information about candle flames in microgravity (CFM), please see http://microgravity.grc.nasa.gov/combustion/CFM/cfm_index.htm.
Candle Experiment

Prerequisite Skills

None – this should be the first experiment that you attempt.

Objectives

When you have completed the following experiment, you will have

1. reinforced your observational skills.
2. become familiar with the basic procedure of carrying out experimental work.
3. learned the correct techniques for writing a good laboratory report.

Candle Experiments

Our statistics draw that the average chemistry student has not done laboratory experiments within the last five years. In this exercise, we will carry out simple kitchen chemistry experiments that will allow you to sharpen your observational skills and get some quick feedback on your laboratory report writing skills. Remember you do not need your all-home microlab kit to do any of these experiments.

Introduction

In the winter of 1859, Michael Faraday, a famous English scientist, gave several lectures centered around the chemistry and physics of a candle! Faraday would tell his listeners:

There is not a law under which any part of this universe is governed which does not come into play and is touched upon in these phenomena. There is no better, there is no more open door by which you can enter into the study of natural philosophy than by considering the phenomena of a candle.

He would then proceed and set out to prove his point by lighting a candle and demonstrating all the processes involved. We will enter the lab portion of Chemistry 217 by that same door and will repeat some of the experiments that Mr. Faraday demonstrated more than a century ago. In so doing, we hope you will exercise your power of observation and sharpen some of your experimental skills to help prepare you for the more concentrated in-lab experiments you will be challenged with later on.
Materials Required

matches (or a lighter)
a small candle
baking soda (NaHCO₃)
vinegar (CH₃COOH)
aluminum foil
ice cubes
metal jar lid
a small bowl
two (2) small drinking glasses or glass jars

Theory

In burning a candle one starts with a solid fuel (wax), which is liquified, rising up into the wick by capillary action to be vapourized in the atmosphere, and then quickly oxidized by the candle flame. In burning, the candle produces energy in the form of heat and light. The burning process is a simple organic chemical reaction represented by the following equation:

\[
\text{wax} + \text{O}_2 \rightarrow \text{H}_2\text{O} (g) + \text{CO}_2 (g) + \text{heat} + \text{light}
\]

If one were to remove the fuel (wax), the oxygen or the initiator (flame) or any combination of the three, the candle would go out. Professional firefighters use this idea constantly when they develop strategies for fighting fires.

**Figure CE.1 Candle Flame**

Figure CE.1 shows a detailed diagram of the flame of a burning candle and will give you a better idea of the mechanics involved.
Procedure

A. Re-igniting a Candle

1. Light a candle and let it burn for about one minute.
2. Have a lighted match or lighter ready.
3. Carefully blow out the candle.
4. Watch the smoke trail coming up from the extinguished candle and quickly place your lighted match into that smoke stream.
5. The candle should re-ignite.

B. Water Suck Up

1. With a few drops of hot wax fix your candle to the center of a bowl.
2. Pour water into the bowl so that it is partially filled.
3. Light the candle.
4. Place an empty jar or glass over the candle so that the edge is under the water line (see Figure CE.2).
5. The candle should extinguish.

![Figure CE.2](Candle under glass)

C. Extinguishing a Candle with Aluminum Foil

1. Mold and cut a square of aluminum foil into the shape shown in Figure CE.3.
2. Place the foil between the candle and flame so that the wick goes through the slit in the foil.
3. The candle should extinguish (the slit may have to be narrowed manually).

![Aluminum foil](image)

**Figure CE.3**
Aluminum foil

### D. Candle Condensation

1. Place several ice cubes into a large square of aluminum foil.
2. Pull together the corners to form a sack filled with ice.
3. Hold this sack over (about 20 cm) a burning candle.
4. Water droplets should form on the outside of the aluminum sack.

### E. Extinguishing a Candle with Carbon Dioxide

1. Fix a candle in a glass jar and light it.
2. In the another glass jar add two tablespoons of baking soda.
3. Then add about 1/5 cup of vinegar to the baking soda.
4. The baking soda will foam.
5. Quickly tip the foaming glass and “pour” the carbon dioxide formed into the jar with the burning candle.
   **Note:** Be careful not to tip the wet vinegar/baking soda mixture into the candle jar.
6. The candle should extinguish.

### Observations and Results

Note that formally observations and results or explanations for the observations are treated separately in a laboratory report. Since these experiments are rather simple, we will note our observations and possible explanations for those observations in tabular form. You can
then incorporate this table into the short-report format described in the “Writing Laboratory Reports” section of this manual.

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Observations</th>
<th>Results and Explanations</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. Reigniting a Candle</td>
<td></td>
<td></td>
</tr>
<tr>
<td>B. Water Suck Up</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C. Extinguishing a Candle with Aluminum Foil</td>
<td></td>
<td></td>
</tr>
<tr>
<td>D. Candle Condensation</td>
<td></td>
<td></td>
</tr>
<tr>
<td>E. Extinguishing a Candle with Carbon Dioxide</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Candle Experiment Questions

1. Why did the candle reignite with a match held away from the wick?

2. a. Explain how the candle was extinguished with aluminium foil and carbon dioxide.
   
   b. Suggest and explain another method to extinguish a candle not already used in Part A of this experiment.
   
   c. Write out the balanced chemical equation for the reaction that produced carbon dioxide in this experiment.

3. What does the formation of water on the aluminium ice sack suggest about the chemistry of a burning candle?

4. a. Suggest a reason why the water is sucked up into the glass jar.
   
   b. A candle flame will use up oxygen as it burns. However, the consumption of oxygen alone does not explain the observed volume change. What other factor(s) need to be considered?

5. Explain the difference in the shapes of the two flames (Normal candle and the nearly identical candle in micro gravity flame) shown on page 51 of this manual.
Block A Experiments

Note: Students must complete the Candle Experiment before proceeding to rest of the experiments.

Safety Tips

- Reread your Safety Pledge.
- Watch the Video clips (see Moodle) for helpful working tips too. (Please note Experiments A2, A3 and Project C have been modified since the creation of the video clips. The video clips does not show the use of the SpectroVis Plus spectrophotometer or the use of the short 10 mL burette (with stopcock).}
Experiment A1:
Mass and Volume Measurement

Prerequisite Skills
You should have completed and submitted the Candle Experiment and have your home lab kit before continuing with this experiment.

Objectives
When you have completed the following experiment, you will have
1. learned how to use the general pan balance.
2. learned how to use a simple method for determining the density of a liquid.
3. learned how to “weigh by difference.”
4. learned the correct technique for using a pipette.
5. become familiar with the precision obtained when measuring liquid volumes using a pipette.

Safety Tips
Experiment location. Work on a steady table in a safe secure place. Keep doors and windows closed to prevent drafts.

Handling of chemicals. Carbon and tin powders is dusting. Avoid inhaling.

Safety glasses should be worn at all times. Wear appropriate clothes and closed-toed shoes.

The experiment also illustrates
1. the use of the mole method.
2. density calculations.
3. the use of dimensional analysis (the unit-factor method).
4. that a certain amount of uncertainty is associated with any volume measurement made in the laboratory, and how the precision of such
measurements can be assessed through the use of deviation, average deviation, and standard deviation.

5. the use of the relationship:

\[
\text{concentration (mol} \cdot \text{L}^{-1}) = \frac{\text{amount of solute (mol)}}{\text{volume of solution (L)}}
\]

Introduction and Theory

The measurement of volume and mass are two fundamental skills that every laboratory worker must master. In this experiment you will be measuring both mass and volume. The volume measurements that you will make will be carried out using either a measuring cylinder (sometimes called a graduated cylinder) or a pipette. Measuring cylinders are used when the volume of the liquid being measured is needed to relatively low precision. A pipette is used when the precise measurement of a specific volume is required.

![Figure A1.1](image)

Pictures of the supplied Precision GX-230 (left), or Denver (middle) or Ohaus (right) general pan weigh balances (see pages 69–70 for instruction on its use and operation)

Most laboratories have two different types of balances available for measuring the mass of an object. The first type is the general-purpose balance, such as an electronic top-loading balance or the older triple-beam balance, which is usually capable of measuring to two decimal places (i.e., to 0.01 g). In your microlab kit, you will find an electronic top-loading balance (see Figure A1.1). The second type is the analytical balance, which is usually electronic in more modern laboratories, and mechanical in older facilities. Analytical balances can measure to four or five decimal places (i.e., to 0.0001 or 0.000 01 g). These balances are too expensive and sensitive to include in a portable microlab kit for home use. Some typical balances are shown in Figure A1.2.
A. Weighing by Difference

If you need to use a precisely known mass of a given substance in an experiment, you would normally measure this mass of substance using a
A process known as “weighing by difference.” A quantity approximately equal to the desired mass is measured into a vial, the mass of the vial and its contents is determined precisely \((m_{v+c})\), and the substance is then tipped into a suitable container. The mass of the “empty” vial is determined \((m_{MTv})\), and the mass of substance that was transferred is given by the equation:

\[
mass \ of \ substance = mass \ of \ vial \ and \ contents \ (m_{v+c}) - mass \ of \ "empty" \ vial \ (m_{MTv})
\]

\[B. \ The \ Mole \ Concept\]

A mole is defined as that mass of substance that contains the same number of particles as exactly 0.012 kg of carbon-12. This number turns out to be \(6.02 \times 10^{23}\), Avogadro’s number. Thus, 1.00 mol of iron contains \(6.02 \times 10^{23}\) atoms of iron, 1.00 mol of oxygen gas contains \(6.02 \times 10^{23}\) molecules of oxygen \((O_2)\), and 1.00 mol of sodium chloride consists of \(6.02 \times 10^{23}\) formula units of sodium chloride (where a formula unit of sodium chloride consists of one sodium ion and one chloride ion). As you cannot measure numbers of atoms, molecules or formula units directly, when you wish to measure a certain number of moles (in SI terms, a certain amount) of given substance you would normally measure its mass, and then convert to moles using the relationship:

\[
amount \ (number \ of \ moles) = \frac{mass}{molar \ mass}
\]

where molar mass = mass (in grams) of one mole of the substance in question. Thus, if you wish to determine the number of moles of iron present in 10.00 g of this metal, you would write:

\[
amount \ of \ Fe = \frac{10.00 \ g}{55.88 \ g \cdot \ mol^{-1}} = 0.1791 \ mol
\]

If you prefer to use the unit-factor method, the molar mass of a substance may be regarded as a unit factor, in this case \((55.88 \ g \ Fe)/(1 \ mol \ Fe)\), although here you must invert the factor before you can use it. Thus:

\[
amount \ of \ Fe = 10.00 \ g \ Fe \times \frac{1 \ mol \ Fe}{55.88 \ g \ Fe} = 0.1791 \ mol \ Fe
\]

\[C. \ Density\]

If you want to use a certain mass of a particular liquid in order to carry out an experiment, you will usually find it more convenient to calculate the volume required and measure this quantity, rather than try to measure the mass directly. You would make use of the relationship:
Experiment A1

\[ \text{density} = \frac{\text{mass}}{\text{volume}} \]

**Note:** Although the mass of a substance or object does not change with temperature, its volume and density do. You must take this fact into account when you are trying to obtain results that are accurate and precise.

**Table A1.1**
The relationship between density of water (g/mL) vs. temperature (°C)

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>Density</th>
<th>Temp (°C)</th>
<th>Density</th>
<th>Temp (°C)</th>
<th>Density</th>
<th>Temp (°C)</th>
<th>Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>-30</td>
<td>0.983854</td>
<td>5</td>
<td>0.99996</td>
<td>21</td>
<td>0.99799</td>
<td>38</td>
<td>0.99299</td>
</tr>
<tr>
<td>-20</td>
<td>0.993547</td>
<td>6</td>
<td>0.99994</td>
<td>22</td>
<td>0.99777</td>
<td>40</td>
<td>0.99224</td>
</tr>
<tr>
<td>-10</td>
<td>0.998117</td>
<td>8</td>
<td>0.99985</td>
<td>23</td>
<td>0.99754</td>
<td>50</td>
<td>0.98807</td>
</tr>
<tr>
<td>0</td>
<td>0.99984</td>
<td>10</td>
<td>0.99973</td>
<td>24</td>
<td>0.99730</td>
<td>60</td>
<td>0.98324</td>
</tr>
<tr>
<td>1</td>
<td>0.99989</td>
<td>12</td>
<td>0.99950</td>
<td>25</td>
<td>0.99707</td>
<td>70</td>
<td>0.97781</td>
</tr>
<tr>
<td>2</td>
<td>0.99994</td>
<td>14</td>
<td>0.99924</td>
<td>26</td>
<td>0.99678</td>
<td>80</td>
<td>0.97183</td>
</tr>
<tr>
<td>3</td>
<td>0.99996</td>
<td>16</td>
<td>0.99894</td>
<td>28</td>
<td>0.99623</td>
<td>90</td>
<td>0.96534</td>
</tr>
<tr>
<td>3.98</td>
<td>1.00000</td>
<td>18</td>
<td>0.99862</td>
<td>30</td>
<td>0.99567</td>
<td>95</td>
<td>0.96192</td>
</tr>
<tr>
<td>4</td>
<td>0.99997</td>
<td>20</td>
<td>0.99823</td>
<td>35</td>
<td>0.99406</td>
<td>100</td>
<td>0.95838</td>
</tr>
</tbody>
</table>


**Note:** Numbers in normal text are from page F-4, Volume properties of water, at 1 atm. Numbers in italics were obtained from page F-10, Density of water, for pure water, free from air.

**D. Calibrating a Pipette**

A volumetric pipette is designed to deliver a specific volume of liquid at a given temperature. The volume and the temperature should be marked on the pipette, as should the letters TD. These letters stand for “to deliver,” and you will see their significance in a few moments. In the first part of this experiment, you will fill a 5 mL pipette with water, allow the water to run out into a beaker of known mass, and then measure the mass of the beaker plus its contents. From this measurement, you will determine the mass of water delivered by the pipette. You will then determine the temperature of the water (which you can assume to be equal to the room temperature unless you are told otherwise) and you will look up the density of water at that temperature in a suitable reference book, such as *The Handbook of Chemistry and Physics*, or from Table A1.1, and determine the volume of water delivered by the pipette.

Sample results:

Mass of beaker + water = 54.95 g
Mass of beaker = 50.05 g
Mass of water = 4.90 g
Temperature of water = 23.2°C

Density of water at 23.2°C = 0.99749 g·mL⁻¹* 

Volume of water delivered by pipette = \( \frac{\text{mass of water}}{\text{density of water}} \)

\[ = \frac{4.90 \text{ g}}{0.99749 \text{ g·mL}^{-1}} \]

\[ = 4.91 \text{ mL} \]

The procedure on the previous page will be repeated four more times, and the average volume delivered by the pipette will be determined, together with the average deviation and the standard deviation. The following relationships are used to determine the two deviations, both of which may be used to assess the precision of your result:

1. For each of your calculated volumes:
   
   deviation (d) = observed value – average value

2. Average deviation, \( a \), is found using
   
   \[ a = \frac{\sum |d|}{n} \]
   
   i.e., by dividing the sum of the deviations, regardless of sign, by the total number of observations, \( n \).

3. Standard deviation, \( \sigma \), is found using
   
   \[ \sigma = \sqrt{\frac{\sum d^2}{n - 1}} \]
   
   where, as before, \( d \) represents the individual deviations and \( n \) represents the number of trials.

   Thus, if you obtain individual results of 4.99 mL, 4.96 mL, 4.96 mL, 4.97 mL, and 5.02 mL, your average volume (i.e., the arithmetic mean) is \( 24.90 \text{ mL} \div 5 = 4.98 \text{ mL} \).

   The individual deviations are +0.01 mL, -0.02 mL, -0.02 mL, -0.01 mL, and +0.04 mL. Thus,
   
   \[ \sum |d| = 0.01 + 0.02 + 0.02 + 0.01 + 0.04 \]
   
   \[ = 0.10 \text{ mL} \]

   and the average deviation, \( a \), is 0.10 mL/5 or 0.02 mL.

   Similarly, the standard deviation may be determined as follows:

* Table A1.1 was used and the density at 23.2°C was interpolated between 23°C and 24°C, assuming a linear relationship.
\[
\sum |d|^2 = (1 + 4 + 4 + 1 + 16) \times 10^{-4} = 26 \times 10^{-4} \text{ or } 2.6 \times 10^{-3}
\]

and
\[
\sigma = \sqrt{\frac{\sum d^2}{n-1}} = \sqrt{\frac{2.6 \times 10^{-3}}{4}} = \sqrt{6.5 \times 10^{-4}}
\]
or 0.02 when rounded off to the correct number of significant figures.

The average deviation gives an approximate estimate of the precision of the result, but has no statistical significance. The standard deviation is statistically more significant, and its significance increases as the number of trials (i.e., n) increases. The standard deviation is usually greater than the average deviation; therefore, many people choose the latter when reporting experimental results. In the above example there is no significant difference between the two deviations, and the volume delivered by the pipette should be reported as:

4.98 (±0.02) mL

Thus, the volume delivered by the pipette is probably between 4.96 mL and 5.00 mL. If you need to use this volume in some kind of calculation, a reasonable value to use is 4.98 mL, as the significant-figure rules would indicate some uncertainty in the second figure after the decimal.

“'To most people, solutions are answers, but to chemists, solutions are things that are still all mixed up.’”

—a 6th grader
Chemical and Material Requirements

general pan balance (calibrated)

2 vials + lids
spatula
carbon powder in vial
tin powder vial
recycled carbon powder vial
recycled tin powder vial

50 mL graduated cylinder
tap water
solution dropper
thermometer (alcohol filled)

50 mL beaker
5 mL volumetric pipette
pipette filler bulb(s)
150 mL beaker plus ~70 mL tap water
soapy water for cleaning

List of Reagents

<table>
<thead>
<tr>
<th>Compound</th>
<th>Chemical Name</th>
<th>Molecular Weight</th>
<th>Hazardous Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>C, powder</td>
<td>carbon powder</td>
<td>12.011 g/mol</td>
<td>Dust irritant</td>
</tr>
<tr>
<td>Sn, powder</td>
<td>tin powder, recycled</td>
<td>118.69 g/mol</td>
<td>Dust toxic</td>
</tr>
<tr>
<td>H₂O</td>
<td>water</td>
<td>18.01534 g/mol</td>
<td>Scalds when hot!</td>
</tr>
</tbody>
</table>
How to Use Your Precision GX-230 General Pan Balance

It is recommended that you carefully read the following information before unpackaging and using your general pan balance.

Power source
The GX-230 and Denver balances operate using 115V power supply and the 115V AC adaptor provided.

Unpacking Your Balance and Turning It On/Off

The Precision GX-230 balance is shipped in styrofoam packaging with the AC adaptor disconnected from the back of the balance, and the stainless steel pan in its own plastic bag. The balance is packaged in a plastic covering too. If you were shipped a Denver or Ohaus balance, please use essentially the same instructions for unpacking your balance. Please also consult the provided Denver or Ohaus instructions for their proper operation.

1. Open the box and take out the AC adaptor, the stainless steel pan and the balance and the 200 g calibration weight.

2. Assemble the unit on a sturdy bench or table. Gently place the pan on top of the of the balance and line up the four peg into the four holes on top the balance. Again gently press the pegs down into the holes until they come to rest.

3. Connect the power adaptor to the back of the balance and plug it into a 115 V power supply. Press the ON button on the keypad of the balance. The balance will go through an internal self test and test the digital display. This takes ~ 3 seconds and eventually it should display a reading of 0.00 g (or very near to 0.00). Allow the unit 2 minutes to warm up before using. Calibrate the balance before using (see next page).

4. To turn off your balance, press the OFF button on the keypad of the balance.
Precision GX-230 Balance Calibration Procedure

Perform the following procedure upon first use of your balance, and perform it again should you ever move your balance to a new work location. If you were shipped a Denver or Ohaus balance, please use the provided Denver or Ohaus calibration instructions.

1. Gently assemble the unit on a sturdy bench or table (see unpacking instructions above), and connect the power adaptor to the back of the balance and plug it into a 115 V power supply.

2. Press the “ON” button on the keypad of the balance and allow the unit 2 minutes to warm up before using.

3. Press “TARE” to electronically zero the balance.

4. Press and hold down the “CAL” button until the display starts flashing “200.00” (the expected weight of the calibration weight to be placed on top of the pan).

5. With the display still flashing “200.00,” press the “OFF” key. The display should now show “Cal 0.” Wait for three seconds and then proceed to step 6.

6. Place the 200.00 g calibration weight on the center of the ss pan tray.

7. The display will now show “Cal F,” and then return on its own to normal operating mode. The calibration is complete when the display shows the weight that is placed on the pan and the display has stabilized.

8. Remove the calibration weight and press “TARE” again to get a 0.00 reading.

9. The balance is now ready for use.

Taking Care of Your Balance

This is a precision instrument. Be careful not to drop, bang, or place too much weight on the balance (max. load is 200 g or < ½ lb). Do not force or drop items to be weighed on to the tray top in order to avoid sensor damage. Do not expose this instrument to prolonged periods of heat or cold. The temperature for optimum performance is 64–77°F or 18–25°C. And finally, try to minimize air currents around the balance work area.

Packaging Your Balance for Shipment

It is important to ship the balance back to Athabasca University in the packaging in which it was sent to you.
Procedure (total time for completion ~ 2 hours)

Measurement of Mass and Volume

A. Weighing by Difference and the Mole Concept

Note: Chemicals are never measured out directly on the pan of a balance. Always use a vial (as in this experiment) or a piece of weighing paper.

1. Measure the mass of a vial and its lid, using the supplied general-purpose balance. Remove the vial from the balance before you proceed to step 2.

2. Add some carbon to the container, check its weight on the balance, and continue adding until you have approximately one gram of carbon in the vial (remember it need not be exactly one gram). Remember to remove the vial from the balance before you make each addition of carbon.

3. Measure and record the mass of the carbon filled vial and lid.

4. Pour the contents of the vial into a small (e.g., 50 mL) beaker. Determine the mass of the empty vial on the general pan balance.

5. The mass of substance actually transferred to the beaker is the difference between the mass found in steps 3 and 4, hence the phrase “weighing by difference.”

6. Obtain a clean vial and repeat the above procedure, using powdered tin. Place the used carbon and tin in the back into their original bottles and return to Athabasca University for disposal.

SAFETY NOTE: A chemical should never be returned to its original container, even if it has not been used.

B. Density of Water

1. Determine the mass of an empty 50 mL graduated cylinder, using a general-purpose balance.

2. Add approximately 25 mL of water to the graduated cylinder, and determine the volume of water contained by the cylinder. (In other words, you do not need to have exactly 25 mL of water, but you must know as precisely as possible the volume that you did use.) It should be possible to read the volume to the nearest 0.5 mL (see Figure A1.3). Note that the bottom of the meniscus is used when the volume of liquid contained in a graduated cylinder is determined.
3. Determine the mass of the cylinder and its contents, again using the general-purpose balance.

4. Determine the temperature of the water, and look up the density in Table A1.1 on page 65 of the Chemistry 217 Home Lab Manual. Dispose of the water by pouring it down the drain.

C. Calibration of a Pipette

You have probably used a pipette before in a high school chemistry course. The emphasis in this experiment is to teach you to use one correctly and safely. Thus, you should follow each step in the procedure very carefully.

