INTRODUCTION
Establish a scientific question and understand how to answer it experimentally
(see lab projects for general considerations)

Collect scientific data by safely and correctly carrying out chemical experiments
(CLICK or see pg 7 for guidelines)

Analyze data to arrive at a scientific conclusion
(see individual lab projects for necessary background)

Effectively communicate through a laboratory report to an audience of peers unfamiliar with the project
(CLICK or see pg 11 for guidelines)
How to achieve Goal #2

GOAL #2

Establish a scientific question and understand how to answer it experimentally (see lab projects for general considerations)

Collect scientific data by safely and correctly carrying out chemical experiments (CLICK or see pg 7 for guidelines)

Collect scientific data by safely and correctly carrying out chemical experiments

then...

Analyze data to arrive at a scientific conclusion (see individual lab projects for necessary background)

Analyze data to arrive at a scientific conclusion

which you will then...

Effectively communicate through a laboratory report to an audience of peers unfamiliar with the project (CLICK or see pg 11 for guidelines)

Effectively communicate through a laboratory report to an audience of peers unfamiliar with the project

which you will do when you...

Lab Safety Rules (CLICK for guidelines or see pg 8)

Lab Safety Rules

following

Significant figures from measurements (CLICK for guidelines or see pg 10)

Significant figures from measurements

including correct

Recording observations and measurements in a laboratory notebook (CLICK for guidelines or see pg 9)

Recording observations and measurements in a laboratory notebook

correctly

Recrystallization (see CHM111 project 1 on pg 23)

Recrystallization

Distillation (see CHM111 project 2 on pg 31)

Distillation

Gravimetric Analysis (CLICK or see pg 49)

Gravimetric Analysis

Volumetric Analysis (CLICK or see pg 71)

Volumetric Analysis

Qualitative Analysis (CLICK or see pg 81)

Qualitative Analysis

employing proper techniques for all procedures, including

employing proper techniques for all procedures, including

Analyze data to arrive at a scientific conclusion (see individual lab projects for necessary background)

Analyze data to arrive at a scientific conclusion

3
**How to achieve Goal #4**

1. **Establish a scientific question and understand how to answer it experimentally** (see lab projects for general considerations)

2. **Collect scientific data by safely and correctly carrying out chemical experiments** *(CLICK or see pg 7 for guidelines)*

3. **Analyze data to arrive at a scientific conclusion** (see individual lab projects for necessary background)

4. **Effectively communicate through a laboratory report to an audience of peers unfamiliar with the project** *(CLICK or see pg 11 for guidelines)*

   - Well written for the specified audience *(CLICK or see pg 11)*
   - Report sections with appropriate content and correct labels *(CLICK or see pg 12)*
   - Numeral formatting rules *(CLICK for guidelines or see pg 160)*
   - Significant figures *(CLICK or see pg 140)*
   - Statistical analysis *(CLICK or see pg 15)*
   - Tables *(CLICK or see pg 15)*
   - Figures *(CLICK or see pg 16)*
   - Clearly and correctly establishing the question to be answered and the background *(CLICK for guidelines or see pg 17)*
   - Data is presented correctly and effectively *(CLICK for summary or see pg 15)*
   - Clearly and logically present a scientific argument *(CLICK for guidelines or see pg 18)*
   - Error *(CLICK or see pg 18)*

   - Effectively communicate through a laboratory report to an audience of peers unfamiliar with the project which you will do when you...

   - Collect scientific data by safely and correctly carrying out chemical experiments which you will do when you...

   - Analyze data to arrive at a scientific conclusion which you will then...

   - Effectively communicate through a laboratory report to an audience of peers unfamiliar with the project whose overall goal is to...

   - Clearly and logically describing the rationale for a scientific conclusion, citing data qualifying the argument with a realistic discussion of...

   - Error
GUIDELINES FOR CARRYING OUT EXPERIMENTS

LAB RULES

• Bring the laboratory manual and a laboratory notebook (not loose-leaf or spiral) to every scheduled laboratory session. (The notebook used in CHM 111 may also be used in CHM 112.)

• Careful notes should be taken during each laboratory lecture. The instructor will generally provide information on the chemistry underlying the project, as well as advice on the techniques that you will use. Some of this information should be included in your laboratory report.

• You may work in the Chemistry 111, 112 laboratories only during your regularly scheduled laboratory period and only when class is in session.