1. Determine the mass of a clean, dry, 50 mL beaker using the supplied general pan balance.

2. Cleaning the pipette. Never assume that the pipette that you are about to use is clean. Begin by squirting some soap solution (1 drop dish soap/100 mL water) into the top of a 5 mL pipette until you have several millilitres of solution in the pipette. Hold the pipette horizontally and rotate it in your hands so that the entire internal surface comes into contact with the soap solution. Allow the solution to drain out through the tip and then rinse the pipette with tap water. Rinse again using water and allow the water to drain from the tip. If the pipette is clean, no droplets of water should adhere to the internal surface. One more rinse (see below) and the pipette is ready for use.

3. Measuring a specific volume of liquid using a pipette. The first step in measuring a specific volume (in this case 5 mL) of liquid with a pipette is to rinse the pipette using some of the liquid in question. Draw some of the liquid into the pipette using a pipette filler (see instructions on page 73), wet the internal surfaces of the pipette by holding it horizontally and rotating (see above), and then allow the liquid to drain out. At this stage it is not essential that you fill the pipette to the graduation mark, but, apart from that, the technique to use for drawing liquid into the pipette is as described below.
Obtain ~100 mL of tap water in a 150 mL beaker. Draw water into the 5 mL volumetric pipette using a pipette filler (see instructions on page 73 so that you fill the pipette to the graduation mark. Carefully transfer the 5 mL of water into the empty beaker.

4. Determine the mass of the beaker and its contents on an analytical balance.

5. Empty the beaker into the sink. Carefully dry the beaker with tissues, and then determine the mass of beaker on the analytical balance again.

6. Repeat steps 3, 4 and 5 four more times, cleaning and weighing the beaker between each trial.

7. Determine the temperature of the water.

8. Look up the density of water at this temperature, using Table A1.1 on page 65 of this manual.

9. The purpose of this exercise has been to calibrate your pipette. That is, to determine the precise volume delivered by the pipette at normal room temperature.

**Pipette Fillers**

Many types of pipette filler are available: a simple bulb type, shown in Figure A1.4 and a 3 valve bulb shown in the Glossary G1.6, and the pipump 2500, shown in Figure G1.7. We have provided you with one that is the simplest to use but it still requires a certain amount of dexterity.

**Simple Bulb Pipette Filler**

Unlike most other types of fillers, the simple bulb pipette filler does not attach to the volumetric pipette, but rather is held snugly to the end of the pipette while drawing up the solution. It is made of durable, chemically resistant rubber and can be cleaned with water and/or acetone.

---

**Figure A1.4**  
Simple bulb pipette filler used in this course

**Steps in Operation of the Simple Bulb Pipette Filler**
Note: If you are right-handed, hold the bulb in your left hand and the volumetric pipette in your right hand. If you are left handed, do the opposite.

1. Squeeze the bulb, thereby forcing the air from the pipette filler.

2. Place the squeezed bulb snugly over the top end of the volumetric pipette.

3. Submerge the bottom tip of the volumetric pipette beneath the surface of the liquid to be drawn into the pipette, all the while keeping the pipette filler bulb squeezed.

4. Slowly begin to draw up the liquid by lessening your pressure on the bulb, thereby allowing the vacuum inside the bulb to draw up the liquid. Be careful to keep the bottom tip of the volumetric pipette beneath the surface of the liquid to avoid entraining air bubbles into the pipette.

5. Once the liquid has been drawn up past the specified mark on the volumetric pipette, very quickly remove the bulb, and replace it with your forefinger of your opposite hand (the one holding the pipette).

6. Allow the liquid to slowly descend to the mark so that the line on the volumetric pipette is at the bottom of the meniscus of the liquid in the pipette (see Figure A1.5). Stop the liquid from descending further by pressing your forefinger snugly on the top of the pipette. It will take some practice to become skillful in controlling the liquid level in the pipette with your finger, i.e., finding out just how much finger pressure is necessary to stop the liquid or allow the liquid to descend.

7. Touch off any drop hanging from the pipette tip and transfer the liquid to the desired vessel. Lift your finger from the top of the pipette and allow the liquid to drain from the pipette.

Figure A1.5
When using a pipette to dispense a known volume of liquid, the eye should be level with the bottom of the meniscus (to limit parallax error) when sighting the meniscus on the graduation mark.
8. There will still be a small amount of liquid left in the pipette. **Do not attempt to blow this liquid out**—it should be left in the pipette. When the manufacturer calibrates the pipette, the calibration is carried out with the knowledge that, when the specified volume (in this case, 5 mL) has run out of the pipette, this small amount of liquid will remain behind. This is why your pipette is marked TD (to deliver) 5 mL. It delivers this volume, but when it is filled to the graduation mark it actually contains a little bit more.
Results, Calculations and Write-Up

Measurement of Mass and Volume

As this is a very simple exercise, an elaborate write-up is unnecessary. We simply expect you to report all your measurements, do the calculations, and answer the questions.

A. Weighing by Difference and the Mole Concept

Results

_______ = mass of vial + carbon
_______ = mass of empty vial
_______ = mass of carbon transferred to beaker
_______ = mass of vial + tin
_______ = mass of empty vial
_______ = mass of tin transferred to beaker

Calculations

_______ = amount (i.e., number of moles) of carbon transferred to beaker
_______ = number of carbon atoms transferred to beaker
_______ = amount (i.e., number of moles) of tin transferred to beaker
_______ = number of tin atoms transferred to beaker

From the above results, calculate

1. the average mass of one atom of carbon (in grams)
2. the average mass of one atom of tin (in grams)
3. the ratio, average mass of one atom of tin: average mass of one atom of carbon

In theory, what is the correct value of the above ratio?
B. Density of Water

\[
\begin{align*}
\text{mass of water in cylinder} & = \text{volume of water in cylinder} \\
\text{mass of water + cylinder} & = \text{mass of water + cylinder} \\
\text{mass of empty cylinder} & = \text{mass of empty cylinder} \\
\text{mass of water in cylinder} & = \text{mass of water in cylinder} \\
\text{water temperature} & = \text{water temperature} \\
\text{density of water at } & = \frac{\text{mass of water in cylinder (g)}}{\text{volume of water in cylinder (mL)}} \\
& = \frac{\text{g}}{\text{mL}} \\
& = \text{literature value for the density of water at this temperature}
\end{align*}
\]

Reference:

C. Calibration of a Pipette

You should now have five data sets, each set consisting of (a) the mass of the beaker plus approximately 25 mL of water and (b) the mass of the empty beaker. For each data set, you will determine the actual volume of water measured out using the following procedure.

\[
\begin{align*}
\text{Mass of beaker + water} &= xg \\
\text{Mass of empty beaker} &= yg \\
\text{Mass of water} &= (x - y)g \\
\text{Temperature of water} &= T{}^\circ C \\
\text{Density of water at } T{}^\circ C &= g \cdot mL^{-1} \\
\text{Volume of water actually dispensed} &= \frac{(x - y)g}{g \cdot mL^{-1}} = v mL
\end{align*}
\]

You should then determine the average volume of water dispensed by the pipette:

\[
\text{average volume dispensed by pipette} = \frac{\text{total volume dispensed in 5 trials}}{5}
\]

Calculate the deviation for each of the five individual trials:

\[
\text{deviation (d)} = \text{result from individual trial – average value}
\]
Calculate the average deviation ($a$) using the formula:

$$ a = \frac{\sum |d|}{n} $$

and the standard deviation ($\sigma$) using the formula:

$$ \sigma = \sqrt{\frac{\sum d^2}{n - 1}} $$

**Experiment A1 Questions**

1. When you transferred the carbon and tin from the vial to the beaker, some of the solid remained behind in the vial. Why is this not important?

2. When you measured the density of water, how would your results have been affected if the graduated cylinder had contained a few drops of water instead of being clean and dry?
Experiment A2:
Use of a Spectrophotometer (3 hours)

Prerequisite Skills

You should have completed and submitted the Candle Experiment before handing in any in-lab experimental reports, and performed Experiment A1.

Objectives

When you have completed the following experiment, you will have

1. learned how to dilute a concentrated solution in order to obtain a dilute solution of known concentration.

2. learned how to use a SpectroVis spectrophotometer and determined the amount of ASA in an Aspirin® or generic brand ASA tablet.

Safety Tips

Experiment location. Work on a steady table in a safe secure place. Keep doors and windows closed to prevent drafts.

Handling of chemicals. Acetylsalicylic acid powder is dusting. Avoid inhaling. Safety glasses should be worn at all times. Wear appropriate clothes and closed-toed shoes.

The experiment also illustrates

1. the use of the relationship:
   \[
   \text{concentration (mol} \cdot \text{L}^{-1}) = \frac{\text{amount of solute (mol)}}{\text{volume of solution (L)}}
   \]
   (Jones and Atkins, Chapter 4.6)

2. the use of the relationship:
   \[
   \text{initial concentration} \times \text{initial volume} = \text{final concentration} \times \text{final volume}
   \]
   as it is applied to dilutions.

3. why certain solutions are coloured.

4. the use of graphs in quantitative analysis.
Introduction and Theory

Spectrophotometric Measurement

Visible light is a mixture of electromagnetic radiation of different wavelengths (\(\lambda\)). Different colours are associated with radiation of different wavelengths; thus, light at a wavelength of about 400 nm appears to be violet in colour, light at a wavelength of about 490 nm appears to be blue, and so on.

Certain substances absorb light (radiation) over a range of wavelengths. When this happens, the substance appears to be coloured, although the colour we see is that of the transmitted light, not the absorbed light. For example, potassium permanganate absorbs light in the green-yellow region, and the transmitted light is a mixture of blue and red radiation. Thus, potassium permanganate appears purple to the human eye. Other examples are shown in Table A2.1.

Table A2.1 Colour and wavelengths in the visible region of the electromagnetic spectrum

<table>
<thead>
<tr>
<th>Colour absorbed</th>
<th>Wavelength (nm)*</th>
<th>Colour transmitted</th>
</tr>
</thead>
<tbody>
<tr>
<td>Red</td>
<td>750–610</td>
<td>Blue-green</td>
</tr>
<tr>
<td>Orange</td>
<td>610–595</td>
<td>Blue</td>
</tr>
<tr>
<td>Yellow</td>
<td>595–580</td>
<td>Violet</td>
</tr>
<tr>
<td>Green</td>
<td>580–500</td>
<td>Purple</td>
</tr>
<tr>
<td>Blue</td>
<td>500–435</td>
<td>Orange</td>
</tr>
<tr>
<td>Violet</td>
<td>435–380</td>
<td>Yellow</td>
</tr>
</tbody>
</table>

* Approximate values only. For example, it is impossible to define exactly where the yellow region ends and the orange region begins.

A spectrophotometer is an instrument that allows you to shine light of known wavelength through a solution, and to measure how much light of that wavelength is absorbed by the solution. This measurement, known as the absorbance of the solution, is dependent upon three factors:

1. the concentration, (c), of the molecules in the solution (i.e., the number of molecules absorbing light),
2. the path length of the light, (b), through the solution, and
3. the molecular absorptivity of the compound, (\(\varepsilon\)), which is an intrinsic property of the compound and is dependent upon its electronic structure.

These factors are related to absorbance, (A), by what is called the Beer-Lambert law (sometimes referred to as Beer’s law):

\[ A = \varepsilon \cdot b \cdot c \]
Where $A$, the absorbance, is the logarithm of the ratio of the intensity of the incident light ($I_0$) to the intensity of the transmitted light ($I_t$), i.e., $A = \log_{10} \left( \frac{I_0}{I_t} \right)$.

The photodetector on your simple spectrophotometer measures intensity of the transmitted light and expresses this as a voltage reading. Because the voltage reading is directly proportional to the intensity of the light received by the detector, one can express absorbance as $A = \log_{10} \left( \frac{V_o}{V_t} \right)$, (where $V_o = kI_o$ and $V_t = kI_t$).

The active ingredient in Aspirin® tablets is a chemical called acetylsalicylic acid. The purpose of this experiment is to determine the actual percentage of acetylsalicylic acid in a commercial Aspirin® tablet. The formula of acetylsalicylic acid (sometimes abbreviated to ASA) is $C_9H_8O_4$ and its structure is shown in Figure A2.2. Do not be too concerned if you do not fully understand this structure.

![Figure A2.2]

The structure of acetylsalicylic acid

In general, it is possible to determine the concentration of a coloured species in a given solution by measuring the absorbance of the solution at a certain specific wavelength and comparing the value obtained with a calibration curve obtained at the same wavelength. The calibration curve consists of a plot of absorbance (vertical axis) against concentration (horizontal axis) for a number of solutions of known concentration. Such a plot is often called a Beer’s law plot, and a typical plot is shown in Figure A2.3.

From Figure A2.3, we can see that if a solution of unknown concentration was found to have an absorbance of 0.42, its concentration can be deduced to be $4.2 \times 10^{-4}$ mol $\cdot$ L$^{-1}$. 
When trying to adopt this procedure to determining the concentration of a given solution of acetylsalicylic acid, we run into the problem that such solutions are colourless, i.e., acetylsalicylic acid does not absorb visible light. This problem is overcome by reacting the acetylsalicylic acid with a base, acidifying the solution, and allowing the anion so formed to react with hexaaquairon(III) ion to produce the intensely violet tetraaquasalicylatoiron(III) ion as shown in the (unbalanced) equation below.

\[
\text{C}_9\text{H}_8\text{O}_4 + 2\text{NaOH} \rightarrow \text{C}_7\text{H}_4\text{O}_3^- + \text{Fe(H}_2\text{O)}_6^{3+}
\]

Provided that the solution is kept acidic, the concentration of the tetraaquasalicylatoiron(III) complex will be equal to the concentration of acetylsalicylic acid in the original solution. Thus, this experiment will involve:

1. preparing a series of solutions containing known concentrations of tetraaquasalicylatoiron(III) ions,
2. measuring the absorbance of each of the solutions at a fixed wavelength and constructing a calibration curve, and
3. determining the concentration of the “unknown” solution. The latter will have been prepared by dissolving a single Aspirin® tablet of known mass in excess sodium hydroxide, diluting with water and adding an acidic solution of iron(III) chloride.

Two important techniques that you will learn in this experiment are (1) dilution and (2) the use of the a spectrophotometer.

**Dilution**

In order to dilute a given solution in a quantitative manner, a known volume of the solution is transferred, via a pipette, to a volumetric flask (see Figure A2.4). Additional solvent (usually water) is then added to the flask until the level of the solution reaches the graduation mark on the neck of the flask. The flask is then stoppered (some flasks have plastic caps rather than stoppers) and the contents thoroughly mixed.

![Figure A2.4](image)

A volumetric flask

**The SpectroVis Spectrophotometer**

You do not need to know how a spectrophotometer works. All you need to know is that the SpectroVis spectrophotometer used in this experiment (see Figure A2.5) is a delicate and intricate unit that allows the operator to determine the absorbance of a given solution at a prescribed wavelength in the visible region of the spectrum.
Transition metals such as iron and chromium have electronic transitions that absorb at different wavelengths of light. A plot of absorbance (y axis) versus wavelength (x axis) is called an absorption spectrum. A typical absorption spectrum is shown in Figure A2.6.

Note that in the above spectrum, the absorbance reaches a maximum at 302 nm, 410 nm and 576 nm. The wavelength at which the absorbance is at a maximum is known as $\lambda_{\text{max}}$.

The spectrophotometer in your kit will need to be set the correct wavelength to record light fluctuations at one of the stronger absorption bands of the tetraaquasalicylatoiron(III) ion.

Your objective in this part of the experiment is to determine the absorbance of a number of solutions containing known concentrations of the
tetraaquasalicylatoiron(III) ion. A plot of absorbance, or in this case voltage (y axis in mV), against wavelength (x axis, nm at $\lambda_{\text{max}}$) should yield a straight line, as described below in Figure A2.7. This calibration curve can then be used to determine the concentration of tetraaquasalicylatoiron(III) ion present in the solution prepared from the commercial Aspirin® tablet.

Figure A2.7
The standard curve for tetraaquasalicylatoiron(III) ion plotted in LoggerPro

Please note that the range of absorbance readings you may see for the tetraaquasalicylatoiron(III) ion standard solutions will be different from the above as each spectrophotometer is to be considered unique, even though each and every SpectroVis Plus has be factory calibrated.
Chemical and Material Requirements

General pan balance (calibrated)
SpectroVis spectrophotometer and USB cable and plastic square cuvettes
Logger Pro Software (free download) and computer with USB port

1000 mL plastic beaker (for 0.02 M FeCl₃)
iron (III) chloride in vial (in excess for ~400 mL acidified 0.02 M FeCl₃)—you will have to weigh the amount indicated in the procedure.
spatula — optional
glass rod
1M HCl in sealed solution dropper

preweighed ASA standard in vial (0.30–0.35 g—must check weight on balance provided)
50 mL beaker
1M NaOH in sealed solution dropper (2 × 2 mL)
tap water
hot plate/stove top
100mL volumetric flask
empty solution droppers (2)

Dilution Series 1
1 × 5 mL volumetric pipette
1 × 50 mL volumetric flask
1 × 25 mL volumetric flask
1 × 10 mL volumetric flask

Dilution Series 2
1 × 1 mL volumetric pipette
1 × 50 mL volumetric flask
1 × 25 mL volumetric flask
1 × 10 mL volumetric flask

Unknown Tablet Dilution
1 × 1 mL volumetric pipette
1 × 100mL volumetric flask
1 × 50 mL volumetric flask

pipette filler bulb

1 Aspirin® or generic brand ASA tablet (per student)
Unknown tablet in vial

Note: tetraaquasalicylatoiron (III) ion can be discarded with dilution down the drain.
### List of Reagents

<table>
<thead>
<tr>
<th>Compound</th>
<th>Name</th>
<th>Molecular Weight</th>
<th>Solubility</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaOH (1M)</td>
<td>sodium hydroxide 1M solution</td>
<td>40.00 g/mol</td>
<td>Sol. cold water (0°C) = 42/100mL, hot water = 347 g/100mL</td>
</tr>
<tr>
<td>ASA</td>
<td>acetylsalicylic acid (or salicylic acid, acetate)</td>
<td>180.17 g/mol</td>
<td>MP 135°C, Sol. (al, eth, chl)</td>
</tr>
<tr>
<td>FeCl₃ · 6H₂O*</td>
<td>ferric (III) chloride hexahydrate</td>
<td>270.3 g/mol</td>
<td>Sol. cold water (20°C) = 91.9 g/100mL, hot water = ∞/100mL</td>
</tr>
<tr>
<td>Aspirin® Tablets</td>
<td>commercial, unbuffered, uncoated Aspirin® or generic brand tablets</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* indicates solution must be made fresh on each lab day
**Procedure (~ 3 hours to complete) - wear safety glasses throughout**

**A. Preparation of 0.02 M·L⁻¹ Iron (III) Chloride Stock Solution**

1. Weigh the provided iron(III) chloride hexahydrate containing vial on the general pan balance.

2. With the aid of a metal spatula, transfer 2.16 g of FeCl₃·6H₂O from the vial to a 1000 mL beaker, and weigh the stock vial (it will still have some ‘left over’ again reagent inside). Use “weighing by difference” to determine the actual amount of iron(III) chloride transferred to the beaker.

3. Add 400 mL of water to the iron(III) chloride hexahydrate in the beaker, and stir with a glass rod to assist in dissolving the solid. When all the iron(III) chloride hexahydrate has dissolved, add ~1 mL of the provided 1 M HCl to the iron(III) chloride solution and mix.

4. The 0.02 mol·L⁻¹ iron(III) chloride is now ready for use in preparing your dilutions (see c. Dilution of the ASA stock solution). In subsequent parts of this experiment we shall refer to the solution as the stock solution.

**B. Preparation of the ASA Stock Solution**

1. Empty the preweighed acetylsalicylic acid vial (between 0.3000 and 0.3500 g) into a clean 50-mL Erlenmeyer flask. Label the flask and record the precise weight indicated on the vial in your lab notebook.

2. Cut open the disposable pipette of 1 M NaOH. Carefully and repeated rinse the residual ASA from the vial with small amounts of NaOH solution and pour the rinses into the 50-mL Erlenmeyer flask. When all the NaOH solution (2 mL) is depleted, continue rinsing the vial with small amounts of water (about another 5–10 mL total) and pour into the 50-mL Erlenmeyer flask. This should ensure that most of the ASA from the vial is now in your 50-mL Erlenmeyer flask.

3. Place the 50-mL flask and heat to boiling on a hot plate. **[Note: Hot sodium hydroxide is extremely corrosive; wear gloves and protect your eyes.]** You can use the element on an electric stove top. If you have a gas stove, do not use the open flame directly. Place a pan or shallow pot on the flame and then use that as a hot plate. Do not heat too strongly as this may cause splattering and subsequent loss of solution. When all the acetylsalicylic acid has dissolved, the inside walls of the flask should be rinsed down with a small quantity of water and the solution allowed to cool. While the solution is cooling, you can proceed with Part f, steps 1 and 2.
4. Transfer the contents of the Erlenmeyer flask to a 100-mL volumetric flask. Wash the Erlenmeyer flask several times with water, and add these washings to the volumetric flask. A funnel can be used to assist in this transfer. Finally, add water to the volumetric flask until the level of the solution reaches the graduation mark on the neck of the flask. Mix well. You have now prepared a standard solution of sodium salicylate, i.e., a solution of sodium salicylate whose concentration is known very precisely. In subsequent parts of this experiment we shall refer to the solution as the ASA stock solution.

C. Dilution of the ASA Stock Solution

You will be completing several dilutions to come up with a total of six samples (labelled A–F). The two series of dilutions are summarized visually in Figure A2.8.
Dilution Series 1

1. Use a clean, dry volumetric pipette and pipette bulb to transfer a 5 mL aliquot (fractional portion) of the ASA stock solution to a 50 mL volumetric flask. Add the 0.02 mol · L⁻¹ iron(III) chloride solution until the level of the solution in the flask reaches the graduation mark on the neck of the flask. Insert the stopper and shake thoroughly. Label this “Sample A” using masking tape.

2. Rinse your 5 mL pipette with water. Dry the outside of the pipette and try to blow out as much of the water from inside as possible using your pipette bulb.

3. Take up 1–2 mL of Sample A into the 5 mL pipette, and allow the solution to rinse the glass surface by rotating the pipette at an angle. Discard the contents.

4. Repeat step 3 using another 1–2 mL of Sample A. This will now ensure that aliquots of Sample A delivered by the 5 mL pipette will not be diluted with residual water.

5. Transfer a 5 mL aliquot of Sample A to a 25-mL volumetric flask. Add the 0.02 mol · L⁻¹ iron(III) chloride solution until the level of the solution in the flask reaches the graduation mark on the neck of the flask. Insert the stopper and shake thoroughly. Pour into a clean container and label this “Sample B.”

6. Repeat step 3 above, and then rinse the pipette with Sample B in the same manner as steps 3 and 4.

7. Transfer a 5 mL aliquot of Sample B to a 10 mL volumetric flask. Add the 0.02 mol · L⁻¹ iron(III) chloride solution until the level of the solution in the flask reaches the graduation mark on the neck of the flask using a solution dropper to accurately add the iron(III) chloride. Insert the stopper and shake thoroughly. Pour into a clean container, and label this “Sample C.”

Dilution Series 2

1. Use a volumetric pipette to transfer 3 mL (3 × 1 mL) of the original ASA stock solution (100%) to a clean 50 mL volumetric flask. Top up to the 50 mL mark with iron(III) chloride solution, mix and label as “Sample D.”

2. Use the same volumetric pipette to now transfer 1 mL of the original ASA stock solution (100%) to a clean 25 mL volumetric flask. Top up to the 25 mL mark with iron(III) chloride solution, mix and label as “Sample E.”
3. Wash with water, dry and rinse the 1 mL volumetric pipette with Sample E.

4. Transfer a 1 mL aliquot of Sample E to a 10 mL volumetric flask. Add the 0.02 mol \( \cdot \) L\(^{-1} \) iron(III) chloride solution until the level of the solution in the flask reaches the graduation mark on the neck of the flask. Insert the stopper and shake thoroughly. Label this “Sample F.”

You should now have six “dilute” solutions containing known concentrations of tetraaquasalicylatoiron(III) ion in addition to the stock solution. The concentrations of these dilute solutions are approximately (assumes stock solution made with 0.32 g ASA (divided by 180.17 g/mol ASA, and 0.100 L solution prepared) = \( 1.776 \times 10^{-2} \) mol \( \cdot \) L\(^{-1} \)).

Sample A (5 mL stock diluted to 50 mL) = \( 1.78 \times 10^{-3} \) mol \( \cdot \) L\(^{-1} \)
Sample B (5 mL A diluted to 25 mL) = \( 3.56 \times 10^{-4} \) mol \( \cdot \) L\(^{-1} \)
Sample C (5 mL B diluted to 10 mL) = \( 1.78 \times 10^{-4} \) mol \( \cdot \) L\(^{-1} \)
Sample D (3 mL stock diluted to 50 mL) = \( 1.07 \times 10^{-3} \) mol \( \cdot \) L\(^{-1} \)
Sample E (1 mL stock diluted to 25 mL) = \( 7.12 \times 10^{-4} \) mol \( \cdot \) L\(^{-1} \)
Sample F (1 mL E diluted to 10 mL) = \( 7.12 \times 10^{-5} \) mol \( \cdot \) L\(^{-1} \)

The precise concentrations of all your solutions must be calculated now, before you try to use your spectrophotometer. The Vernier LoggerPro software will prompt you to enter the [solution] immediately after making your reading, and then will plot your data pair (conc., abs.).

D. Using Your SpectroVis Plus Spectrophotometer. Installing your software and Calibrating your SpectroVis Plus.

Before starting Part D, it is advised you also do Part G and weigh, dissolve (with NaOH and H\(_2\)O) and dilute (with FeCl\(_3\)) your unknown ASA tablet. Thus you would have seven “dilute” solutions ready to read in the spectrophotometer. It is recommended you do all seven readings in as short a time frame as possible, to avoid any potential electronic drift in the spectrophotometer.

1. Find the following items in your all-home lab kit: SpectroVis Plus spectrophotometer, USB cable and 2 cuvettes.

Figure A2.9
Use of your spectrophotometer (see pp.91-97 for all steps)
2. Start the Logger Pro 3 (version 3.6 or newer) program on your computer. If you need to download the LoggerPro software (AU has a Site Licence), please perform the following steps:

   i) Turn on your computer, connect to the internet, open your web browser, and Go to your course Moodle web page:

   http://science.lms.athabascau.ca/file.php/24/VernierInstructions.txt

   Logger Pro System Requirements
   Windows® XP SP2/SP3, Vista, Vista 64, Windows 7, or Windows 7 64.
   Pentium® processor or equivalent running at 500 MHz or faster
   512 MB total minimum RAM
   200 MB of hard disk space for a minimum installation
   Available USB or serial port. Only LabPro uses a serial port; all other devices require USB.
   Mac:
   Macintosh OS® OS X 10.4.11, or 10.5.1 or newer
   512 MB RAM
   200 MB of hard disk space for a minimum installation
   Available USB port.

   ii. Download the program and then run the Logger Pro.exe file to install the program. Follow the Installation Wizard instructions. You do not have to go online to find and install the device drivers. The device drivers were installed when you installed the program.

3. Connect your SpectroVis Plus to your computer’s powered USB port or powered hub using the provided USB cable. Make sure to ‘unstatic’ your hand before plugging the cable into the port to avoid any static discharge/sparking.

4. When the Logger Pro v.3.6+ program opens, you will see the following menu. The default selection for the unit is to read absorbance.
5. Before you can use your spectrophotometer, it must be calibrated using the following steps:

   i. Take 2 of the provided square cuvettes that fit your SpectroVis Plus spectrophotometer and draw a dot on one of the opaque sides of it, about 1 cm down from the opening. From now on, every time you place the cuvette into the instrument make sure that the dot is consistently faces the same direction. This will ensure that any distortions in the plastic cuvette will not unduly affect your results (any error will be consistent).

   ii. Fill one of the cuvettes 3/4 full (beneath the dot) with your iron(III) chloride stock solution. This is your blank solution. Place the cuvette it in the instrument so the the light path shines through the two clear window sides of the cuvette (see Figure A2.11).
iii. As shown in Figure A2.12, under Experiment on the main menu, select Calibrate and then select Spectrometer.
A calibration dialog box will display the message: ‘Waiting .seconds for lamp to warm up. The minimum warmup time is 90 seconds.’

Do not press the ‘Skip Warmup’ button. You must allow the lamp to prewarm. When prompted select Finish calibration and then finally press ‘OK’. Your SpectroVis is now ready for reading your samples.

6. Remove the ‘blank’ cuvette (containing your 0.02M stock of FeCl₃) from the SpectroVis Sample Compartment and proceed to Part E. When you remove your “blank” cuvette, do not discard it yet, in case of having to repeat the experiment.

E. Determination of $\lambda$ maximum for the Fe-ASA Complex.

1. From Part D, the SpectroVis Plus lamp is already pre-warmed and calibrated. Select Configure Spectrophotometer from the toolbar as shown in Figure A2.13.

![Figure A2.13](image)

Selecting Configure Spectrophotometer

2. Place a cuvette containing the solution which contains about 4.4 x 10^-4 mol x L⁻¹ of tetraaquasalicylatoiron(III) ion; e.g., Sample A solution prepared in Part C.

NOTE: The absorbance scale is logarithmic, whereas the % transmittance scale is linear. The two measurements can be interconverted using the following relationship:

$$\text{absorbance} = 2 \cdot \log(\% \text{ transmittance})$$

3. Click “OK” and then select “Collect” from the toolbar to generate a spectrum. Click “Stop” to end data collection.
4. Right click on your Logger Pro Graph window and select Graph Options. Name your graph.

5. Print your Data Table and Graph (see under Logger Pro “File” menu) and include both in your lab report. From your graph, you should realize that the tetraaquasalicylatoiron(III) ion has a $\lambda_{\text{max}}$ between 500 and 550 nm. Determine the and record the precise $\lambda_{\text{max}}$ for tetraaquasalicylatoiron(III) and then use this wavelength for determination of the absorbance of solutions in Part F.

6. Save your *.cmbl file using the Save As selection under the File menu, and name it as your “ExpA2 Wavelength Spectrum of Fe-ASA Complex.”

F. Construction of a Calibration Curve

1. Determine the absorbance readings for each of your blank and the six dilute solutions. Make sure that the cuvette is cleaned (use water) between each reading. To remove the drops of water that remain in the cuvette, rinse the cuvette thoroughly with the solution that you are about to test. Wipe the two clear plastic sides of the cuvette clean with paper towel before you insert it into the instrument, to remove fingerprints, etc., which could affect your results. Be careful not to erase your dot!

2. Obtain a second cuvette and rinse it twice with small amounts of the 0.020 M FeCl₃ solution. Fill the cuvette ¾ full with the 0.020 M FeCl₃ solution and place it in the spectrometer.

3. Click on the ‘Configure Spectrometer’ Data Collection icon on the toolbar as shown in Figure A2.14.

Figure A2.14
Selecting the ‘Configure Spectrophotometer’ icon from the toolbar.
A dialog box will appear as shown in Fig. A2.15.

**Figure A2.15**
Setting up the LoggerPro software for Absorbance vs Concentration readings

First select Abs vs. Concentration under Set Collection Mode. Next set the wavelength to 525 nm by scrolling to and then checking the box next to the desired wavelength. Then from the dropbox, select ‘Individual Wavelengths’. Finally click ‘OK’ to proceed.

You are ready to make your first reading (you can see your absorbance reading in the bottom left hand corner of the window). Press ‘Collect’ to read the 0.02 M FeCl₃ solution.

**Figure A2.16**
Making a Reading by pressing the ‘Collect’ button
4. Press ‘Keep’ to save the reading and fill in the [ASA] prompt box by typing 0.00 Mol/L.

Figure A2.17
To keep the reading press ‘Keep’

5. Continue to collect absorbance-concentration data for the remaining six standard solutions by repeating steps 6-8. Now that you have readings it is important to save your graph. At your convenience, select ‘Save As’ from the ‘File’ menu, and store the file on your drive.

6. Discard the cuvette contents as directed. Use the next solution to double rinse and then fill the cuvette ¾ full. Wipe the cuvette and place it in the spectrometer. When the absorbance reading stabilizes, record it in your notebook and then click ‘Keep’. Enter the appropriate concentration for each solution as you have calculated earlier (see page 91).

7. To see your graph grow as you continue to read your standards, select ‘Append to Latest’ between each reading when prompted as in Figure A2.18.
Figure A2.18
Appending readings to build a graph as you make your readings

Figure A2.19 below shows what a graph might look like after making 4 or the 7 readings for this calibration curve.

Figure A2.19
Data Collection in progress. Four solutions now read.

8. When you have read all the standard ASA stock solutions, and wish to stop the data collection –Press Stop as shown in Figure A2.20.
In LoggerPro, the ASA-FeCl₃ standard solutions graph linear fit can be determined using the ‘Linear Fit’ selection under ‘Analyze’ in the main menu, or by using the ‘linear fit button’ on the toolbar as shown in Figure A2.21. It is advisable that you also write down the equation for the standard solutions in your data table or lab book.
G. Determination of the Mass of Acetylsalicylic Acid in an Aspirin® Tablet

1. Use the balance to determine the mass of an empty 50 mL Erlenmeyer flask. Place an Aspirin® tablet in the flask and determine the mass of the flask plus the unbuffered/uncoated Aspirin® tablet.

2. Boil the ASA tablet with one disposable pipette full (~2 mL) of 1 M sodium hydroxide solution and 10 mL of water as in step 3 of Part A. Remember that the Aspirin® tablet may contain insoluble starch or filler, so do not be concerned if the solution appears somewhat milky. Allow the solution to cool while you return to the preparation of the stock solution.

3. Dilute the solution to 100 mL as in step 3 of Part B, i.e., transfer the contents of the Erlenmeyer flask to a 100 mL volumetric flask. Wash the Erlenmeyer flask several times with water and add these washings to the volumetric flask. Finally, add water to the volumetric flask until the level of the solution reaches the graduation mark on the neck of the flask. You have now prepared an unknown solution of sodium salicylate.

4. Transfer 1.00 mL of the dilute solution to a 50 mL volumetric flask. Add the iron(III) chloride solution until the level of the solution in the flask reaches the graduation mark. Mix well by inversion.

   [You will determine the absorbance of this solution using your SpectroVis Plus, after you have made all your 7 readings for the standard curve.]

5. To determine the concentration of the unknown ASA solution, first rinse the cuvette twice with the unknown solution, and fill it about ¾ full. Remember to wipe the outside of the cuvette.

6. Please the cuvette containing your unknown solution in the SpectroVis Plus. After reading the solution dispose of any of the remaining solutions as directed below under ‘Waste Disposal’.

7. In LoggerPro, select Interpolation Calculator from the Analyze menu. A dialog box will appear that displays the concentration of your unknown at the measured absorbance. Click ‘OK’ and also record the absorbance and [unknown ASA] in your data table or lab book.

8. (optional) Print a copy of your graphs and/or data table.

9. (optional) To save you data file, go to the LoggerPro main menu under ‘File’ and select ‘Save As’ and save your experiment file to your disk. Exit the Logger Pro software to turn off your SpectroVis Plus.

   WASTE DISPOSAL: All solutions prepared in this experiment may be washed down the drain with plenty of water.
Results, Calculations and Write-Up

This report will require more detail than Experiment A1, and the entire report will take the format of a formal report. For your results section, please use the suggested format outlined below.

A. Mass of FeCl₃ Used to Prepare 400 mL of Solution

1. Mass of iron(III) chloride used to prepare 400 mL of stock solution = __________.
2. Amount (i.e., number of moles) of iron(III) chloride used to prepare 400 mL of stock solution = __________.
3. Concentration of iron(III) chloride stock solution = __________.

B. Preparation of the ASA Stock Solution

1. Mass of acetylsalicylic acid used to prepare 100 mL of stock solution = __________.
2. Amount (i.e., number of moles) of acetylsalicylic acid used to prepare 100 mL of stock solution = __________.
3. Concentration of acetylsalicylic acid in stock solution = __________.

C. Dilution of an ASA Stock Solution

By the relationship: initial concentration \times initial volume = final concentration \times final volume, the concentrations of the six dilute solutions were:

1. Sample A ASA Concentration =
2. Sample B ASA Concentration
3. Sample C ASA Concentration
4. Sample D ASA Concentration
5. Sample E ASA Concentration
6. Sample F ASA Concentration
D. Using Your Spectrophotometer

SpectroVis Plus Settings

Mode (%T, Kinetic, Abs, etc.?) _________________________
Wavelength (nm) _________________________
10nm band or Individual Wavelength _________________________

E. Determination of $\lambda_{\text{max}}$

1. The data table showing the absorbance of your approximately $4.4 \times 10^{-4}$ mol $\cdot$ L$^{-1}$ solution at all the wavelengths (nm) will be many pages long. Do not print it all off. Instead just show the raw data for the part of the scan that shows where the $\lambda_{\text{max}}$ occurs plus and minus 15 nm.

2. Plot a graph of absorbance (y axis) vs. wavelength (x axis). Identify the wavelength at which the absorbance is at a maximum.

F. Construction of a Calibration Curve

1. Prepare a table that shows the absorbance for each of your six dilute solutions and the blank (0.02 M FeCl$_3$).

2. Plot a graph of Absorbance (y axis) vs. concentration (x axis).

G. Determination of the Concentration of the Solution Prepared from Commercial Aspirin® or generic brand

1. The absorbance of diluted solution prepared from ASA tablet was ______ (absorbance units).

2. From the graph plotted in F, such an absorbance corresponds to a concentration of ____________ M ASA.

H. Determination of the Percentage of Acetylsalicylic Acid in an Aspirin® or generic brand Tablet

1. From G, the concentration of tetraaquasalicylatoiron(III) in the dilute solution was ____________.

2. The concentration of the dianion of salicylic acid in the 100 mL sodium hydroxide stock solution was ____________.

3. The number of moles of the dianion of salicylic acid in the 100mL sodium hydroxide stock solution was ____________.

4. The number of moles of acetylsalicylic acid initially present and therefore also in the Aspirin® tablet was ____________.
5. The mass of acetylsalicylic acid in the Aspirin® tablet was __________.

6. The percentage of acetylsalicylic acid in the Aspirin® tablet was _____.

WARNING: As with all the experiments in this course, your grade for this experiment will depend primarily on the accuracy and precision of your results. Do not proceed to the next experiment until you are satisfied that you have completed this experiment to the best of your ability.
Experiment A2 Questions

1. The absorption spectrum of a solution that contains chromium(III) ions shows two maxima, one at about 400 nm and the other at about 575 nm.
   a. What colour light is associated with electromagnetic radiation of these wavelengths?
   b. How does this account for the colour of the chromium(III) solution? (Hint: See pp. 80, 84 of this lab manual.)

2. Acetylacetone (acac) forms a trisacetylacetonateiron(III) complex (i.e., Fe(acac)₃) in aqueous solution. A 5 mL aliquot of acetylacetone stock solution is diluted to 200 mL with an iron(III) chloride solution. Spectrophotometry indicates that its iron(III) concentration is $4.23 \times 10^{-4}$ M. What is the concentration of acetylacetone in the stock solution?
Experiment A3: Acid-Base Titrations

Prerequisite Skills

Before attempting this experiment you must have completed Experiment A2, and be familiar with the correct use of a pipette and burette.

Objectives

When you have completed the following experiment, you will have become familiar with the techniques used in acid-base titrations.

This experiment also provides you with an opportunity to

1. apply the mole concept and stoichiometry to titration data.
2. write molecular and net ionic equations for acid-base reactions.
3. perform calculations associated with dilutions.
4. observe a practical example of the concept of neutralization.
5. learn how to operate a burette.

Safety Tips

Experiment location. Work on a steady table in a safe secure place. Keep doors and windows closed to prevent drafts.

Handling of chemicals. Sodium hydroxide and hydrochloric acid are corrosive. Avoid skin contact.

Safety glasses should be worn at all times. Wear appropriate clothes and closed-toed shoes.
**Introduction and Theory**

In Experiment A2 you used a spectrophotometric technique to determine the percentage of acetylsalicylic acid present in a commercial Aspirin® tablet. In this experiment you will use another technique, volumetric analysis, to perform the same analysis. At the end of the experiment you will compare the results obtained from the two techniques and be asked to decide which method gives the most reliable results.

When an acid reacts completely with a base, the acidic and basic properties of the solutions are destroyed (neutralized). During a neutralization reaction, hydrated hydrogen ions (H\(^+\)\(_{(aq)}\)) react with hydrated hydroxide ions (OH\(^-\)\(_{(aq)}\)) to form water.

\[ \text{H}^+\(_{(aq)}\) + \text{OH}^-\(_{(aq)}\) \rightarrow \text{H}_2\text{O} + \text{heat} \]

Thus, one mole of hydrochloric acid HCl\(_{(aq)}\), will neutralize one mole of sodium hydroxide, NaOH\(_{(aq)}\); but one mole of sulfuric acid, H\(_2\text{SO}_4\)\(_{(aq)}\), will neutralize two moles of sodium hydroxide.

\[ \text{HCl} \(_{(aq)}\) + \text{NaOH} \(_{(aq)}\) \rightarrow \text{NaCl} \(_{(aq)}\) + \text{H}_2\text{O} \(_{(l)}\) \]
\[ \text{H}_2\text{SO}_4 \(_{(aq)}\) + 2\text{NaOH} \(_{(aq)}\) \rightarrow \text{Na}_2\text{SO}_4 \(_{(aq)}\) + 2\text{H}_2\text{O} \(_{(l)}\) \]

In most titrations, you would use a solution of known concentration of one substance (a standard solution) to determine the concentration of some other substance in a second solution. For example, you could determine what volume of hydrochloric acid of known concentration is required to neutralize a known volume of sodium hydroxide of unknown concentration. From the results obtained you could calculate the concentration of the sodium hydroxide solution.

An acid-base titration is the process whereby you determine the volume of standard solution required to neutralize a solution of acid or base of unknown concentration. In these titrations, an indicator, such as phenolphthalein or cresol red, is used to determine the end-point of the titration. The end-point is the point at which equal concentrations of hydrogen ions and hydroxide ions are present in the solution. The procedure for an acid-base titration is explained below.

A known volume of solution (acid or base) is measured out into an Erlenmeyer flask or vial by means of a pipette, and a small amount of a dye solution, called an indicator, is added. Indicators have different colours in acidic and basic solutions. Most indicators are extremely sensitive and produce a sharp change in colour when the solution changes from acidic to basic. You are probably already familiar with the use of litmus to identify acids and bases. However, in a titration an indicator is needed that gives a much more dramatic colour changes than litmus. Phenolphthalein is often
used as an indicator in acid-base titrations. It is colourless in the presence of excess acid, and pink in the presence of excess base.

The second solution (acid if you have base in the Erlenmeyer flask, base if you have acid) is poured into the burette, and the initial volume is recorded. Small amounts of this solution are then allowed to run out of the burette into the Erlenmeyer flask or vial, until the indicator changes colour. The change occurs when equal numbers of hydrogen ions and hydroxide ions are present in the Erlenmeyer flask or vial. (This is not quite true. There will be a slight excess of one or other of these ions, but the excess is so small that you need not worry about it.) The volume of solution remaining in the burette can be determined, and you can then calculate the volume that was added to the solution already present in the Erlenmeyer flask or vial.

Once you know the volumes of acid and base used in the reaction, and the concentration of either the acid or the base, the concentration of the other can be calculated. The following example shows how such a calculation is carried out. It also shows you an acceptable method for presenting your results.

**Sample Calculation**

*Titration of a sodium hydroxide solution with standard hydrochloric acid*

**Note:** The term “standard hydrochloric acid” means that the concentration of the hydrochloric acid solution was accurately known. This will be the case in Part A of the present experiment.

Concentration of hydrochloric acid: 0.150 mol \( \cdot \) L\(^{-1} \)

<table>
<thead>
<tr>
<th>Burette readings (mL)</th>
<th>Trial 1</th>
<th>Trial 2</th>
<th>Trial 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final</td>
<td>9.28</td>
<td>17.43</td>
<td>24.75</td>
</tr>
<tr>
<td>Initial</td>
<td>1.77</td>
<td>10.10</td>
<td>17.42</td>
</tr>
<tr>
<td>Volume used (mL)</td>
<td>7.51</td>
<td>7.33</td>
<td>7.33</td>
</tr>
</tbody>
</table>

Average from Trials 2 and 3 = 7.33 mL

Amount (number of moles) of hydrochloric acid present in Erlenmeyer flask

\[ = 0.0100 \text{ L} \times 0.150 \text{ mol} \cdot \text{L}^{-1} \]

\[ = 1.50 \times 10^{-3} \text{ mol HCl} \]

Balanced equation:

\[ \text{HCl (aq)} + \text{NaOH (aq)} \rightarrow \text{NaCl (aq)} + \text{H}_2\text{O (l)} \]
From the equation you can see that the number of moles of sodium hydroxide that reacted is equal to the number of moles of hydrochloric acid consumed:

\[
\text{Number of moles } \text{NaOH reacted} = \frac{1.50 \times 10^{-3} \text{ mol HCl} \times 1 \text{ mol NaOH}}{1 \text{ mol HCl}} = 1.50 \times 10^{-3} \text{ mol NaOH}
\]

This amount of sodium hydroxide must have been added from the burette, and was therefore present in 7.33 mL \( (7.33 \times 10^{-3} \text{ L}) \) of solution. Thus,

\[
\text{concentration of NaOH solution} = \frac{1.50 \times 10^{-3} \text{ mol}}{7.33 \times 10^{-3} \text{ L}} = 0.205 \text{ mol} \cdot \text{L}^{-1}
\]

(or, if you prefer, 0.205 M).

In the preceding example, you will have noticed the results of the first trial were discarded. This is normal practice when performing a titration. Most chemists use the first trial just to obtain a ball park figure for the volume of solution that is required. Normally, the tendency is to overshoot on this first trial, i.e., to add rather more solution than is absolutely necessary. This is because it is often difficult to ascertain exactly when the end-point is approaching, and one tends to be adding solution at such a rate that it is difficult to stop the addition at precisely the right moment.

Occasionally you may find that one (or more) of the titres that you obtain is unexpectedly high or low. How do you decide whether such a result should be included when you calculate the average titre, or discarded? There are at least three ways of approaching this problem, all of which have certain limitations.

1. The 2.5a rule. This rule tells you to reject a result if its deviation from the mean (calculated without the doubtful value) is greater than 2.5 times the average deviation \( a \).

For example, suppose you have four titres: 21.45, 21.47, 21.86, and 21.49 mL. Your instinct would be to assume that there is something wrong with the third result (21.86 mL) and to exclude it from your calculation of the average titre. If you do just that:

\[
\text{Average titre} = \frac{21.45 + 21.47 + 21.49}{3} = 21.47 \text{ mL}
\]

The average deviation, \( a \), of these three results is

\[
a = \frac{\sum |d|}{n} = \frac{0.02 + 0.00 + 0.02}{3} = \frac{0.04}{3} = 0.013 \text{ mL}
\]
The deviation of the “suspect” result is $21.86 - 21.47 = 0.39 \text{ mL}$, which is clearly more than 2.5 times the average deviation, thus the result should be discarded.

2. The 4a rule. This rule is similar to the 2.5a rule described above, except that a result is only discarded if its deviation is four times greater than the average deviation.

3. The Q test. This is a test which is more valid statistically than the 2.5a or 4a rules, but which is of limited use when the number of results is small. The Q test is carried out as follows:
   - find the difference between the suspect result and the nearest result to it. In the above example this would be $21.86 - 21.49 = 0.37 \text{ mL}$.
   - find the range of the results, i.e., the difference between the highest and lowest results, including the questionable result. In the above example this would be $21.86 - 21.45 = 0.41 \text{ mL}$.
   - divide the difference (found in step 1) by the range (found in step 2), to obtain the quotient, $Q$. In the above example,
     \[ Q = \frac{0.37 \text{ mL}}{0.41 \text{ mL}} = 0.90 \]
   - Compare the value of $Q$ to the value of $Q_{0.90}$ in the table below:

<table>
<thead>
<tr>
<th>Number of Results</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
</tr>
</thead>
<tbody>
<tr>
<td>$Q_{0.90}$</td>
<td>0.94</td>
<td>0.76</td>
<td>0.64</td>
<td>0.56</td>
<td>0.51</td>
<td>0.47</td>
<td>0.44</td>
<td>0.41</td>
</tr>
</tbody>
</table>

The value of $Q_{0.90}$ gives the maximum allowable value that $Q$ can have in order for there to be a 90% chance that the result is valid. In our example $Q(0.90) > Q_{0.90}$ for four results (0.76), which means that the result should be rejected.

As you are already aware from the information provided in Experiment A2, the active ingredient in Aspirin® tablets is acetylsalicylic acid.

![Acetylsalicylic acid](image)

**Figure A3.1**
Acetylsalicylic acid

Although it might appear that one should be able to carry out a direct titration of acetylsalicylic acid with a base such as sodium hydroxide, there are a number of practical difficulties associated with doing this. Instead, we shall resort to the use of a technique called “back titration.”
In this process we shall react the acetylsalicylic acid in an Aspirin® tablet with an excess of sodium hydroxide (of known concentration) to produce the sodium salt of salicylic acid and sodium acetate.

\[
\text{acetylsalicylic acid} \quad + \quad 2\text{OH}^- \quad \Delta \quad \text{dianion of salicylic acid} \quad \text{acetate} \quad \text{water}
\]

The excess sodium hydroxide that remains in the reaction mixture is then titrated against a standard solution of hydrochloric acid. Knowing the amount of sodium hydroxide initially added to the reaction mixture, the amount of sodium hydroxide that does not react and knowing the stoichiometry of the reaction of acetylsalicylic acid with sodium hydroxide, we can readily determine the amount of sodium hydroxide that was consumed. From the latter it is a relatively simple matter to determine the mass of acetylsalicylic acid that must have been present in the Aspirin® tablet.
Chemical and Material Requirements

Part A

top-loading general pan balance
100 mL volumetric flask and label
1.2 g Sodium Hydroxide (NaOH) pellets
(excess supplied, student must weigh-out 1.2 g)
5 mL volumetric pipette
~10 mL burette with a Teflon™ stopcock
retort stand + burette clamp
20 mL 0.1000 N HCl
50 mL Erlenmeyer flask
pipette filler
cresol red indicator
meniscus reader (home made)

Part B

100 mL volumetric flask and label
50 mL volumetric flask and label
50 mL Erlenmeyer flask and label
~20 mL 0.1000 N HCl
50 mL Erlenmeyer flask
pipette filler
~10 mL burette with a Teflon™ stopcock
retort stand + burette clamp
10 mL volumetric pipette
4 Aspirin® or generic brand tablets
phenolphthalein indicator
tap H₂O
# List of Reagents

<table>
<thead>
<tr>
<th>Compound</th>
<th>Name</th>
<th>Molecular Weight</th>
<th>Solubility</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaOH (pellets)</td>
<td>sodium hydroxide 0.1M solution</td>
<td>40.00 g/mol</td>
<td>Sol. cold water (0°C) = 42/100mL, hot water = 347 g/100mL</td>
</tr>
<tr>
<td>HCl (0.1000N)</td>
<td>hydrochloric acid</td>
<td>36.5 g/mol</td>
<td>Sol. cold water (20°C) = 91.9 g/100mL, hot water = ∞/100mL</td>
</tr>
<tr>
<td></td>
<td>cresol red indicator (0.04%)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Phenolphthalein indicator (0.05%)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Aspirin® tablets</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Aspirin® tablets: commercial, unbuffered, uncoated
Aspirin® or generic brand tablets.
On the Proper care and Handling of a Burette

1. The short ~10 mL burette is a precision instrument and needs to be handled carefully. The outlet tip of the burette below the stopcock is very fragile and can be easily chipped. When cleaning your burette, always protect this region of the burette.

2. To place and remove the burette from the burette clamp, use your thumb press the middle bar on the clamp to widen the gap and allow you to slip the burette into the clamp.

3. The Teflon™ stopcock fittings should be attached to the burette as shown below: Colored nut outermost, black ‘O’ ring next and white washer innermost/closest to the burette.
Procedure (total time for completion ~ 3 hours)

A. Standardization of a Solution of Sodium Hydroxide

1. Prepare 300 mL of sodium hydroxide solution having a concentration of about 0.1 mol \( \cdot \) L\(^{-1} \) by dissolving the appropriate mass of sodium hydroxide pellets in 300 mL of water. Use a 1000 mL beaker.\(^*\) Label the solution.

   **Hint:** Use the formula \( g = C \times MM \times V \), where \( g \) = mass of substance to weigh (grams), \( C \) = desired concentration in molarity (M), \( MM \) = molar mass of substance being weighed (g/mol), and \( V \) = volume of solution to prepare in liters (L).

   **CAUTION:** Do not permit the sodium hydroxide pellets or solution to come into contact with your body or clothes. Be particularly careful of your eyes. Advise the instructor of any mishap that may occur. Note that heat will be evolved when the sodium hydroxide pellets are dissolved in water.

2. Obtain a 5 mL volumetric pipette. Clean the 5 mL pipette carefully (see Experiment A2).

3. Assemble your titration apparatus with a ~10 mL burette, burette clamp and stand, as shown below in Figure A3.2.

![Figure A3.2 ~10 mL burette with a Teflon™ stopcock, with stand and clamp, plastic burette funnel, and waste beaker](image)

\(^*\) **Note:** because you are going to standardize this solution yourself, the precise measurement of mass and volume are not required. The solution can be prepared in a 1000 mL beaker (instead of a 1000 mL volumetric flask), using a general purpose balance to measure the required mass of sodium hydroxide. Be careful not to waste any of this solution, as it is needed for both Part A and Part B. The solution is not stable over long periods and must be used right away.
If for some reason it is necessary to clean the burette, detach the Teflon™ stopcock and wash the barrel (inside part) of the burette using a dilute soap solution. Rinse with cold water from a kitchen tap. Rinse the whole burette with water, holding the burette horizontally and rotating it so that the entire internal surface is wetted, and then allow the water to drain out. Reattach the stopcock. If the burette is clean, no droplets of water should adhere to the inside. Make sure that the outside of the burette is dry.

During a titration, the normal procedure is to give the burette a final rinse using the solution that is going to be dispensed from it. As you are going to be using sodium hydroxide solution in the burette, rinse the burette with very little of the well-mixed sodium hydroxide solution prepared in step 1. To do this, place the burette into the burette clamp, and add about 2 mL of sodium hydroxide using the plastic funnel (see Figure A3.3). Remove the funnel and take the burette from the clamp. Tip the burette almost horizontal and rotate the burette so that all the internal surface of the glass is wetted with the solution.

4. Return the burette to the burette clamp. Allow the wash sodium hydroxide to drain from the burette into a waste beaker. Add fresh sodium hydroxide using the funnel so that the level of the liquid is near the top of the burette. Again remove the funnel from the burette before proceeding.

5. As you allow the sodium hydroxide solution to escape through the tip of the burette into a waste container, check to make sure there is no air bubble/lock in the tip of the burette and so that the level of the NaOH in the burette descends to just on or physically below a x.00 mL level.

Figure A3.3 Charging your burette with titrant.
To rid yourself of the air-lock/bubble in the stopcock tip, you may have to take the burette out of the clamp and do a gentle shake in a vertical up and down motion to dislodge the air bubble.

**Note:** It is a common misconception that the level of the liquid must always be at 0.00 mL at the beginning of a titration. In fact, it does not matter where the level is, as long as it is read correctly and there is enough solution in the burette to carry out the titration.

6. Record the volume in the burette by reading the bottom of the meniscus and estimating to the nearest 0.01 mL (see Figure A3.4). Use a meniscus reader (homemade, not supplied with kit) if it will help you to locate the exact position of the meniscus.

![Diagram](image)

**Figure A3.4**
Reading the burette

7. Find your container of standard hydrochloric acid 0.1 mol · L⁻¹, but note the precise concentration) and pour out ~20 mL in a clean, dry container. Label it with the concentration of the standard hydrochloric acid. Record the precise concentration in your lab notebook. Try to cover the flask if you do not use it immediately to prevent evaporation.

8. Take your cleaned 5 mL volumetric pipette (from Step 2), attach the rubber bulb pipette filler and draw a small quantity of the standard hydrochloric acid into the pipette. Remove the pipette filler and rinse the pipette/burette as you did in Experiment A1. Now use the 5 mL volumetric pipette to transfer 5.00 mL of the standardized hydrochloric acid to a clean container, e.g., 50 mL Erlenmeyer flask.

9. Add about 1–2 drops of cresol red indicator to the 0.1000N hydrochloric acid in the 50 mL Erlenmeyer flask.

10. Set up the burette and flask/vial as shown in Figure A3.5. The tip of the burette should be about 2 cm below the rim of the vial/flask containing your HCl plus cresol red, as shown in the figure. Allow the base to run slowly from the burette into the acid solution, while swirling the vial/flask frequently.
Figure A3.5
Part Set-up for burette and vial/flask for titration

Note: When you are manipulating the stopcock on the burette, you should adopt the following procedure. If you are right-handed, hold the flask needing swirling in this hand, while controlling the stopcock with your left hand. After each addition into the vial/flask, swirl the vial/flask to mix the base just added from the burette into the acid in the vial. Careful not to damage the burette tip!

As the base runs into the acid you will see the colour of the solution change from red to yellow. As more base is added, you will notice a change from yellow to purple in the area where the two solutions mix. However, with the constant swirling, the bulk of the solution will remain yellow in colour. When the whole solution remains pink rather than yellow you have reached the end-point. (Pink is the mid-colour between yellow and purple.) Then you should read the final volume from your pipette/burette. (Two places past the decimal!) A white paper placed beneath the vial can help you identify the end-point. If the solution assumes a permanent purple colour, you have overshot the end-point and should repeat the titration, this time adding the base more slowly in the region of the end-point. Remember that as you approach the end-point, you can rinse around the neck of the vial with a very small volume of water from a wash bottle.

11. Wash out the vial/flask thoroughly. Replenish your burette with sodium hydroxide, and repeat the titration procedure until you have three results that fall within a 0.10-mL range.

Do not throw out your sodium hydroxide solution as it is required in Part B.
B. Determination of the Percentage of Acetylsalicylic Acid in Aspirin®

The following steps should be carried out in triplicate (three times).

Preparation of the Aspirin® Tablet Hydrolysate

1. Determine the mass of three ASA tablets, one at a time, on your general pan balance. Record the masses of tablets 1, 2 and 3, and then place the first tablet into an empty, clean 125 mL Erlenmeyer flask.

2. Use a 50 mL volumetric flask to measure and transfer 50.00 mL of your sodium hydroxide solution (stored in the 1000 mL beaker) to the 125 mL Erlenmeyer containing the Aspirin® tablet. Heat the flask on a cast iron fry pan on the stove until the solution begins to boil, and allow the solution to boil gently for about five minutes. [Note: Hot sodium hydroxide is extremely corrosive; wear gloves and protect your eyes.] Do not heat too strongly as this may cause splattering and subsequent loss of solution. Remember that the Aspirin® tablet may contain insoluble starch and filler, so do not be concerned if the solution appears milky. Any solid material that becomes deposited on the inside walls of the flask may be rinsed down into the solution using water. Allow the solution to cool while you prepare for your next titration.

Preparation of the Pipette/Burette for Titration

1. In this titration, the burette will be used to dispense standard hydrochloric acid (0.1000 N), thus it is necessary to clean the burette thoroughly before proceeding. Clean the burette as described in steps 3 through 5 in Part A of the experiment, except this time you should use the standard hydrochloric acid whenever the instructions call for sodium hydroxide solution. By the time you have made all the necessary preparations, your solution of Aspirin® in sodium hydroxide should be cool enough to titrate.

Preparation of the Reaction Flask and Titration of the Excess, Unreacted NaOH

1. Use a 10 mL volumetric pipette to transfer 10.00 mL* of your now dissolved and cooled Aspirin® in sodium hydroxide to a 50 mL Erlenmeyer flask. Add a drop of phenolphthalein to the vial containing the solution to be titrated. The solution should turn pink.

*because you only titrate 10.00 mL of the 50.00 mL of NaOH (see step B2 above), you will have to multiply the number of mL of HCl titrant by 5 in your calculations.
2. Ensure that you have taken your initial burette reading and proceed to titrate the solution of Aspirin® in sodium hydroxide against hydrochloric acid. The end-point is when the pink colour of the phenolphthalein disappears (i.e., colourless). Record the burette reading when the end-point is reached.

Repeat the titration using the two remaining solutions of Aspirin® in sodium hydroxide.
Results, Calculations and Write-Up

A short report is required for this experiment. Report your results, do the necessary calculations, answer any questions, and write a conclusion. An outline of the required results and calculations is given below.

A. Standardization of a Sodium Hydroxide Solution

1. Tabulate your results as shown below (also see in the examples under the heading “Sample calculation”).

<table>
<thead>
<tr>
<th>Trial</th>
<th>Initial burette reading (mL)</th>
<th>Final burette reading (mL)</th>
<th>Volume of titrant (NaOH) added (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2. Estimate the approximate concentration of the sodium hydroxide solution from the mass of sodium hydroxide and the volume of water used. This concentration is just approximate, and is not to be used in subsequent calculations. However, it may be used as a rough to check to the answer obtained in step 7, below.

3. Write the equation for the reaction between hydrochloric acid and sodium hydroxide.

4. From the volume of the hydrochloric acid aliquot (the precise volume delivered by your burette, as determined in Experiment A1), and the known concentration, calculate the number of moles of acid that reacted in the neutralization reaction.

5. Use the equation for the reaction to calculate the number of moles of base that are used in the neutralization reaction.

6. If you have not already done so, calculate the average volume of sodium hydroxide used. Be sure to explain why you may have chosen to disregard certain results when computing this average.

7. From the results obtained in steps 5 and 6, determine the concentration of the sodium hydroxide solution.
B. Determination of the Percentage of Acetylsalicylic Acid in Aspirin®

1. Tabulate your results as shown in the table below.

<table>
<thead>
<tr>
<th>Trial #</th>
<th>Mass of Aspirin® tablet (g)</th>
<th>Volume of hydrochloric acid* required in back titration (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>___________________________</td>
<td>___________________________</td>
</tr>
<tr>
<td>2</td>
<td>___________________________</td>
<td>___________________________</td>
</tr>
<tr>
<td>3</td>
<td>___________________________</td>
<td>___________________________</td>
</tr>
<tr>
<td>(4)</td>
<td>___________________________</td>
<td>___________________________</td>
</tr>
</tbody>
</table>

(if necess.)

*because you titrate 10.00 mL of the 50.00 mL of NaOH used to dissolve your tablet, multiply the number of mL of HCl titrant in the back titration by 5.

Do the following calculations for each of the three trials.

2. Determine the number of moles of sodium hydroxide used to dissolve the Aspirin® tablet. (This will be the same in all three cases.)

3. From your titration data, determine the number of moles of hydrochloric acid added, and hence determine the number of moles of sodium hydroxide that did not react with the Aspirin®.

4. From the quantities determined so far, calculate the number of moles of sodium hydroxide that reacted with the acetylsalicylic acid in the Aspirin®.

5. Remembering that 1 mole of acetylsalicylic acid reacts with 2 moles of sodium hydroxide, calculate the number of moles of acetylsalicylic acid in the Aspirin® tablet.

6. Calculate the mass of acetylsalicylic acid in the Aspirin® tablet.

7. Determine the percentage of acetylsalicylic acid in the Aspirin® tablet.

   From your results, what is the average percentage of acetylsalicylic acid in an Aspirin® tablet?

WARNING: As with most of the experiments in this course, your grade for this experiment will be largely determined by the accuracy and precision of your results. Do not proceed to the next experiment until you are satisfied that your titration results are (i) complete and (ii) determined to the required degree of precision.
Additional Reading

For a slightly more extensive review of errors and the rejection of results see:


or


Both books are available from the Athabasca University library.
Experiment A3 Questions

1. Write the net ionic equation for
   
   a. the reaction of hydrochloric acid with sodium hydroxide
   
   b. the reaction of acetic acid with sodium hydroxide
   
   (Hint: Look in your textbook, if you are uncertain about how to write net ionic equations. Also note that acetic acid is a weak acid, and should be written in molecular form in the equation for 1 b. above.)
Experiment A4:  
The Determination of the Universal Gas Constant

Prerequisite Skills

Before you begin this experiment, you must have completed both Experiments A2 and A3. In particular, you must know how to prepare a standard solution and how to use a volumetric pipette.

Objectives

When you have completed the following experiment, you will have

1. obtained further practice at preparing solutions of known concentration.  
2. carried out a chemical reaction in a quantitative manner.  
3. collected a sample of gas “over water.”

The experiment also illustrates

1. the application of the concept of limiting reagent.  
2. the ideal gas equation.  

Safety Tips

Experiment location. Work on a steady table in a safe secure place. Keep doors and windows closed to prevent drafts.

Handling of chemicals. Sulfamic acid powder is dusting and corrosive. Avoid inhaling.

Safety glasses should be worn at all times. Wear appropriate clothes and closed-toed shoes.
Introduction and Theory

The ideal gas equation relates the pressure (P), volume (V) and temperature (T) of a gas to the amount (i.e., number of moles, n) of gas present:

\[ PV = nRT \]

where R is a proportionality constant known as the universal gas constant.

In this experiment you will prepare a known amount of nitrogen gas, and collect it, over water, at a known temperature and pressure. By measuring the volume of nitrogen gas collected, you will obtain values for all the variables in the ideal gas equation, and will be able to determine the value of R.

The nitrogen gas will be produced by the reaction of a known amount of sulfamic acid, HSO₃NH₂, with an excess of sodium nitrite, NaNO₂:

\[
\text{HSO}_3\text{NH}_2 (aq) + \text{NaNO}_2 (aq) \rightarrow \text{N}_2 (g) + \text{H}_2\text{O} (l) + \text{NaHSO}_4 (aq)
\]

This reaction gives a quantitative yield of nitrogen gas, and, when sulfamic acid is the limiting reagent:

\[
\text{amount of nitrogen produced} = \text{amount of sulfamic acid used}
\]

(Remember, in the SI, an “amount of substance” is expressed in moles.)

When a gas is collected “over water,” as in this experiment, the pressure exerted by the gas results both from the molecules of the gas itself, and from the molecules of water vapour that are also present. Hence, you must take the latter quantity into account before substituting the observed pressure into the ideal gas equation. The pressure that must be used in this equation is the partial pressure of the gas concerned, P(N₂), which you can obtain using Dalton’s Law of Partial Pressures:

\[ P(\text{Tot}) = P(\text{N}_2) + P(\text{H}_2\text{O}) \]

where P(Tot) is the total (atmospheric) pressure,

and P(H₂O) is the water vapour pressure at the temperature of the gas.
Chemical and Material Requirements

150 mL beaker
10 mL plastic pipette/burette
syringe + tubing + glass bead
retort stand and clothespin clamps
pipette bulb

125 mL Erlenmeyer flask
latex tubing + plastic tube and rubber stopper
1 glass vial (that fits inside the filter flask)
long tweezers (for holding onto the vial)

top loading/general pan balance
vial for measuring out sulfamic acid
sulfamic acid (preweighed, 0.20xx g per student, use as supplied)
100 mL volumetric flask
water

vial for measuring out sodium nitrite
sodium nitrite (>0.3 g per student, excess supplied. Must be weighed out by student.)
50 mL volumetric flask
water

5 mL volumetric pipette
10 mL volumetric pipette

List of Reagents

<table>
<thead>
<tr>
<th>Compound</th>
<th>Name</th>
<th>Molecular Weight</th>
<th>Solubility</th>
</tr>
</thead>
<tbody>
<tr>
<td>distilled H₂O</td>
<td>water, 55 M</td>
<td>18.02 g/mol</td>
<td>Scalds if hot!</td>
</tr>
<tr>
<td>*HSO₃NH₂</td>
<td>sulfamic acid</td>
<td>97.09 g/mol</td>
<td>Corrosive</td>
</tr>
<tr>
<td>*NaNO₂</td>
<td>sodium nitrite</td>
<td>69.00 g/mol</td>
<td>Oxidizer, toxic</td>
</tr>
</tbody>
</table>

* indicates solution must be made fresh on each lab day
Procedure (total time for completion ~2.5 hours)

A. Apparatus Setup for Determination of the Universal Gas Constant

1. Obtain about 100 mL of water in a 150 mL beaker and place the beaker on the base of a retort stand.

2. Fill a 10 mL pipette/burette to the 10-mL mark with water. Keeping the tap of the pipette/burette closed, place your thumb over the open end of the pipette/burette and immerse the open end of the pipette/burette in the water contained in the 150 mL beaker (see Figure A3.1). Some air should be present at the top end of the pipette/burette so that the level of the water is within the graduated part of the pipette/burette.

3. Thoroughly clean a 125 mL Erlenmeyer flask, giving it a final rinse with water.

4. The other end of the rubber tubing (bent straw U tube) tucks loosely inside the bottom end of the 10 mL gas trap plastic pipette/burette, as shown in Figure A4.1. Next, place the rubber stopper, with the short length of rubber tubing attach to it, into the top of the Erlenmeyer flask, making sure it forms a tight seal (see Figure A4.1). To minimize the chances of the whole apparatus being upset, it is a good idea to clamp the pipette/burette to a retort stand.

B. Reagent Setup for Determination of the Universal Gas Constant

1. Locate the provided, “precisely” pre-measured, (~0.20xx g) sulfamic acid in a glass vial. Record the indicated mass of the sulfamic acid.

2. Transfer all the sulfamic acid to a 100 mL volumetric flask, add a small amount of water in order to dissolve the crystals, and then add water until the level of the solution reaches the graduation mark on the neck of the flask. Mix the solution thoroughly by inverting the flask several times. Also measure and record the mass of the now “empty” vial, using the general pan balance.

3. Use a 10 mL volumetric pipette in order to transfer 10 mL of the sulfamic acid solution from the volumetric flask to the 125 mL Erlenmeyer flask.

4. Calculate the mass of sodium nitrite necessary to react with all of the sulfamic acid used to prepare your standard solution. Add about 0.15 g to the figure you obtain and measure out approximately this total mass of sodium nitrite into a glass vial using a general-purpose balance. Determine the precise mass of the vial and its contents using the general pan balance.
5. Transfer the calculated amount (x.x +0.15g) sodium nitrite to a 50 mL volumetric flask, add a small amount of water in order to dissolve the crystals, and then add water until the level of the solution reaches the graduation mark on the neck of the flask. Mix the solution thoroughly by inverting the flask several times. Measure the mass of the “empty” glass vial using your general pan balance.

![Diagram of apparatus for determining the value of the universal gas constant](image)

**Figure A4.1**
Apparatus for determining the value of the universal gas constant

6. Use a 5 mL volumetric pipette to transfer 5 mL of the sodium nitrite solution from the volumetric flask to a glass vial having a diameter of about 15 mm (that you can lower into the 125 mL Erlenmeyer flask).

C. Gas Formation and Collection for Determination of the Universal Gas Constant

1. Using the tweezers, carefully lower the glass vial containing the sodium nitrite into the 125 mL Erlenmeyer flask containing the sulfamic acid solution. Be sure not to spill any of the sodium nitrite solution, otherwise the reaction between the sodium nitrite and the sulfamic acid will begin prematurely and you will have to begin the experiment again. Carefully insert a rubber stopper into the mouth of the 125 mL Erlenmeyer flask.

2. Your apparatus should now be fully assembled as in Figure A4.1. Make sure that the system is airtight. Record the level of the water in
the pipette/burette (to two places past the decimal, as usual). The level should be at or below the top mark.

3. Start the reaction by tilting the 125 mL Erlenmeyer flask so that the vial containing the sodium nitrite tips over and the solutions of sodium nitrite and sulfamic acid are mixed together. Gently swirl the flask so that a complete reaction occurs. Nitrogen gas will be evolved and will be collected in the pipette/burette. This will cause the level of the water in the pipette/burette to fall. Continue to swirl the flask for at least ten minutes, by which time the level of the water in the pipette/burette should have stabilized.

4. Record the final level of the water in the pipette/burette.

5. Determine the distance (in cm) between the level of the water in the pipette/burette and the level of the water in the beaker (see Figure A4.2).

![Figure A4.2](image)

Note: Bent straw fits inside pipette loosely

6. Record the room temperature. This should be the same as the temperature of the gas in the pipette/burette.

7. Repeat the experiment two more times, using the solutions of sodium nitrite and sulfamic acid that you have already prepared.

8. Obtain the atmospheric pressure. (Consult Environment Canada’s Web site to find out today’s barometric pressure [http://weatheroffice.ec.gc.ca/canada_e.html](http://weatheroffice.ec.gc.ca/canada_e.html), or call Environment Canada at telephone: 819-997-2800 or 1-800-668-6767.

THE DISPOSAL OF EXCESS REAGENTS: Under certain circumstances, the disposal of nitrites can present problems. However, as the sodium nitrite solution used in this experiment is very dilute, any excess may be poured down the drain with a copious amount of water.
Results, Calculations and Write-Up

A formal report is required for this experiment, refer to the “Writing laboratory reports” section of this manual. An outline of the required results and calculations is given below.

A. Determination of the Universal Gas Constant

Results

\[
\begin{align*}
\text{mass of vial + sulfamic acid} & = \text{mass of vial} + \text{sulfamic acid} \\
\text{mass of “empty” vial} & = \text{mass of “empty” vial} \\
\text{mass of sulfamic acid used} & = \text{mass of sulfamic acid used} \\
\text{mass of vial + sodium nitrite} & = \text{mass of vial} + \text{sodium nitrite} \\
\text{mass of “empty” vial} & = \text{mass of “empty” vial} \\
\text{mass of sodium nitrite used} & = \text{mass of sodium nitrite used}
\end{align*}
\]

<table>
<thead>
<tr>
<th>Trial 1</th>
<th>Trial 2</th>
<th>Trial 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial reading of burette</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Final reading of burette</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Volume of gas collected</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Room temperature</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Height of water column*</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Distance between level of water in the pipette/burette and in the beaker at the end of the run

Calculations

\[
\begin{align*}
\text{amount (number of moles) of sulfamic acid used to prepare 100 mL of solution} & = \frac{\text{mass of sulfamic acid}}{\text{molar mass of sulfamic acid}} \\
\text{concentration of sulfamic acid solution} & = \frac{\text{amount (number of moles) of sulfamic acid used in the reaction}}{\text{volume of solution}} \\
\text{amount (number of moles) of sodium nitrite used to prepare 50 mL of solution} & = \frac{\text{mass of sodium nitrite}}{\text{molar mass of sodium nitrite}} \\
\text{concentration of sodium nitrite solution} & = \frac{\text{amount (number of moles) of sodium nitrite used in the reaction}}{\text{volume of solution}}
\end{align*}
\]
Note: The amount of sodium nitrite used should be greater than the amount of sulfamic acid used, thus the sulfamic acid is the limiting reagent.

According to the balanced chemical equation for the reaction that has taken place,

\[
\text{NaNO}_2 (\text{aq}) + \text{HSO}_3\text{NH}_2 (\text{aq}) \rightarrow \text{N}_2 (g) + \text{H}_2\text{O} (l) + \text{NaHSO}_4 (\text{aq})
\]

for each mole of sulfamic acid reacted, one mole of nitrogen gas will be produced. Thus, because sulfamic acid was the limiting reagent, you can say:

\[
\text{amount (number of moles) of nitrogen produced} = \text{amount (number of moles) sulfamic acid used}
\]

As the amount of sulfamic acid used was the same in each of your three trials, the amount of nitrogen produced was the same in each trial.

In order to determine the value of the ideal gas constant, R, using the ideal gas law:

\[
P V = nRT
\]

you must know:

P, the pressure of the nitrogen gas in the pipette/burette (kPa)
V, the volume of gas in the pipette/burette (L)
n, the number of moles of nitrogen gas in the pipette/burette (mol)
T, the absolute temperature (K)

Note again that n, the number of moles, is known. Similarly, you have determined both the volume, V, and temperature, T, in your experiment. You have also obtained a value for the pressure, P, but this is not the value that you should use in your calculations. Remember that the gas in the pipette/burette is not pure nitrogen, but contains a significant amount of water vapour. In principle, you can calculate the partial pressure of the nitrogen by using Dalton’s Law of Partial Pressures:

\[
P(\text{Tot}) = P(\text{N}_2) + P(\text{H}_2\text{O})
\]

where

P(\text{Tot}) = \text{the total (atmospheric) pressure}
P(\text{N}_2) = \text{the desired partial pressure of nitrogen}
$P(H_2O) = \text{the partial pressure of water (i.e., the vapour pressure of water at temperature T) obtained from p. 199 of the text (Jones and Atkins, 4th ed.) or from the CRC handbook.}$

However, the atmospheric pressure, $P(Tot)$, is actually equal to the partial pressure of nitrogen, $P(N_2)$, plus the partial pressure of water $P(H_2O)$, plus the pressure due to the column of water in the pipette/burette, $P(Col)$. Thus,

$$P(Tot) = P(N_2) + P(H_2O) + P(Col) \quad (1)$$

If the difference between the level of water in the pipette/burette and the level of water in the beaker is $h$ cm, then

$$P(Col) = \frac{h \text{ cm}}{1.00 \times 10^3 \text{ cm}} \times 101.3 \text{ kPa}$$

The latter relationship is derived from the fact that a column of water that is $1.00 \times 10^3$ cm high exerts a pressure of 101.3 kPa at 298 K.

Use relationship (1) to calculate the value of $P(N_2)$ and use this partial pressure in your determination of $R$.

Calculate the value of $R$, using the results from each of your three trials. Determine the mean value, and give some indication of the precision of your result. Look up the accepted value of $R$ and calculate your percentage error.

**Note:** When calculating $R$ as described above, you will obtain units of $\text{kPa} \cdot \text{L} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$. Most textbooks that employ SI units will quote a value for $R$ given in $\text{J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$. This is not a problem, as $1 \cdot \text{J} = 1 \cdot \text{kPa} \cdot \text{L}$.

**WARNING:** As with most of the experiments in this course, your grade for this experiment will depend primarily on the accuracy and precision of your results. Do not proceed to the next experiment until you are satisfied that you have completed this experiment to the best of your ability.
Experiment A4 Questions

1. When 5.00 mL of a sodium nitrite solution containing 17.04 g/L of sodium nitrite is added to 25.00 mL of sulfamic acid solution containing 2.754 g/L of sulfamic acid, the following reaction occurs:

\[
\text{NaNO}_2(\text{aq}) + \text{HSO}_3\text{NH}_2(\text{aq}) \rightarrow \text{N}_2(\text{g}) + \text{H}_2\text{O}(\text{l}) + \text{NaHSO}_4(\text{aq})
\]

sodium nitrite sulfamic acid nitrogen water sodium sulfate

a. Determine the mass of nitrogen gas produced, and calculate the volume occupied by this mass of gas at a temperature of 23°C and a pressure of 99.5 kPa.

b. When this experiment was carried out by a student and the nitrogen gas was collected over water, the volume of “wet” nitrogen produced (at 23°C and 99.5 kPa) was 14.81 mL. What was the percentage yield obtained by the student? The vapour pressure of water at 23°C is 2.81 kPa.

Note: \[
\text{percentage yield} = \frac{\text{actual yield}}{\text{theoretical yield}} \times 100\%
\]

2. Show (via mathematical proof) that 1 kPa · L = 1 J.
Block B Experiments

Note: Students must have read and understood the Safety Rules for home-study found on page 32 of this manual and have signed the Safety Pledge on page 7. No short or formal written reports are required for any of the experiments in this block. You need only complete the accompanying observation and question sheets.
Experiment B1: Solutions

Prerequisite Skills
No prerequisite skills have been assigned to this laboratory.

Objectives
When you have completed the following experiment, you will have
1. learned of the different factors involved in the solution process.
2. learned of the different factors affecting solubility.
3. seen illustrated some of the colligative properties of solutions.
4. determined the conductivity of various common electrolytic and non-electrolytic solutions.

Safety Tips

Experiment Location. Work on a steady table in a safe secure place. Keep doors and windows closed to prevent drafts. Your kitchen stove will be used as a heat source. Do no cooking while the experiment is in progress.

Handling of Chemicals. All are common household chemicals, but should be treated with care.

Safety glasses should be worn at all times. Wear appropriate clothes and closed-toed shoes.
Introduction and Theory

Dissolving a solute into a solvent forms a solution. For example, salt water (a solution) is formed by dissolving salt (a solute) into water (a solvent).

The rate of dissolution of a solute into a solvent is dependent on several factors. These factors include solvent temperature, particle size, the chemical nature of both solvent and solute, mixing rate and concentration of the solution. In general, a solute will dissolve more readily in a vigorously stirred hot solution when it is finely divided. Also, the more dilute the solution is the more readily the solute can dissolve into it.

A solution that is concentrated to the point where no more solute will dissolve into it, is said to be “saturated.” If the solution can dissolve more solute it is unsaturated. It is possible to add more solute to a saturated solution by warming it. If the solution is then cooled so it holds more solute for the lower temperature, it is said to be “supersaturated.”

A solution is said to be an electrolyte if it conducts electricity. The conductivity is enhanced by the number of ions in solution. Ionic substances (e.g., NaCl) dissolve in water to form ions (e.g., Na\(^+\) and Cl\(^-\)) that conduct electricity. Covalent compounds (e.g., sugar) dissolve in water to form molecules (e.g., sugar molecule), which do not aid in conducting electricity. Strong electrolytes completely ionize in water and weak electrolytes only partially ionize.

In this experiment your conductivity apparatus indicates the amount of conductivity by how brightly the light bulb shines. The more electrolytic the solution the brighter the light bulb will shine.

Colligative properties are a collective group of physical properties of a solution dependent on the number of particles in that solution. These properties include vapour pressure, freezing point, boiling point and osmotic pressure. Both freezing point depression and boiling point elevation are due to the decrease in vapour pressure of the solution compared to the pure solvent (See Figure B1.1).

![Figure B1.1](image_url)

Effect of vapour pressure on boiling and freezing points
We take advantage of these colligative properties in our everyday lives. For example, salt is used on icy roads and sidewalks to aid the melting of the ice. When salt is added to boiling water the temperature is higher and food cooks faster.

Osmosis occurs when two solutions of different concentrations are separated by a semipermeable membrane. The solvent flows from the dilute solution (hypotonic solution) towards the more concentrated solution (hypertonic solution) until both solutions are of equal concentration (isotonic solution). There are several interesting examples of osmosis. A small cucumber placed in brine loses water by osmosis and shrivels into a pickle. People eating a lot of salt can retain water in their tissue cells by osmosis.

Osmotic pressure is the amount of pressure required above the concentrated solution to prevent osmosis. Often the relative osmotic pressure is referred to rather than a solution’s concentration.
Chemical and Material Requirements

You are supplied with:

2 small test tubes
2 large test tubes
1 50 mL beaker
1 150 mL beaker
1 stir stick (glass rod)
1 alcohol thermometer
1 dialysis tube (~30 cm, cut into 2 equal lengths)
1 conductivity apparatus
1 rock salt
1 instant coffee crystals

You must provide:

corn syrup
ammonia (household brand, ~3% solution)
2 cotton threads (20 cm)
paper towels
ice cubes or crushed ice
pencil or pen
table salt
sugar
bleach
vinegar
stove or heating surface
cookie sheet
small pot
oven
string (used to tie dialysis tubing)
2 bowls (for dialysis tubing)
measuring cups
small ruler
stop watch for Part A
oven mitts
Procedure

A. Solubility

1. Dissolution vs. Particle Size Dissolution

Fill one small test tube to a depth of about 0.3 cm with table salt and fill the other to a depth of 0.3 cm with rock salt. Now add about 8 mL (1/2 tbsp) of cold tap water to each. Cover both tubes with a thumb and shake. Note the time it takes to dissolve the salt in each tube.

2. Dissolution vs. Stirring

Fill two small test tubes to 0.3 cm depth with table salt and add about 8 mL of cold tap water to each. Set one tube aside and shake the other one covering the mouth of the test tube with your thumb. Note the time it takes to dissolve the salt in each tube.

3. Dissolution vs. Temperature

Measure out two salt samples, each 1/8 of a teaspoon. Fill one small test tube with 8 mL of cold tap water and the other tube with 8 mL of hot tap water. Cover the mouth of both tubes with a thumb and then shake. Note the time it takes to dissolve the salt in each tube.

4. Dissolution vs. Diffusion

Take two to three crystal chunks of instant coffee and wrap in a small piece of paper towel. Place this packet at the bottom of one of the large test tubes and fill with cold tap water. Add cold tap water to a second large test tube. Prepare a second coffee packet and carefully place this packet on top of the water, making sure it is completely submerged in the water. Set both tubes aside in an upright position. Leave these test tubes undisturbed for at least two hours, checking occasionally on the progress of both tubes. You may need to carefully lift both tubes up and hold them against a white sheet of paper to notice any results. Record your observations.

B. Saturation

1. Saturated Solution

Fill a 150 mL beaker one-third full of cold tap water, and add sugar slowly while stirring. Keep adding sugar until no more sugar dissolves. (Be patient—it takes a lot of sugar!) You will know that the solution is saturated when a few crystals of sugar remain at the bottom of the beaker.
2. Increased Temperature

Fill a small pot about one-quarter full of hot tap water and put on low heat on a stove or heating surface. **CAUTION: Do not touch the hot pot or the hot water. You may get burned!** Place the beaker with sugar water (from step 1 above) into the hot water, making sure it will not tip over. Warm the sugar solution gently while stirring. Note what happens to the sugar crystals at the bottom of the beaker. Now continue to add more sugar so that you have a saturated solution (again—be patient!) This time make sure there are no extra crystals of sugar at the bottom of the beaker at your saturation endpoint. The easiest way to accomplish this is to follow the same procedure as in step 1 and then add a little water to dissolve the last few crystals. Save this solution and keep it warm, you will need it for later.

3. Evaporation

Pour half of your solution from step 2 into a 50 mL beaker and place in the oven at 175°C (350 degrees F). Allow about half the water in this 50 mL beaker to evaporate by boiling. (Don’t forget about it!) Take the beaker out of the oven and allow it to cool. Record your results. While waiting continue on with step 4.

4. Supersaturation

Remove the other half of the warm sugar solution from step 2 and allow it to cool undisturbed. The sugar should remain in solution and upon cooling will form a supersaturated solution. The excess sugar can be brought out of solution by “seeding” the solution with a crystal of sugar. Tie a coarse string (frayed a little is OK) to the center of a pencil. Dip the end of the thread in some corn syrup and stick a few sugar crystals to the thread. Lower the thread and sugar gently into the saturated solution and support the thread by laying the pencil across the mouth of the beaker. You can adjust the height of the suspended thread by rotating the pencil in the appropriate direction and winding the thread in and out. Allow this setup to sit undisturbed for a few days. After a few days you should have some nice rock candy. Record your observations. **CAUTION: Do not eat the rock candy formed in this experiment!**

C. Colligative Properties

1. Freezing Point Depression

Add lots of ice to a cup of water and stir. Allow the water to come to as low a temperature as possible. Measure and record the water temperature. Add 25 g of table salt to the mixture and stir. After about five minutes record the temperature of the cold salt water. You should observe a noticeable temperature change. Just for fun, take a cup of cold water and float an ice cube in it. Carefully dangle a cotton thread
above the ice-cube and lay the bottom end onto the top of the floating ice cube. Shake a small amount of salt on top of the ice cube around the area that the thread touches the ice. Wait one minute and then carefully pull up on the thread. The ice cube and thread should be welded together. Try the same procedure without the salt.

2. **Boiling Point Elevation**

Boil water in a small pot on the stove. Measure and record the temperature of the water. **CAUTION: Do not let the tip of the thermometer touch the bottom of the pot.** Now slowly add 25 g (about 1 tablespoon, or 15 ml) of salt to the boiling water. The water will stop boiling momentarily, because the salt has a slight cooling effect (it is at room temperature when added). Allow the water to boil again and then record the new temperature.

3. **Hypotonic and Hypertonic Solutions**

Soak two pieces of dialysis tubing in a beaker of tap water for 2–3 minutes. Open the tubing by rubbing one end between your index finger and thumb. Use an object like an unsharpened pencil to open the tube all the way. After the tube is opened, tie one end closed with a piece of string or a twist tie. Place the tube back in water and repeat with the other tube. In a small container mix 60 ml (1/4 cup) corn syrup with 250 ml (1 cup) water and stir well. Take one of the knotted dialysis tubes and fill it about 5 cm full with the corn syrup solution. Leave a small air space in the tube. Twist and seal the end with a piece of string. Follow the same procedure with the other tube, but fill it only with an equal volume of tap water. Make sure both tubes are the same size and have the same size air bubble. Put the corn syrup tube in a bowl of tap water and place the tap water tube in a bowl of the corn syrup solution. Leave overnight and record your results the following day.

D. **Electrolytes**

Using the conductivity apparatus in your home lab kit (see Figure B1.2), measure the conductivity of a beaker of tap water by placing both the leads into the water. Do not touch the leads to each other as it will drain your battery. Note the intensity of the light from the light bulb (dim room lights?). Repeat this measurement on the following list of solutions, making sure to rinse the leads with tap water between measurements:

- salt water
- vinegar
- ammonia
- sugar water
- bleach

**CAUTION:** These are all common household chemicals, but should be treated with care. Wear safety goggles and gloves at all times! After you
have taken the measurements, make sure that the battery is disconnected from the conductivity apparatus.

Figure B1.2
Conductivity apparatus
Report Form

Microlab Lab B1 – Solutions

Name: ____________________ ID No.: ____________ Date: ____________

A. Solubility

Particle Size

time required to dissolve the salt

____________ rock salt

____________ table salt

Stirring

time required to dissolve the salt

____________ shaken test tube

____________ undisturbed test tube

Temperature

time required to dissolve the salt

____________ cold water

____________ hot water

Diffusion

Record Observations

packet at bottom of test tube

packet at top of test tube
Questions

1. Complete the following table.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Rate of Dissolution Increased or Decreased?</th>
<th>Why?</th>
</tr>
</thead>
<tbody>
<tr>
<td>Increased Temperature</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Small Particle Size</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Stirring or Agitation</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2. Describe and explain the difference between the processes of dissolving and diffusion.
B. Saturation

Saturation and Increased Temperature

Observations (effects of increased temperature on saturation)

Evaporation

Observations

Supersaturation

Observations
Questions

1. What can be said about the sugar water when the crystals on the thread finally stop growing?

2. What happens to the concentration of the sugar solution as water evaporates from it?
C. Colligative Properties

Freezing Point Depression

temperature of the ice water

____________ without salt

____________ with salt

Boiling Point Elevation

temperature of the boiling water

____________ without salt

____________ with salt

Hypotonic and Hypertonic Solutions

Observations

corn syrup dialysis tube in water

water dialysis tube in corn syrup
Questions

1. How many grams of sugar (C\textsubscript{12}H\textsubscript{22}O\textsubscript{11}) would cause the same amount of freezing point depression as 25 g of salt (NaCl)?

2. Explain why salt causes an increase in the boiling point of water.

3. Label the hypertonic and hypotonic side and direction of water flow in the following diagram.
d. Electrolytes

<table>
<thead>
<tr>
<th>Liquid/Solution</th>
<th>Brightness of Light Bulb</th>
</tr>
</thead>
<tbody>
<tr>
<td>water</td>
<td></td>
</tr>
<tr>
<td>salt water</td>
<td></td>
</tr>
<tr>
<td>vinegar</td>
<td></td>
</tr>
<tr>
<td>ammonia</td>
<td></td>
</tr>
<tr>
<td>sugar</td>
<td></td>
</tr>
<tr>
<td>bleach</td>
<td></td>
</tr>
</tbody>
</table>

Questions

1. Which of the test solutions are ionic? Defend your answer(s).

2. In some cases tap water will conduct electricity. Why?
Experiment B2: 
The Determination of an Enthalpy Change 
that Cannot Be Measured Directly

Prerequisite Skills

No prerequisite skills have been assigned to this laboratory.

Objectives

When you have completed the following experiment, you will have learned how enthalpy changes are measured using a simple calorimeter.

The experiment also illustrates

1. how the enthalpy change, $\Delta H$, may be calculated for a reaction taking place in a simple calorimeter.

2. how Hess’s Law may be used to determine the enthalpy change for a reaction that cannot be studied directly.

Safety Tips

Experiment location. Work on a steady table in a safe secure place. Keep doors and windows closed to prevent drafts.

Handling of chemicals. Hydrochloric acid is corrosive. Avoid skin contact. Flush any acid contacted/affected area immediately and thoroughly with tap water.

Safety glasses should be worn at all times. Wear appropriate clothes and closed-toed shoes.
Introduction and Theory

In many reactions, heat is either given off or absorbed. Such enthalpy changes result from the difference in energy between the bonds that are broken in the reactants and those that are formed in the products, and is also dependent upon the amount of substance taking part in the reaction.

Enthalpy changes are measured in calorimeters. The latter can be as simple as a Styrofoam cup, or as complex as a bomb calorimeter. In all cases, the determination depends upon the fact that the heat evolved (or absorbed) by the reaction is equal to the heat absorbed (or evolved) by the calorimeter and its contents.

In this experiment you will determine the heat capacity of your calorimeter, \( C_p \) (cal), and measure the enthalpy change for each of two reactions. You will then use your results to calculate the enthalpy change for a reaction that cannot be measured directly, and you will compare your results to the literature value.

The ultimate objective of this experiment is to determine the enthalpy of formation \( (\Delta H_f) \) of magnesium oxide. In other words you will determine the enthalpy change for the reaction:

\[
\text{Mg (s) + 1/2 O}_2 (g) \rightarrow \text{MgO (s)} \quad (1)
\]

You will do this by determining the enthalpy change for the reaction of magnesium with hydrochloric acid:

\[
\text{Mg (s) + 2 H}^+ (aq) \rightarrow \text{Mg}^{2+} (aq) + \text{H}_2 (g) \quad (2)
\]

and for the reaction of magnesium oxide with hydrochloric acid:

\[
\text{MgO (s) + 2 H}^+ (aq) \rightarrow \text{Mg}^{2+} (aq) + \text{H}_2\text{O (l)} \quad (3)
\]

Using these results, and the standard enthalpy of formation of water \((-286 \text{ kJ} \cdot \text{mol}^{-1})\), you will apply Hess’s Law to find the standard enthalpy of formation of magnesium oxide.

Before starting this experiment, you should satisfy yourself that \( \Delta H_f(\text{MgO}) \) can be obtained from \( \Delta H \) for equation (2), \( \Delta H \) for equation (3), and \( \Delta H_f^\circ \) (\( \text{H}_2\text{O (l)} \)).

In order to determine how much heat is released by the reaction of magnesium or magnesium oxide with hydrochloric acid, you must be able to calculate how much of this heat is absorbed by the calorimeter, and how much is absorbed by the reaction mixture itself. For present purposes, you may assume that the latter consists only of water.
Thus:

heat released during reaction
= heat gained by calorimeter + heat gained by reaction mixture

\[ q = q_{\text{cal}} + q_{\text{mix}} = C_p(\text{cal}) \Delta T + m c \Delta T \]

where

\( C_p(\text{cal}) \) = the heat capacity of the calorimeter
\( \Delta T \) = the observed temperature change
\( m \) = the mass of the reaction mixture
\( c \) = the specific heat of the reaction mixture (assumed to be 4.18 J \( \cdot \) g\(^{-1}\) \cdot K\(^{-1}\))

In order to determine \( C_p(\text{cal}) \), you will carry out a preliminary experiment in which you will pour a known mass of hot water at temperature \( T_1 \) into a calorimeter containing a known mass of cold water at \( T_2 \), and measure the temperature of the resulting liquid.

**Calculating Heat Capacity of the Calorimeter \( C_p(\text{cal}) \)**

Heat lost by hot water = heat gained by calorimeter and cold water, i.e.,

\[ m_1 c \Delta T_1 = C_p(\text{cal}) \Delta T_2 + m_2 c \Delta T_2 \]

where

\( m_1 \) = mass of hot water used (assume 10 mL = 10 g)
\( c \) = specific heat capacity of water = 4.184 J \( \cdot \) g\(^{-1}\) \cdot K\(^{-1}\)
\( \Delta T_1 \) = temperature change experienced by hot water = \( |T_i(\text{hot}) - T_f| \)
\( m_2 \) = mass of cold water used (assume 10 mL = 10 g)
\( \Delta T_2 \) = temperature change experienced by calorimeter and its contents

\[ = |T_f - T_i(\text{cold})| \]

\( C_p(\text{cal}) \) = heat capacity of the calorimeter
\( T_i(\text{hot}) \) = the initial temperature of the hot water
\( T_i(\text{cold}) \) = the initial temperature of the calorimeter and its contents
\( T_f \) = the final temperature of the calorimeter and its contents

Of the required data, only \( T_f \), the final temperature of the calorimeter and its contents cannot be measured directly. This is because the calorimeters that we use are not perfectly insulated. Therefore, some heat leaks out of the system into the surroundings. Also, it takes a finite time for any change
in temperature to equilibrate throughout the calorimeter, and for the liquid in the thermometer to respond to this change. To correct for these sources of error, you will determine $T_f$ graphically by plotting temperature ($y$ axis) against time ($x$ axis), and extrapolating back to time $= 0$ s (see Figure B2.1).

Figure B2.1
Method of determining $\Delta T$ in a simple calorimetry experiment

Once $T_i$ has been determined in this way, $\Delta T$ can be calculated as shown and then it is simply a matter of substituting the appropriate values into the given equation in order to find the heat capacity of the calorimeter.

**Note:** The heat capacity of the calorimeter should be in the order of $10–20$ J°C$^{-1}$. 
Chemical and Material Requirements

You are supplied with:

1 alcohol thermometer
1 stirring wire
1 calorimetric vial
1 rubber stopper
1 large beaker + insulating material or
  1 foam block for calorimetric vial
4 bottles dilute HCl (1M), 20 mL/bottle
2 vials magnesium powder (preweighed, use as supplied 0.12xx g)
2 vials magnesium oxide (preweighed, use as supplied 0.25xx g)

You will need:

1 stopwatch or clock with a sweep second hand
Procedure

A. Determination of the Heat Capacity of the Calorimeter

1. Obtain a 10-mL measuring vessel and measure out 10 mL of cold tap water into the vial of the calorimeter.

2. Monitor the temperature of the water in the calorimeter for several minutes. When the reading appears to be constant, record the temperature.

3. Measure out 10 mL of hot tap water (about 40°C or so) into the 10 mL measuring vessel.

4. Quickly measure the temperature of this hot water then pour the contents of this measuring vessel into the cold water in the calorimeter. Record the temperature of the contents of the calorimeter as soon as possible, and then every 15 seconds for three minutes. Stir constantly during this period.

5. Pour the liquid contents of the calorimeter down the drain, and wipe the glass vial dry with tissue paper.

6. Repeat steps 1 through 5 using the same calorimeter vial as before. Be sure to use this same vial in Part B of the experiment.
B. **Determination of the Enthalpy of Formation of Magnesium Oxide**

**Magnesium Reaction**

1. Ensure that the calorimeter vial used in Part A is clean and dry.

2. Assemble the calorimeter as shown in Figure B1.2. Pour the entire contents of the bottle of premeasured 20 mL of hydrochloric acid (1 mol \( \cdot \) L\(^{-1} \)) into the vial. Ensure that the thermometer bulb is below the surface of the liquid, and determine the temperature of the hydrochloric acid.

3. Pour the entire contents of one of the preweighed vials of magnesium powder (approximately 0.12 g) into the hydrochloric acid contained in the calorimeter. Stir the mixture vigorously, taking care not to hit the thermometer with the stirrer. Read the temperature of the reaction mixture every 15 seconds, until it reaches a maximum. Thereafter, read the temperature every minute for five minutes.

4. Check the contents of the calorimeter. If all the magnesium did not dissolve, you must start Part B again.

5. Dispose of the contents of the calorimeter vial down the drain with plenty of water. Wash the vial with water, and dry it with tissues or paper towels.

6. Repeat steps 1 though 5 for a second determination.

**Magnesium Oxide Reaction**

1. Set up the calorimeter as before. Again pour the entire contents of the bottle of premeasured 20 mL of hydrochloric acid (1 mol \( \cdot \) L\(^{-1} \)) into the calorimeter vial, and measure the temperature of the acid.

2. Pour the entire contents of one of the preweighed vials of magnesium oxide (approximately 0.25 g) into the hydrochloric acid contained in the calorimeter, stir thoroughly, and monitor the temperature change in exactly the same manner as you did in step 3, above.

3. Check the contents of the calorimeter. If you can see any unreacted magnesium oxide you must repeat the experiment.

4. Dispose of the contents of the calorimeter down the drain. Wash the cup with water, and dry it with paper towels or tissues.

5. Repeat steps 1 through 4 for a second determination.
Report Form

Microlab Lab B2—Enthalpy

Name: ___________________ ID No.: ____________ Date: ____________

A. Determination of the Heat Capacity of the Calorimeter

<table>
<thead>
<tr>
<th>Temperature of calorimeter contents (°C)</th>
<th>Time</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Run #1</td>
<td>Run #2</td>
<td>0 min</td>
</tr>
<tr>
<td></td>
<td></td>
<td>5 min</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Graph to Determine Heat Capacity of Calorimeter $C_p$ (cal)
Complete the following table to calculate $C_p(\text{cal})$:

<table>
<thead>
<tr>
<th>Run</th>
<th>$\Delta T_1$</th>
<th>$\Delta T_2$</th>
<th>$C_p(\text{cal})$</th>
</tr>
</thead>
<tbody>
<tr>
<td>#1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>#2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
B. Determination of the Enthalpy of Formation of Magnesium Oxide

Magnesium Reaction

Run #1 _____________ Run #2 _____________ Mass of magnesium powder

Run #1 _____________ Run #2 _____________ Moles of magnesium

<table>
<thead>
<tr>
<th>Temperature of calorimeter contents (°C)</th>
<th>Time</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Run #1</td>
<td>Run #2</td>
<td>0 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>HCl (aq) temperature stable before addition</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Mg added</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Reading every 15 s till maximum</td>
</tr>
<tr>
<td></td>
<td>15 s</td>
<td>Maximum reading</td>
</tr>
<tr>
<td></td>
<td>30 s</td>
<td>Reading every minute for 5 minutes</td>
</tr>
</tbody>
</table>
Graph to Determine $\Delta T$ Magnesium Reaction
Magnesium Oxide Reaction

<table>
<thead>
<tr>
<th>Temperature of calorimeter contents (°C)</th>
<th>Time</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Run #1</td>
<td>Run #2</td>
<td>0 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>15 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>30 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Graph to Determine $\Delta T$ Magnesium Oxide Reaction
Calculations

In this experiment you have studied two reactions:

\[
\text{Mg (s) + 2 HCl (aq) } \rightarrow \text{ H}_2 \text{ (g) + MgCl}_2 \text{ (aq) } \quad (\Delta H_1)
\]

\[
\text{MgO (s) + 2 HCl (aq) } \rightarrow \text{ H}_2 \text{O (l) + MgCl}_2 \text{ (aq) } \quad (\Delta H_2)
\]

You can find \( \Delta H \) for each of these reactions as follows:

Heat released by reaction (q)

\[= \text{heat gained by calorimeter} + \text{heat gained by contents} \]

\[= \text{Cp(cal)} \Delta T + mc\Delta T \]

where

\[\text{Cp(cal)} = \text{heat capacity of the calorimeter, as determined in Part A.}\]

\[\Delta T = \text{final temperature (Tf)} - \text{initial temp. (Ti)}; \text{Tf is found graphically; see Part A.}\]

\[m = \text{mass of the contents of the calorimeter}\]

\[c = \text{specific heat capacity of the contents of the calorimeter. We shall assume this to be the same as that of water, i.e., 4.184 } \text{ J} \cdot \text{g}^{-1} \cdot \text{K}^{-1}, \text{ as the contents of the calorimeter is mainly water.}\]

### Magnesium Reaction

<table>
<thead>
<tr>
<th>Run</th>
<th>( \Delta T ) (from graph)</th>
<th>q heat released by reaction</th>
<th>Amount of Mg (moles)</th>
<th>( \Delta H_1 ) Mg/HCl Reaction (kJ/mole)</th>
</tr>
</thead>
<tbody>
<tr>
<td>#1</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>#2</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Magnesium Oxide Reaction

<table>
<thead>
<tr>
<th>Run</th>
<th>( \Delta T ) (from graph)</th>
<th>q heat released by reaction</th>
<th>Amount of MgO (moles)</th>
<th>( \Delta H_2 ) MgO/HCl Reaction (kJ/mole)</th>
</tr>
</thead>
<tbody>
<tr>
<td>#1</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>#2</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
6. Look up the standard enthalpy of formation of water, and hence determine $\Delta H^\circ_f (\Delta H_3)$ for the reaction:

$$H_2(g) + \frac{1}{2}O_2(g) \rightarrow H_2O(l) \quad (\Delta H_3)$$

$\quad \qquad \text{______________} = \Delta H_3$

7. Apply Hess’s Law to the above results in order to calculate $\Delta H$ for the reaction:

$$Mg(s) + \frac{1}{2}O_2(g) \rightarrow MgO(s) \quad (\Delta H_7)$$

and hence determine $\Delta H_f$ for magnesium oxide.

$\quad \qquad \text{______________} = \Delta H^\circ_f \text{ for MgO}$

8. Use the textbook to look up and obtain the accepted value for $\Delta H^\circ_f \text{MgO(s)}$. Use this value to determine your percentage error.

$\quad \qquad \text{______________} = \text{percentage error}$
Experiment B3:
The Reactions and Identification of Some Common Cations

Prerequisite Skills

No prerequisite skills have been assigned to this laboratory.

Objectives

When you have completed the following experiment, you will have

1. learned how to test for the presence of certain cations in a substance, using small quantities of chemicals.

The experiment also illustrates

2. the characteristic reactions of a number of common cations.

3. net ionic equations.

4. line spectra.

Safety Tips

Experiment location. Work on a steady table in a safe secure place. Keep doors and windows closed to prevent drafts.

Handling of chemicals. Hydrochloric acid and sodium hydroxide solutions are corrosive. Handle the silver nitrate and potassium chromate solution droppers very carefully. When cutting open the solution droppers, hold the dropper upside down when cutting off the end of the dropper with scissors. Rinse your scissors off immediately after cutting the dropper open.

Avoid skin contact. Flush any chemically contacted/affected area immediately and thoroughly with tap water.

Safety glasses should be worn at all times. Wear appropriate clothes and closed-toed shoes.
Introduction and Theory

You will develop a scheme for determining which one of a number of common cations is present in a given compound. The cations you will investigate are silver, magnesium, calcium, barium, iron(III), potassium, sodium, and ammonium.

You will carry out a series of tests on a number of known cations. Your strategy will be to assign each of the eight cations to one of a number of “groups” by carrying out a minimum number of tests. You will then carry out one or more confirmatory tests that will allow you to distinguish among the different cations within any given group. The flowsheet, Figure B3.1, on the next page illustrates this process.

You will notice that the flame test is used to distinguish between compounds containing sodium and those containing potassium. This test is based on the fact that when energy is provided to an atom or ion, the electrons can be promoted from the ground state to an excited state, and when these electrons return to lower energy states, they emit radiation of a very specific wavelength. In the case of sodium and potassium ions, this radiation falls in the visible region of the electromagnetic spectrum; thus, when compounds of these elements are strongly heated under the conditions of the flame test, characteristic colours are produced. Other ions that give a positive response in the flame test, together with the colours imparted to the flame, include:

- lithium — crimson
- cesium — blue
- strontium — brilliant red
Figure B3.1
Flowsheet of tests for cation solutions
Chemical and Material Requirements

You are supplied with:

3 multi-cell well rows
1 alcohol burner (with wick and cap)
1 nichrome wire
8 cation solution droppers for Ag⁺, Ca²⁺, Ba²⁺, Mg²⁺, Fe³⁺, K⁺, Na⁺ and NH₄⁺
2 unknown cation solution droppers
1 HCl solution dropper
1 \((\text{NH}_4)_2\text{CO}_3\) solution dropper
1 NaOH solution dropper
1 K₂CrO₄ solution dropper
1 container of fuel for the alcohol burner

You must provide:

1 pair of scissors (use an old pair)
Procedure

A. Properties of Some Common Cations

Carry out the following tests using the small cell wells provided. It may be easiest to place the cell plates on a piece of paper, and write information about the contents of each cell on the paper next to the cell well. When mixing two liquids, always make sure that the two are mixed thoroughly by tapping the cell well. Write down your observations as you go along. Do not try to rely on your memory. CAUTION: These solutions are all toxic.

Test for Group I Cations

1. Fill eight cell wells about half way with each of the eight different labelled cation solutions and two with your unknown solutions. Add a few drops of the HCl solution to each of the wells and mix thoroughly by tapping the cells. Record which compounds give a precipitate. You will not use these cell wells again, but do not discard solutions yet.

Test for Group II Cations

1. Into clean cell wells, add a small amount (as in step 1) of each cation solution that did not form a precipitate above (step 1). Add a few drops of the (NH₄)₂CO₃ solution. Mix thoroughly and record results.

Test for Group III Cations

1. Into clean cell wells, add a small amount of each cation solution that did not form a precipitate in either step 1 or 2. Add a few drops of NaOH solution to the first well, mix thoroughly and record your results. If a precipitate does not form here check for the smell of ammonia. Record your results and do the same to each of the remaining wells. Do not add the NaOH solution to all the wells at once as it may be difficult to accurately check for the evolution of ammonia.

Confirmatory Tests for Group II Cations

1. Into clean cell wells, add a small amount of each cation solution that did form a precipitate with (NH₄)₂CO₃ (step 2). Add a few drops of K₂CrO₄ solution. Formation of a yellow/white precipitate confirms the presence of Ba.

2. To clean cell wells filled with the same solutions used in step 4, add a few drops of NaOH. Formation of a red-brown precipitate confirms the presence of Fe.

Confirmatory Tests for Group III

1. Fill clean cell wells with the cation solutions you used in step 3 that did not form a precipitate or smell of ammonia when reacted with NaOH.
in that step. You can distinguish between potassium and sodium salts by using the flame test.

Fill a clean cell well about half way with the contents of the HCl dropper. Using an alcohol burner, clean the nichrome flame-test wire by heating it to incandescence (i.e., until you see a bright red glow) in the hottest part of the flame. Carefully dip the heated wire into the cell well containing the hydrochloric acid, and then reheat the wire. Continue this treatment until no colour is imparted to the flame, then dip the tip of the clean wire into a cell well containing the fresh solution of the unknown cation. (Note that it may help if the tip of the wire has been fashioned into a loop, as in Figure B3.2, so that a small “bead” of sample forms at the end of the wire.) Heat the tip of the wire in the flame and watch for a characteristic colour to be (very briefly!) imparted to the flame. A yellow-orange colour indicates sodium and faint violet denotes potassium. Repeat if necessary.

![NICHROME WIRE](image)

a. heat wire to incandescence  
b. dip hot wire in concentrated HCl  
c. reheat  
d. transfer sample to wire  
e. heat in alcohol burner

**Figure B3.2**  
Flame-test procedure

**Identification of Unknown Cations**

From the tests performed in steps 1, 2 and 3 you should have learned which groups of cations might be present in each of your unknowns. Use the confirmatory tests to further determine the identity of the unknown cation in each of your two assigned unknown cation solutions. Be systematic, and be sure to record the code letters of the unknown samples. Record details of all the tests that you perform and the observations that you make.

**IMPORTANT: Remember to include your unknown codes in your report.**
Report Form

Microlab Lab B3 – Cations

Name: ___________________ ID#: __________________ Date: ____________

Complete the following table of observations. Note that you are not required to do all tests to complete the table; just the ones listed in the procedure.

<table>
<thead>
<tr>
<th>Cation</th>
<th>HCl</th>
<th>(NH₄)₂CO₃</th>
<th>NaOH</th>
<th>K₂CrO₄</th>
<th>Flame Test</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ag⁺</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ca²⁺</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ba²⁺</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fe³⁺</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mg²⁺</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NH₄⁺</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Na⁺</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>K⁺</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unknown</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unknown</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

State the “group” that your cation belongs to and identify the cation. Defend your answer with evidence.

Group I  Ag⁺
Group II Ca²⁺, Ba²⁺, Fe³⁺
Group III Mg²⁺, NH₄⁺, Na⁺, K⁺

<table>
<thead>
<tr>
<th>Unknown Code</th>
<th>Group Identity</th>
<th>Cation Identity</th>
<th>Evidence to Support Your Choice</th>
</tr>
</thead>
</table>

To confirm the presence of sodium or potassium ions it is necessary to use the flame test. Why is it not possible to precipitate these ions by reactions similar to those used to identify barium, silver, etc.?
Experiment B4: 
Quantitative Determination of Phosphorus

Prerequisite Skills

No prerequisite skills have been assigned to this laboratory.

Objectives

When you have completed the following experiment, you will have

1. learned to do a simple quantitative chemical analysis.
2. learned to use the mole concept and stoichiometry in a gravimetric analysis.

The experiment also illustrates

3. elementary gravity filtration using a fluted filter paper.
4. the concept of percentage yield.

Safety Tips

Experiment location. Work on a steady table in a safe secure place. Keep doors and windows closed to prevent drafts.

Handling of chemicals. All are common household chemicals, but should be treated with care.

Safety glasses should be worn at all times. Wear appropriate clothes and closed-toed shoes.
Introduction and Theory

To maintain life, plants, like animals, require essential nutrients. The three primary nutrients a plant requires are nitrogen, phosphorus and potassium. The source of these nutrients is the soil in which the plants are rooted. If the soil is lacking in these nutrients it can be supplied artificially through the use of fertilizers or plant food. Three numbers are usually displayed on the label of a plant food or fertilizer, indicating the percentages of:

- nitrogen (N)
- phosphorus (as P₂O₅)
- potassium (as K₂O)

If a product contains 15% N 30% P₂O₅ 15% K₂O it is labelled “15-30-15.”

Note that, by convention, P and K are quoted as P₂O₅ and K₂O even though there is no P₂O₅ or K₂O in the fertilizer. The phosphorus exists in the fertilizer as the phosphate anion (PO₄³⁻).

Gravimetric determination of the phosphate is achieved in this experiment by the precipitation and weighing of magnesium ammonium phosphate hexahydrate (MgNH₄PO₄·6H₂O). The precipitate forms upon slow neutralization with ammonia of an acidic solution of the phosphate-containing fertilizer.

\[
5\text{H}_2\text{O}(l) + \text{HPO}_4^{2-}(aq) + \text{NH}_4^+(aq) + \text{Mg}^{2+}(aq) + \text{OH}^-(aq) \rightarrow \text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}(s)
\]

Note that in acidic solution all the phosphate (PO₄³⁻) is converted to the hydrogen phosphate (HPO₄²⁻).

\[
\text{PO}_4^{3-}(aq) + \text{H}_3\text{O}^+(aq) \rightarrow \text{HPO}_4^{2-}(aq) + \text{H}_2\text{O}(l)
\]

The use of a strong base, such as NaOH, would cause the precipitation of Mg(OH)₂ and would therefore interfere with the analysis of the phosphorus.

Calculations†

1. Determination of the grams of phosphorus (P) in MgNH₄PO₄·6H₂O.

   \[\text{Grams of phosphorus (P) in } MgNH_4PO_4 \cdot 6H_2O = \text{grams } MgNH_4PO_4 \cdot 6H_2O \times \frac{Mwt (P)}{Mwt (MgNH_4PO_4 \cdot 6H_2O)}\]
2. Determination of the percent phosphorus (% P) in the plant food fertilizer. Converting from above grams of phosphorus (P) in MgNH₄PO₄·6H₂O.

Percent phosphorus in the plant food fertilizer

\[
\% P = \frac{\text{grams of phosphorous (P) in } MgNH_4PO_4 \cdot 6H_2O}{\text{grams of initial sample}} \times 100\%
\]

3. Determination of the percent P₂O₅ (%P₂O₅) in the plant food fertilizer. Converting from above % P.

To compare your % P result with the label on the plant food, you will need to convert % P to % P₂O₅.

\[
\% P_2O_5 = \frac{\% P \times Mwt(P_2O_5)}{2 \times Mwt \, P}
\]
Equipment and Material List

You are supplied with:

1 small funnel
1 125 mL Erlenmeyer flask (or 150 mL beaker)
1 150 mL beakers
1 50 mL graduated cylinder

4 filter papers
1 spatula
1 stir rod
1 vial (must be weighed out by the student)
   10-52-10 plant food (in excess of 1.2 g supplied)
   Epsom salts (MgSO₄ · 7 H₂O, in excess of 4.00 g supplied)
1 bottle of household ammonia (3%)
1 bottle rubbing alcohol (2-propanol)
1 general pan balance
Procedure (time to complete = 2 hours + overnight drying)

A. Fluted Filter Paper

You will need to fold your filter paper in a “fluted” manner to increase the surface area that is in contact with your filtrate in this experiment. The following instructions will show you how to flute your filter paper. It is essentially basic Origami for chemists.

1. Fold paper in half, then in half again and then in half again in the same direction. You should have a 1/8 section cone.

2. Unfold this cone twice so it looks like a semicircle.

3. Now try a “fan fold.” Alternately fold up and down every eighth section of the semicircle.

4. Open the fan until you get a fluted filter cone.
5. As a final touch, try to find the two opposing sections that are not folded correctly. Fold them inward to complete your perfect fan-folded filter paper.

Note: To make all creases, fold and press the paper. Do not run your finger or thumbnail along the folds. It may weaken the paper enough to introduce unwanted holes during filtration.

B. Phosphorus Analysis

1. Make up the solution of MgSO₄ · 7 H₂O. Weigh-out and transfer the 4 grams of MgSO₄ · 7 H₂O to the Erlenmeyer flask. Add 40 mL of tap water. Swirl the flask until the entire solid is dissolved.

2. Weigh-out 1.2 g of (10-52-10) plant food, and transfer the plant food to a clean beaker and add 20 mL of tap water. Swirl until the solid is dissolved.

3. If small, undissolved particles remain in the beaker, filter the solution into the other beaker using a fluted filter paper and the small funnel.

4. To this solution, add 35 mL of the (MgSO₄ · 7 H₂O) solution made up in step 1. Swirl well to mix.

5. Add 25 mL of ammonia to the solution. CAUTION: Do not breathe in the vapours! A fluffy white precipitate should form immediately. Swirl or stir the solution well.

6. Let the solution stand for at least 15 minutes. Cover the top so not as many fumes will enter into the room.

7. Filter off the precipitate using a clean filter and fresh filter paper, and wash it with two 5 mL aliquots of 2-propanol. Use more alcohol if the blue colour remains in the precipitate.

8. Let the precipitate stand in the filter for a few minutes to finish draining, then transfer the filter paper and precipitate to a pile of paper towels. To accelerate the drying process, carefully press the extra water out of the solid by using another filter paper to cover the solid, more paper towel to absorb the water and your hand to apply the force.

9. Uncover the solid on the filter paper and set it aside to dry at least overnight. Do not warm the precipitate to increase drying speed.
10. The next day, scrape the solid MgNH₄PO₄ · 6 H₂O off the filter paper and into the pre-weighed vial. Determine the weight of the vial + sample on a general pan balance provided.

11. Report the weight of the product (= wt. of vial + sample – wt. of vial).
Report Form

Microlab Lab B4 – Phosphorus Analysis

Name: __________________________ ID No.: ___________ Date: ______________

Observations (Solution colours? Undissolved particles? Precipitate?)

Complete the following table.

__________ Air-dry weight of MgNH₄PO₄ · 6 H₂O
__________ Molecular weight of MgNH₄PO₄ · 6 H₂O
__________ Grams of phosphorus in plant food
__________ % P in plant food
__________ % P₂O₅ equivalent to % P above
__________ % P₂O₅ in 10-52-10 plant food
__________ Theoretical weight of MgNH₄PO₄ · 6 H₂O

1. Compare your % P₂O₅ value with the theoretical value by calculating the percent error.
2. Now calculate the percent yield of MgNH₄PO₄ · 6 H₂O obtained in your experiment using the 10-52-10 label as a guide to determining the theoretical yield of MgNH₄PO₄ · 6 H₂O.

3. Why was ammonia used instead of sodium hydroxide to isolate MgNH₄PO₄ · 6 H₂O in this experiment?
Block C Experiment

Note: Students must have completed all of the previous experiments in this home lab manual before proceeding to Project C. A formal report is required for this project.
Project C:  
The Stoichiometry of a Reaction

Prerequisite Skills

To begin this experiment, you must have completed the required number of experiments from Blocks A and B. In addition, your tutor must have approved your proposed procedure.

Objectives

When you have finished the following experiment, you will have obtained further experience in the application of volumetric analysis.

Safety Tips

Safety glasses should be worn at all times. Wear appropriate clothes and closed-toed shoes.

Experiment location. Work on a steady table in a safe secure place. Keep doors and windows closed to prevent drafts.

Handling of chemicals. Potassium iodide and potassium iodate are both irritants. Avoid inhaling dust. Avoid skin contact. If skin contact occurs, wash the affected area immediately with water. Rinse any spillage off furniture and floors as soon as you can. Tend to a spill on your body first!
Introduction and Theory

In Experiment A3, you saw how an acid-base titration could be used to determine the acetylsalicylic acid content of commercially available Aspirin®. However, the use of titrations is not restricted to situations that involve acids and bases; another common type of titration involves so-called redox (oxidation-reduction) reactions. Although redox reactions are not studied in detail until Chemistry 218, you have already seen examples of such reactions in the laboratory and should be familiar with the concept of oxidation number (see your textbook).

Simply stated, a redox reaction is a chemical reaction in which two or more of the atoms or ions undergo a change in oxidation number. If the oxidation number of an atom or ion is increased, the atom or ion concerned is said to have been oxidized. On the other hand, if the oxidation number of an atom or ion is decreased, reduction has occurred. You should note that it is not possible to have a reaction in which only oxidation or reduction occurs. The two processes always occur simultaneously.

A simple example of a redox reaction is the single replacement of the copper(II) ion in copper(II) sulfate by zinc:

\[
\text{Zn (s)} + \text{CuSO}_4 (aq) \rightarrow \text{ZnSO}_4 (aq) + \text{Cu (s)}
\]

\[
\text{zinc} \quad \text{copper} \quad \text{zinc} \quad \text{copper}
\]

\[
\text{metal} \quad \text{sulfate} \quad \text{sulfate} \quad \text{metal}
\]

When this equation is written in net ionic form, it becomes apparent that copper(II) ions are reduced to copper metal, and that metallic zinc is oxidized to zinc ions.

oxidation number:

\[
\begin{align*}
0 & \quad +2 & \quad +2 & \quad 0 \\
\text{Zn (s)} & + \text{Cu}^{2+} (aq) & \rightarrow & \text{Zn}^{2+} (aq) & + & \text{Cu (s)} \\
\text{zinc} & \quad \text{copper} & \quad \text{zinc} & \quad \text{copper} \\
\text{metal} & \quad \text{ion} & \quad \text{ion} & \quad \text{metal}
\end{align*}
\]

In this experiment you will attempt to determine the stoichiometry (i.e., the balanced equation) for the redox reaction that occurs between iodate ions and iodide ions in acid solution. In other words, you will determine the values of \(a\), \(b\), \(c\), \(d\), and \(e\) in the following equation:

\[
a \text{IO}_3^- (aq) + b \text{H}^+ (aq) + c \text{I}^- (aq) \rightarrow d \text{I}_2 (aq) + e \text{H}_2\text{O} (l)
\]

\[
\text{iodate} \quad \text{hydronium} \quad \text{iodide} \quad \text{iodine} \quad \text{water}
\]

You will accomplish this by reacting a known amount of potassium iodate with excess acidic potassium iodide and determining the amount of molecular iodine produced (i.e. thereby first determining the ratio of \(d:a\)).
The latter will be found by titrating the iodine formed against a given solution containing 0.1000 N (normal) of sodium thiosulfate. The balanced equation for the reaction of sodium thiosulfate with molecular iodine is given below.

\[
2S_2O_3^{2-} \text{ (aq)} + I_2 \text{ (aq)} \rightarrow 2I^- \text{ (aq)} + S_4O_6^{2-} \text{ (aq)}
\]

**Note:** Molecular iodine (I$_2$ (s)) will react with iodide (I$^-$ (aq)) to form stable triiodide (I$_3^-$ (aq)) in aqueous solutions.
Chemical and Material Requirements

vial
spatula
KIO₃ (~0.321 g per student)-preweighed-out vial...use entire contents
100 mL volumetric flask
tap water
50 mL beaker
KI (supplied > 8.3 g per student)
(Student must weigh-out 8.3 g from the vial)
50 mL volumetric flask
10 mL burette + Teflon™ stopcock
retort stand and burette clamp
burette funnel
soapy water
0.1000N sodium thiosulfate in bottle (~60 mL per student)
1.0 M HCl (~20 mL per student)
125 mL Erlenmeyer flask
1 × 5 mL volumetric pipette
1 × 1 mL volumetric pipette
1 × 125 mL Erlenmeyer flask
starch indicator in solution dropper
meniscus reader

List of Reagents

<table>
<thead>
<tr>
<th>Compound</th>
<th>Name</th>
<th>Molecular Weight</th>
<th>Hazardous Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>tap H₂O</td>
<td>water, 55 M</td>
<td>18.02 g/mol</td>
<td>Scalds if hot!</td>
</tr>
<tr>
<td>*KI</td>
<td>potassium iodide, 1.0 M</td>
<td>166 g/mol</td>
<td>Moisture sensitive, irritant</td>
</tr>
<tr>
<td>*KIO₃</td>
<td>potassium iodate, 0.015 M</td>
<td>214 g/mol</td>
<td>Oxidizer, irritant</td>
</tr>
<tr>
<td>HCl</td>
<td>hydrochloric acid, 1.0 M (commercial preparation available)</td>
<td>36.5 g/mol</td>
<td>Corrosive, toxic</td>
</tr>
<tr>
<td>Na₂S₂O₃</td>
<td>sodium thiosulfate, 0.1000 N (commercial preparation available)</td>
<td>158.11 g/mol</td>
<td>Irritant</td>
</tr>
<tr>
<td></td>
<td>starch indicator</td>
<td></td>
<td>Highly toxic, irritant</td>
</tr>
</tbody>
</table>

* indicates solution must be made fresh on lab day
**Procedure** *(time to complete 3 hours)*

Use this laboratory manual, your laboratory notebook and the hints given below to determine the exact procedure that should be followed and the type of results expected. Submit your proposed procedure and results table to your tutor before you intend to perform the experiment.

The following information may be of use to you:

1. You will need to prepare solutions of approximately 0.015 mol·L\(^{-1}\) potassium iodate (KIO\(_3\)), and 1 mol·L\(^{-1}\) potassium iodide (KI). Calculate the amounts you need to use to prepare sufficient quantities of the solutions for your own use in the lab. You will have to note the exact concentrations of the solutions you make. Make sure you check your calculations with your tutor before proceeding.

2. The acidic potassium iodide should be prepared by mixing about 3 mL of potassium iodide (KI) solution with an equal volume of 1 mol·L\(^{-1}\) hydrochloric acid. A 6 mL aliquot of the potassium iodate solution (KIO\(_3\)) should be added to this mixture, which should then be titrated against the standard sodium thiosulfate. Hint: You’ll need to make enough of each reagent in the appropriate sized volumetric flask for a least three trials.

3. A large excess of iodide is necessary because the solid iodine that is initially produced in the reaction reacts with iodide ion to produce the soluble triiodide ion:

   \[ I_2 \text{ (s)} + I^- \text{ (aq)} \rightarrow I_3^- \text{ (aq)} \]

   iodine  iodide  triiodide

4. As the iodine/thiosulfate titration proceeds, the colour of the solution changes from its initial red-brown, through yellow, to colourless. However, the colour change from yellow to colourless is not sharp enough to be used for determining the end-point. Instead, starch indicator is used. This indicator gives an intense blue colour when iodine is present and turns colourless once all the iodine has reacted with the thiosulfate:

   \[ I_2 \text{ (s)} + \text{starch} \rightarrow \text{starch/I}_2 \text{ complex} \]

   iodine  (intense blue/black colour)

The change from blue to colourless is very quick and easy to observe, so that this titration has the potential to be very accurate. Note that the starch indicator should not be added to the solution being titrated until the end-point has nearly been reached (i.e., when the solution has a pale yellow colour).
Figure C.1
Apparatus for redox titration

WASTE DISPOSAL: Waste materials from this experiment may be washed down the drain with copious amounts of water.
Results, Calculations and Write-Up

A formal report is required for this experiment.
Questions

1. High altitude balloons used by Environment Canada to measure O₃ levels in the atmosphere also use a similar idiometric titration where the iodine liberated is measured by back titration with sodium thiosulfate. In this case the iodine is generated by oxidation of iodide by ozone:

\[
\text{O}_3 (\text{g}) + 2\text{I}^- (\text{aq}) + 2\text{H}^+ (\text{aq}) \rightarrow \text{I}_2 (\text{aq}) + \text{O}_2 (\text{g}) + \text{H}_2\text{O} (\text{l})
\]

If 10,000 L of air were bubbled through an acidic iodide solution (at STP) and was then back titrated with 19.4 mL of a $1.2 \times 10^{-4}$ M Na₂S₂O₃ solution to reach equivalence, what is the concentration of ozone in that sample of air (in ppm)?
Procedure

A. Preparation of Stock Solutions and Reagents

1. 0.015 M Potassium iodate (KIO₃) stock solution was prepared by…
2. 1.0 M Potassium iodide (KI) stock solution was prepared by…
3. 1M Hydrochloric acid (HCl) solution
   Note: This solution was commercially manufactured and supplied.
4. 0.1000N Sodium thiosulfate (Na₂S₂O₃) titration standard
   Note: This solution was commercially manufactured and supplied.
5. Starch Indicator solution
   Note: This solution was commercially manufactured and supplied.

B. Preparation of the Reaction Mixture

1. To prepare the acidic KI solution, use a _____ volumetric pipette to transfer _____ mL of the 1.0 M KI stock solution into an empty container. Using the same _____ pipette, transfer _____ mL of the 1.0 M HCl solution into the container containing the 1.0 M KI, and mix well.
2. Transfer 6 mL of the mixed acidic potassium iodide (KI + HCl) solution into a clean reaction vessel (e.g., 125 mL Erlenmeyer flask).
3. Using clean 1- and 5-mL volumetric pipettes, 6 mL of the 0.015 M KIO₃ stock solution was transferred into the reaction vessel.

C. Titration

1. The procedure used for titration in Experiment A3 were followed, except for the following modifications:
Results:

Sample Calculations:
Appendix
## Table of Reagents

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Chemical Formula</th>
<th>Experiment #</th>
<th>Solid (S) or Liquid (L)</th>
<th>Formula Weight</th>
<th>MP or BP (°C)</th>
<th>Density (g/mL)</th>
<th>Hazardous Properties*</th>
</tr>
</thead>
<tbody>
<tr>
<td>acetic acid, 5% (vinegar)</td>
<td>CH$_3$CO$_2$H</td>
<td>B1</td>
<td>L</td>
<td>60.05</td>
<td></td>
<td></td>
<td>Corrosive</td>
</tr>
<tr>
<td>acetylsalicylic acid (Aspirin®)</td>
<td>CH$_3$CO$_2$C$_6$H$_4$CO$_2$H</td>
<td>A2, A3</td>
<td>S</td>
<td>180.16</td>
<td>138–140</td>
<td></td>
<td>Irritant, toxic</td>
</tr>
<tr>
<td>ammonia, 3% solution</td>
<td>NH$_4$OH</td>
<td>B1, A3</td>
<td>L</td>
<td>35.05</td>
<td>0.90</td>
<td></td>
<td>Corrosive, lachrymator</td>
</tr>
<tr>
<td>ammonium carbonate</td>
<td>(NH$_4$)$_2$CO$_3$</td>
<td>B3</td>
<td>S</td>
<td>114.10</td>
<td>d58</td>
<td></td>
<td>Irritant</td>
</tr>
<tr>
<td>ammonium carbonate, 1.0 M</td>
<td>(NH$_4$)$_2$CO$_3$</td>
<td>B3</td>
<td>L</td>
<td>114.10</td>
<td></td>
<td></td>
<td>Irritant</td>
</tr>
<tr>
<td>ammonium nitrate</td>
<td>NH$_4$NO$_3$</td>
<td>B3</td>
<td>S</td>
<td>80.04</td>
<td>169.6</td>
<td>1.720</td>
<td>Oxidizer, irritant</td>
</tr>
<tr>
<td>ammonium nitrate, 1.0 M</td>
<td>NH$_4$NO$_3$</td>
<td>B3</td>
<td>L</td>
<td>80.04</td>
<td></td>
<td></td>
<td>Oxidizer, irritant</td>
</tr>
<tr>
<td>Aspirin®</td>
<td>CH$_3$CO$_2$C$_6$H$_4$CO$_2$H</td>
<td>A2, A3</td>
<td>S</td>
<td>(see also salicylic acid, acetate ester, or acetylsalicylic acid)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>barium nitrate</td>
<td>Ba(NO$_3$)$_2$</td>
<td>B3</td>
<td>S</td>
<td>261.35</td>
<td>592</td>
<td>3.240</td>
<td>Oxidizer, toxic</td>
</tr>
<tr>
<td>barium nitrate, sat. sol’n. ~1.0 M</td>
<td>Ba(NO$_3$)$_2$</td>
<td>B3</td>
<td>L</td>
<td>261.35</td>
<td></td>
<td></td>
<td>Oxidizer, corrosive</td>
</tr>
<tr>
<td>bleach, 5.25%</td>
<td>NaOCl</td>
<td>B1</td>
<td>L</td>
<td>74.44</td>
<td></td>
<td>1.097</td>
<td>Oxidizer, corrosive</td>
</tr>
<tr>
<td>calcium nitrate</td>
<td>Ca(NO$_3$)$_2$ 4H$_2$O</td>
<td>B3</td>
<td>S</td>
<td>236.15</td>
<td>44</td>
<td>1.860</td>
<td>Oxidizer, irritant</td>
</tr>
<tr>
<td>calcium nitrate, 1.0 M</td>
<td>Ca(NO$_3$)$_2$</td>
<td>B3</td>
<td>L</td>
<td>236.15</td>
<td></td>
<td></td>
<td>Oxidizer, irritant</td>
</tr>
<tr>
<td>carbon powder</td>
<td>C</td>
<td>A1</td>
<td>S</td>
<td>12.01</td>
<td></td>
<td></td>
<td>Irritant</td>
</tr>
<tr>
<td>coffee, instant powder</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>AWF taste</td>
</tr>
<tr>
<td>corn syrup</td>
<td>fructose+glucose</td>
<td>B1</td>
<td>L</td>
<td></td>
<td></td>
<td></td>
<td>Sticky</td>
</tr>
<tr>
<td>cresol red indicator</td>
<td></td>
<td>A2</td>
<td></td>
<td>382.43</td>
<td></td>
<td></td>
<td>Toxic, irritant</td>
</tr>
<tr>
<td>ethanol, 95%</td>
<td>CH$_3$CH$_2$OH</td>
<td>B3</td>
<td>L</td>
<td>46.07</td>
<td>78.5</td>
<td>0.785</td>
<td>Flammable, poison</td>
</tr>
<tr>
<td>hydrochloric acid, 0.1 M</td>
<td>HCl</td>
<td>A3</td>
<td>L</td>
<td>36.46</td>
<td></td>
<td></td>
<td>Corrosive, highly toxic</td>
</tr>
<tr>
<td>hydrochloric acid, 1.0 M</td>
<td>HCl</td>
<td>A3,B2–B3,C</td>
<td>L</td>
<td>36.46</td>
<td></td>
<td></td>
<td>Corrosive, highly toxic</td>
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<tr>
<td>iodine</td>
<td>I$_2$</td>
<td>C</td>
<td>S</td>
<td>253.81</td>
<td>113</td>
<td>4.930</td>
<td>Toxic, corrosive</td>
</tr>
<tr>
<td>iron (III) chloride hexahydrate</td>
<td>FeCl$_3$·6H$_2$O</td>
<td>A2</td>
<td>S</td>
<td>270.30</td>
<td>37</td>
<td>1.820</td>
<td>Corrosive, hygroscopic</td>
</tr>
<tr>
<td>iron (III) chloride, 0.02 M</td>
<td>FeCl$_3$</td>
<td>A2</td>
<td>L</td>
<td>270.30</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>iron (III) nitrate</td>
<td>Fe(NO$_3$)$_3$·9H$_2$O</td>
<td>B3</td>
<td>S</td>
<td>404.00</td>
<td>47</td>
<td>1.680</td>
<td>Oxidizer, irritant</td>
</tr>
<tr>
<td>iron (III) nitrate, 1.0 M</td>
<td>Fe(NO$_3$)$_3$·9H$_2$O</td>
<td>B3</td>
<td>L</td>
<td>404.00</td>
<td></td>
<td></td>
<td>Oxidizer, irritant</td>
</tr>
<tr>
<td>magnesium, metal</td>
<td>Mg</td>
<td>B2</td>
<td>S</td>
<td>24.31</td>
<td>648</td>
<td>1.740</td>
<td>Flamm. solid, moist.sens.</td>
</tr>
<tr>
<td>magnesium nitrate hexahydrate</td>
<td>Mg(NO$_3$)$_2$·6H$_2$O</td>
<td>B3</td>
<td>S</td>
<td>256.41</td>
<td>89</td>
<td>1.636</td>
<td>Oxidizer, irritant</td>
</tr>
<tr>
<td>magnesium nitrate, 1.0 M</td>
<td>Mg(NO$_3$)$_2$</td>
<td>B3</td>
<td>L</td>
<td>256.41</td>
<td></td>
<td></td>
<td>Oxidizer, irritant</td>
</tr>
<tr>
<td>magnesium oxide</td>
<td>MgO</td>
<td>B2</td>
<td>S</td>
<td>40.31</td>
<td>2826</td>
<td>3.580</td>
<td>Moisture sensitive</td>
</tr>
<tr>
<td>magnesium sulfate, 7-hydrate</td>
<td>MgSO$_4$·7H$_2$O</td>
<td>B4</td>
<td>S</td>
<td>246.48</td>
<td></td>
<td>1.670</td>
<td>(epson salt)</td>
</tr>
<tr>
<td>phenolphthalein indicator</td>
<td></td>
<td>A3</td>
<td></td>
<td>318.33</td>
<td>263</td>
<td></td>
<td>Toxic, irritant</td>
</tr>
<tr>
<td>plant food, fertilizer</td>
<td></td>
<td>B4</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Toxic, irritant</td>
</tr>
<tr>
<td>potassium chromate, 1.0 M</td>
<td>K$_2$Cr$_2$O$_7$</td>
<td>B3</td>
<td>L</td>
<td>194.20</td>
<td></td>
<td></td>
<td>Canc. susp. agent, oxidizer</td>
</tr>
<tr>
<td>potassium iodate</td>
<td>KIo$_3$</td>
<td>C</td>
<td>S</td>
<td>214.0</td>
<td>560</td>
<td>3.930</td>
<td>Oxidizer, irritant</td>
</tr>
<tr>
<td>potassium iodide</td>
<td>KI</td>
<td>C</td>
<td>S</td>
<td>166.01</td>
<td>681</td>
<td>3.130</td>
<td>Moist. sens., irritant</td>
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<tr>
<td>potassium nitrate</td>
<td>KNO$_3$</td>
<td>B3</td>
<td>S</td>
<td>101.11</td>
<td>334</td>
<td>2.109</td>
<td>Oxidizer, irritant</td>
</tr>
<tr>
<td>potassium nitrate, 1.0 M</td>
<td>KNO$_3$</td>
<td>B3</td>
<td>L</td>
<td>101.11</td>
<td></td>
<td></td>
<td>Oxidizer, irritant</td>
</tr>
<tr>
<td>2-propanol</td>
<td>(CH$_3$)$_2$CHOH</td>
<td>B4</td>
<td>L</td>
<td>60.11</td>
<td>82.4</td>
<td>0.7855</td>
<td>Flammable, irritant</td>
</tr>
<tr>
<td>salicylic acid, acetate ester</td>
<td>CH$_3$CO$_2$C$_6$H$_4$CO$_2$H</td>
<td>A2–A3</td>
<td>S</td>
<td>180.16</td>
<td>138–140</td>
<td></td>
<td>Irritant, toxic</td>
</tr>
<tr>
<td>salt, rock</td>
<td>NaCl</td>
<td>B1</td>
<td>S</td>
<td></td>
<td></td>
<td></td>
<td>See sodium chloride</td>
</tr>
<tr>
<td>salt, table</td>
<td>NaCl</td>
<td>B1</td>
<td>S</td>
<td></td>
<td></td>
<td></td>
<td>See sodium chloride</td>
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<table>
<thead>
<tr>
<th>Compound Name</th>
<th>Chemical Formula</th>
<th>Experiment #</th>
<th>Solid (S) or Liquid (L)</th>
<th>Formula Weight</th>
<th>MP or BP (°C)</th>
<th>Density (g/mL)</th>
<th>Hazardous Properties*</th>
</tr>
</thead>
<tbody>
<tr>
<td>silver nitrate</td>
<td>AgNO₃</td>
<td>B3</td>
<td>S</td>
<td>169.88</td>
<td>212</td>
<td>4.352</td>
<td>Highly toxic, oxidizer</td>
</tr>
<tr>
<td>silver nitrate, 1.0 M</td>
<td>AgNO₃</td>
<td>B3</td>
<td>L</td>
<td>169.88</td>
<td></td>
<td></td>
<td>Highly toxic, oxidizer</td>
</tr>
<tr>
<td>sodium chloride</td>
<td>NaCl</td>
<td>B1</td>
<td>S</td>
<td>58.44</td>
<td>801</td>
<td>2.165</td>
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<tr>
<td>sodium hydroxide solid (19.1 M)</td>
<td>NaOH</td>
<td>A2, B3</td>
<td></td>
<td>40.00</td>
<td>318.4</td>
<td>1.53</td>
<td>Corrosive, toxic</td>
</tr>
<tr>
<td>sodium hydroxide, 1.0 M</td>
<td>NaOH</td>
<td>A2, B3</td>
<td>L</td>
<td>40.00</td>
<td></td>
<td></td>
<td>Corrosive, toxic</td>
</tr>
<tr>
<td>sodium nitrate</td>
<td>NaNO₃</td>
<td>B3</td>
<td>S</td>
<td>84.99</td>
<td>306</td>
<td>2.260</td>
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</tr>
<tr>
<td>sodium nitrate, 1.0 M</td>
<td>NaNO₃</td>
<td>B3</td>
<td>L</td>
<td>84.99</td>
<td></td>
<td></td>
<td>Oxidizer, toxic</td>
</tr>
<tr>
<td>sodium nitrite</td>
<td>NaNO₂</td>
<td>A4</td>
<td>S</td>
<td>69.00</td>
<td>271</td>
<td>2.168</td>
<td>Oxidizer, toxic</td>
</tr>
<tr>
<td>sodium thiosulfate stand. (0.1N)</td>
<td>Na₂S₂O₃</td>
<td>C</td>
<td>L</td>
<td></td>
<td></td>
<td></td>
<td>Irritant</td>
</tr>
<tr>
<td>starch indicator</td>
<td>(C₆H₁₀O₅)n</td>
<td>C</td>
<td>L</td>
<td></td>
<td></td>
<td></td>
<td>Highly toxic, irritant</td>
</tr>
<tr>
<td>sucrose</td>
<td>C₁₂H₂₂O₁₁</td>
<td>B1</td>
<td>S</td>
<td>342.30</td>
<td>185–6</td>
<td>1.5805</td>
<td>Tooth decay</td>
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<tr>
<td>sugar</td>
<td>C₁₂H₂₂O₁₁</td>
<td>B1</td>
<td>S</td>
<td>97.09</td>
<td>d200</td>
<td>2.126</td>
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</tr>
<tr>
<td>sulfamic acid</td>
<td>HSO₃NH₂</td>
<td>A4</td>
<td>S</td>
<td></td>
<td></td>
<td></td>
<td>See sucrose</td>
</tr>
<tr>
<td>tin</td>
<td>Sn</td>
<td>A1</td>
<td>S</td>
<td>118.69</td>
<td>231.9</td>
<td>7.310</td>
<td>Flamm. solid, moist. sens.</td>
</tr>
<tr>
<td>vinegar</td>
<td>CH₃CO₂H</td>
<td>B1</td>
<td>L</td>
<td></td>
<td></td>
<td></td>
<td>See acetic acid</td>
</tr>
<tr>
<td>water</td>
<td>H₂O</td>
<td>all</td>
<td>L</td>
<td>18.02</td>
<td>100</td>
<td>1.00</td>
<td>Will burn skin when hot</td>
</tr>
</tbody>
</table>

Glossary

absorb to take up a substance in bulk.

absorbance the common logarithm of the reciprocal of the transmittance of a pure solvent, a.k.a absorbency

absorbency penetration of one substance into another.

absorption the taking up of matter in bulk by other matter, as in dissolving of a gas by a liquid or photons of light by a sample.

absorption spectrum the amount of light absorbed by a sample as a function of wavelength.

acid (or base) acid (or base) alone normally means Brønsted-Lowry acid (or Brønsted-Lowry base) see below.

actual yield the mass or moles of product obtained in a reaction.

adsorb to hold gas, liquid or solid to its surface.

adsorption the surface retention of solid, liquid, or gas molecules, atoms, or ions by a solid or liquid (as opposed to absorption, which is the penetration of substances into the bulk of the solid or liquid).

amphiprotic (from Greek, amphi = both) a substance that can act as both a proton donor and proton acceptor (e.g., water)

Arrhenius acid is a molecule or polyatomic ion that contains hydrogen and reacts with water to produce hydronium ions.

Arrhenius base is a molecule or ion that produces hydroxide ions in water.

atmospheric pressure is the pressure exerted on the surface of the earth by the atmosphere.

Avogadro’s law states “equal volumes of any two gases at the same temperature and pressure that contain the same number of molecules,” (i.e., a mole of any gas should occupy the same volume as a mole of any other gas. At STP, this volume is $22.414 \text{ L} = \text{STP molar volume of gas}$.

Avogadro’s number is the number of atoms or molecules in a mole = $6.022 \times 10^{23}$ atoms or molecules/mole.

barometric pressure atmospheric pressure as determined by a barometer. First barometer was developed by Evangelista Torricelli, a pupil of Galileo.

Boyle’s law $V = k/P$, describes the quantitative relationship between volume and pressure and was first summarized by Robert Boyle in 1662.

Brønsted-Lowry acid is a proton donor ($\text{H}^+$).

Brønsted-Lowry base is a proton acceptor ($\text{H}^+$).

chelating agents (from Greek, chele = claw) are polydentate ligands (“many toothed” ligands) which complex with a metal using two or more donor atoms. e.g.,
ethylenediaminetetraacetate ion (EDTA).

**complex ions** are metal ions surrounded by a group of anions or neutral molecules (ligands). It is an electrostatic interaction between the positive metal cation and the surrounding negative ions or dipoles of neutral compounds oriented with their negative ends toward the metal ion.

**complementary colours** when a sample absorbs light (400 nm–700 nm) we see the remaining colours that strike our eyes. In the case of complementary colours, when a sample absorbs only one colour we perceive the sum of all the other colours as its complimentary, i.e., if a sample absorbs only blue, we perceive orange; if red is absorbed, we see green; and if yellow is absorbed, we see purple (and vice versa for all respectively).

**coordination sphere** the central metal ion and the ligands bound to it constitute the coordination sphere.

**crystallization** a process where a solute comes out of solution as a crystal(s).

**Dalton’s law of partial pressures** states the total pressure of a mixture of gases is equal to the sum of the partial pressures of the component gases.

\[ P_{\text{total}} = p_1 + p_2 + p_3 + \ldots \]

**donor atom** the atom of the ligand bound directly to the metal. e.g., in \([\text{Ag(NH}_3\text{)}_2]^+\), nitrogen is the donor atom. In \([\text{Fe(H}_2\text{O)}_6]^3\), oxygen is the donor atom.

**end point** is a stage in a titration at which enough titrant has been added to bring the indicator to a colour half way between its initial and final colours.

**equivalence point** (a.k.a stoichiometric point), the stage in a titration when exactly the right volume of titrant has been added to complete the reaction. Usually estimated from the “end point.”

**gas** (from Greek, *chaos*), named coined by Johann van Helmont, is a fundamental state of matter.

**Ideal Gas Law** By combining Boyle’s, Charles’s, and Avogadro’s law, it is possible to relate temperature, pressure and volume via \(PV = nRT\). It is a limiting law and is most accurate as pressure approaches zero.

**kinetic theory** states that (1) gases consist of molecules widely separated by space, (2) gas molecules are in constant and random motion, (3) gas molecules translate heat into kinetic energy, collisions between gas molecules are elastic, (4) attractive or repulsive forces are negligible between molecules of an ideal gas, and (5) the average kinetic energy of the molecules is proportional to the absolute temperature.

**ligands** (from Latin, *ligare* = to bind) the molecules or ions that surround a metal ion in a complex (a.k.a complexing agents; complexed to or coordinated to the metal). Normally either ions or polar molecules and have at least one pair of unshared valence electrons. They function as Lewis bases (electron pair donors). May be monodentate or polynedentate (“many toothed” ligands a.k.a chelating agents are bidentate, tridentate etc.).
ligand exchange reaction or substitution reactions are reactions where one ligand replaces another in the coordination sphere of a metal ion. Some ligand-exchange reactions are fast (labile complexes) and others are slow (inert complexes).

limiting reagent is the reactant that governs the theoretical yield of product in a given reaction.

neutralization is a reaction between an acid and a base. The products of the reaction are a salt (ionic compound) and water.

“over water” when gas is collected in a column filled initially with water.

oxidation refers to combination with oxygen, or a reaction in which an atom, ion or molecule loses an electron, or a reaction in which the oxidation number of an element is increased, e.g., \( \text{Mg} (s) + \frac{1}{2} \text{O}_2 (g) \rightarrow \text{MgO} (s) \)

oxidation number is the effective charge on an atom in a compound. Note that they are fictitious charges in the case of covalent species. They are calculated according to a set of rules (see below). An increase in oxidation number means an oxidation has occurred. A decrease in oxidation number means a reduction has taken place.

Rules for Assigning Oxidation Numbers:

1. Any uncombined atom (an element in its elemental form) or any atom in a molecule of an element is assigned an oxidation number of 0, e.g., in \( \text{He} (g) \) \( \text{He} = 0 \), in \( \text{Cl}_2 (g) \) each \( \text{Cl} = 0 \).

2. The sum of the oxidation numbers of the atoms in an electrically neutral compound is 0, e.g., in \( \text{NaCl} (s) \) \( \text{Na} = 1^+ \) and \( \text{Cl} = 1^- \).

3. Monatomic ions have the same charge as that on the ion (its electrovalence number). Polyatomic ions have a charge equal to that of the ion and this must equal the sum of the oxidation numbers of the atoms that make up the polyatomic ion. For example, in \( \text{SO}_4^{2-} \), the charge is 2\(^-\), therefore the sum of the charges on the S and all the Os must also = 2\(^-\). Because each O is 2\(^-\), there is a 8\(^-\) charge on all the Os, thus S must be 6\(^+\). (8\(^-\)) + (6\(^+\)) = 2\(^-\).

4. Fluorine (most electronegative element) is 1\(^-\) in all fluorine-containing compounds.

5. In most oxygen containing compounds, the oxidation number of O is 2\(^-\). Exceptions are:
   - in peroxides \( \text{O}_2^2^- \), since each O is equivalent, each O is 1\(^-\).
   - in superoxide ion \( \text{O}_2^- \), each O is 1/2\(^-\).
   - in \( \text{OF}_2 \), the O is 2\(^+\) (see rule 4 above).

6. The oxidation number for H is 1\(^+\) in all its compounds, except the metal hydrides in (e.g. CaH\(_2\) and NaH), where all the H are 1\(^-\).

7. The halogens (except fluorine) have an oxidation number of 1\(^-\) unless the halogen is in combination with oxygen or another halogen higher
in the group.

**oxidation state**

It is the actual condition of a species with a specified oxidation number, e.g., \( \text{Mg}^{2+} \) is an oxidation state of Mg with an oxidation number of 2.

**Pascal**

Standard unit of pressure in SI units = 1 N/m\(^2\), named after Blaise Pascal (1623–1662), a French mathematician and scientist.

**percentage yield**

Is the mass or moles of product obtained divided by the theoretical yield in mass or moles times 100%.

**pH**

Is equal to the negative logarithm (base 10) of the concentration of the hydronium ion or \( \text{pH} = -\log_{10}[\text{H}_3\text{O}^+] \).

**pH indicator**

A substance that changes colour when it goes from its acidic form to its basic form.

**Pipette fillers**

For your information, the 3 valve bulb-type pipette filler, and the pi-pump 2500 are also described below. In the event that you go on to take a supervised lab in the future, you are likely to encounter these models of pipette fillers.

**3-valve bulb type**

Notice that there are three valves on the pipette filler, labelled A, E, and S (see Figure G1.6). The purposes of these three valves are:

- **A** (air) — this valve enables you to expel air from the pipette filler
- **E** (empty) — this valve allows air back into the bulb, which in turn causes the liquid to drain out of the pipette
- **S** (suck) — this valve enables you to draw liquid into the pipette.

![Figure G1.6](image)

Pipette filler, with 3 valves

**Safety Note:** It is important that you learn how to use the pipette filler correctly as you will be using it frequently throughout the course. **Never try to suck liquid into a pipette by mouth** as you could (a) cut your mouth on a chipped pipette, (b) transfer a harmful substance from the outside of the pipette into your mouth, (c) suck toxic or corrosive liquid into your mouth, or (d) have some kind of mishap with the pipette penetrating the roof of your mouth.

Press valve **A** using your thumb and forefinger, and, with the other hand, squeeze the bulb so that air is expelled. **Gently** and carefully insert the top of the pipette into the pipette filler, as shown in Figure G1.7. Grip the pipette just below the top of the pipette as you insert it into the pipette.
filler. “Shortening your grip” will prevent a serious accident should the pipette snap as you push it into the pipette filler.

Figure G1.7
Pipette and filler

Place the tip of the pipette into the liquid to be measured out, and carefully squeeze valve S so that the liquid is slowly drawn up into the pipette. Take care to keep the tip of the pipette below the surface of the liquid, otherwise air will be drawn into the pipette. Continue to squeeze valve S until the level of the liquid is just above the graduation mark on the pipette. Take it easy. Do not suck the liquid into the pipette filler. Hold the pipette over a second beaker that you will use for temporary storage of waste materials, and squeeze valve E until the level of liquid in the pipette reaches the graduation mark. The bottom of the meniscus should be level with the mark. Keep the graduation mark at eye level when you are doing this (see Figure A1.5).

Note: If you permit the liquid to fall below the level of the graduation mark, you must draw in some more liquid, and repeat the above procedure until you get it right.

Make sure that there are no drips adhering to the tip of the pipette, place the tip of the pipette inside your pre-weighed 50 mL beaker, and allow the water to drain into the beaker. You will notice that a few drops of liquid will not drain out. Place the tip of the pipette against the side of the beaker and hold it there for 15 seconds. There will still be a small amount of liquid left in the pipette. Do not attempt to blow this liquid out—it should be left in the pipette. When the manufacturer calibrates the pipette, the calibration is carried out with the knowledge that, when the specified volume (in this case, 5 mL) has run out of the pipette, this small amount of liquid will remain behind. This is why your pipette is marked TD (to deliver) 5 mL. It delivers this volume, but when it is filled to the graduation mark it actually contains a little bit more.

In order to use the pi-pump 2500 pipette filler (see Figure G1.8), the filler is first carefully attached to the pipette. The tip of the pipette is then placed in the liquid to be measured out, and the wheel (1 in Figure G1.8) is rotated in a clockwise direction until the level of liquid in the pipette has risen to just above the calibration mark. Hold the pipette over a second beaker that you will use for temporary storage of waste materials, and rotate the wheel counter-clockwise until the level of liquid in the pipette descends to the graduation mark. The bottom of the meniscus should be level with the mark. Keep the graduation mark at eye level when you are doing this (see
Note: If you permit the liquid to fall below the level of the graduation mark, you must draw in some more liquid, and repeat the above procedure until you get it right.

Make sure that there are no drips adhering to the tip of the pipette, place the tip of the pipette inside your pre-weighed 50 mL beaker and allow the water to drain into the beaker by pressing the release bar (2 in Figure G1.8). You will notice that a few drops of liquid will not drain out of the pipette. Place the tip of the pipette against the side of the beaker and hold it there for 15 seconds. There will still be a small amount of liquid left in the pipette. Do not attempt to blow this liquid out—it should be left in the pipette. When the manufacturer calibrates the pipette, the calibration is carried out with the knowledge that, when the specified volume (in this case, 5 mL) has run out of the pipette, this small amount of liquid will remain behind. This is why your pipette is marked TD (to deliver) 5 mL. It delivers this volume, but when it is filled to the graduation mark it actually contains a little bit more. When using the pi-pump 2500, resist the temptation of using the plunger (3 in Figure G1.8) to force liquid out of the pipette. Use of the plunger can result in all of the liquid being forced out of the pipette, thus the volume delivered will be greater than is required.

**Figure G1.8**
A pi-pump 2500 pipette filler

**precipitation**
A process where an insoluble solid product is produced and comes rapidly out of solution as a finely divided powder called a precipitate.

**pressure**
Is defined as force per unit area, e.g., 1 Pa = 1 N/m²

**reduction**
Refers to removal of oxygen or addition of hydrogen. Reaction in which an atom, ion or molecule gains an electron or a reaction in which the oxidation number of an element is decreased, e.g., Cl₂ (g) → 2Cl⁻ (aq)

**redox reaction**
Is a type of reaction where both oxidation and reduction occur, e.g., S (s) + 3 F₂ (g) → SF₆ (g)

**standard atmosphere**
Or (atm) is the atmospheric pressure as measured at sea level at 0°C. A column of Hg at atm is 760 mm in height.

**stoichiometric coefficients**
Are the numbers multiplying chemical formulas in chemical equations. Note: if the coefficient is 1, it is not indicated in the equation, e.g., 1 H₂ + 1 Br₂ → 2 HBr = H₂ + Br₂ → 2 HBr

**stoichiometry**
Refers to the quantitative relation between the amounts of reactants consumed and products formed in chemical reactions as expressed by the
### Appendix

- **(reaction)** balanced chemical equation for the reaction.
- **STP** standard temperature and pressure is 0°C (273°K) and 1 atm pressure.
- **strong acid (or base)** is almost completely ionized in solution.
- **theoretical yield** the maximum mass or moles of product expected from a reaction, taking into account the stoichiometry of the reaction and the number of moles of the limiting reagent used in the reaction.
- **universal gas constant** \( R \) is the constant that appears in the ideal gas law: \( R = 8.31451 \text{ J} \times \text{K}^{-1} \times \text{mol}^{-1} \)
- **vacuum filtration** or suction filtration, is a common method of collecting crystalline product. (see also recrystallization). It involves the use of a Büchner funnel, filter paper, filter flask and water aspirator (with water trap).

![Diagram of vacuum filtration](image)

- **water aspirator** a small device attached to a water faucet. It is used to create an inexpensive source of vacuum for use in vacuum filtrations.
weak acid (or base) is incompletely ionized in solution.
How to Return my Chemistry 217 Home Lab Kit

When you are finished with the CHEM217 lab kit and have successfully completed all the required experiments, follow the instructions below to return your Chemistry 217 lab kit to Athabasca University.

Use the checklist (pages 46-48 in the lab manual) to ensure all items are put back in the lab kit container (blue box) and returned to Athabasca University:

**Athabasca University**  
**Science Lab**  
1 University Drive  
Athabasca, AB  
T9S 3A3  
(780) 675-6276

![Figure 1. Empty Kit](image)

Return all glassware to the foam cut-out and place in the bottom of the box.

**Fig.2. Glassware in Styrofoam cutout**  
**Fig.3. Glassware in first**

Return the balance, power supply and 200g calibration mass to its rigid foam cut-out and place on top of the re-packaged glassware.

**Fig.4. Balance replaced in Styrofoam**  
**Fig. 5. Balance in second**
Place the calorimetric vial, alcohol lamp, one litre plastic beaker, spectrophotometer and multimeter, disassembled stand/clamp, etc. around the box containing the balance. Fig. 6

Return all empty vials and bottles (discard used droppers) to re-sealable bags and place on top of the balance box. Fig. 7

Fig. 6. Placement of other items  Fig. 7. Return of empties

Please use DHL Courier service to return your lab kit. You have been supplied with a pre-addressed DHL waybill Fig. 8 to simplify the return of your kit. Ensure that the return address to Athabasca University is complete and that the following boxes are checked: “Ground”, “Collect” and the “Receiver Account No.” is entered.

Please use the supplied plastic cable tie (taped inside of box lid) to secure the lid of the box for return shipment. Simply peel and stick the last section of the waybill to the outside of the closed and secured lab kit Fig. 9.

Fig. 8. Pre-Addressed DHL Waybill
Retain the top portion of the waybill as your proof of shipment. The return shipment of the kit is paid collect by Athabasca University. When ready to return your kit contact DHL Courier at 1-800-CALL-DHL to arrange a time for them to pick up your kit.

Fig. 9. Attachment of waybill

If you have any questions regarding the lab kit return process as described above, please feel free to contact Mr. Neil Sexton, Kit Manager/Lab Assistant at Athabasca University Centre for Science at neils@athabascau.ca or 1-800-788-9041 ext. 6277.
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