• If you miss a laboratory for a legitimate reason (illness, death in family, etc.) you must obtain permission from your regular laboratory instructor to make-up the lab at another time. Make-up labs are only allowed when space allows and with the approval of the host professor. During the make-up laboratory you must move your equipment to an unoccupied lab bench.

• During the first scheduled laboratory period, you will be assigned a laboratory locker to which you alone will have the combination. The locker equipment is your responsibility while you are in the course and you should be diligent to keep the equipment clean.

• Test tube brushes and soap (Alconox) are available at each sink.

• The Chemistry Department maintains a stockroom on the third floor of the Hackman Physical Sciences Laboratories (HAC P-304). This stockroom is open during all scheduled laboratory periods for the acquisition of equipment necessary to replace broken items.
LAB SAFETY

Training

- Attendance is required at a presentation on laboratory safety that will be shown during your first scheduled laboratory. Each student must give the instructor a signed form indicating that the presentation was attended and that any associated training materials were examined.
- The laboratory is equipped with fire blanket, showers, eye wash, and first aid supplies. Learn the locations and proper use of these items.

Safety Rules

At all times when you are working in the chemistry laboratory you should use prudent practices. **Recognize that safety is, ultimately, everyone’s individual responsibility.**

- Never work alone in any laboratory.

**Avoid the most common causes of accidents:**

- Exercise care when picking up potentially hot objects.
- Insert glass objects into rubber stoppers and corks with extreme care.

**Avoid contact with laboratory chemicals:**

- Wear clothing that protects as much of your body as possible. Closed-toe shoes are required. All skin below the waist must be covered.
- Use department-approved eye-protection at all times. (Goggles are available for purchase from the College Bookstore.)
- Keep the laboratory bench and work area orderly, clean, and free of items not related to the experiment at all times. Specifically, electronic devices are not allowed on the bench.
- Never sit on or lean against the laboratory bench.
- Use a fume hood when directed to do so.
- Food or drink should only be consumed in the lecture area of the room. Do not chew gum during laboratory sessions.
- Dispose of waste materials and excess chemicals in the appropriate containers as indicated by your instructor.

**When emergencies do occur:**

- Always keep in mind that the first response to the exposure of the eyes or skin to a chemical is immediate, thorough irrigation with water.
- Report all accidents, however minor, to the laboratory instructor immediately.
- Know the exact location of all safety equipment and how to use it.
Preparation is important:

- Perform only assigned experiments. Do not attempt to modify the written procedures unless instructed to do so.
- When conducting experiments ask yourself, “What are the worst possible things that could go wrong?” and “How will I deal with them?” Don’t do the experiment until you are certain of your answers.
- Read the label on the container to be certain it contains the required chemical.

LABORATORY NOTEBOOKS

One of your goals in this laboratory course should be to learn to keep proper records of your work. Your laboratory reports will be based on the data in your notebook, and the more complete the data are the more likely it is that you will be able to prepare a good report. Furthermore, discrepancies and unexpected results can be accounted for only by referring to complete records of your work. In a broader sense, a notebook is essential in any research laboratory where it may be necessary to review data months or years after they were taken; hence full details are necessary.

You will be required to keep your laboratory records in a hard-cover, bound notebook (available for purchase in the College Bookstore). The notebook should contain all experimental data and pertinent observations recorded in ink at the time they are obtained. Data may not be recorded elsewhere for later recopying in the notebook, and, in particular, loose scraps of paper are not permissible for records. The following are the requirements for your notebook:

- Each day’s work should be dated. The project being performed should be indicated clearly.
- No erasures should be made; mistakes should be crossed out with a single line but remain legible.
- Pages must not be removed from the notebook.
- The notes must be neat and orderly enough for someone else to follow them.
- The first few pages of the notebook should be left blank. These pages should be used to keep a running table of contents.
- Use tables to organize data whenever possible.
- The following should be included in the laboratory notebook:
  - All experimental data, such as masses, buret readings, temperature, etc.
  - Notable occurrences (especially phase or color changes).
  - Full details of a procedure need not be recorded, but each step should be noted as it is performed.
  - All mathematical computations during and after the laboratory session.
The following is an example of a portion of notes taken during a laboratory project.

Sept. 23, 2011

The Purification and Identification of an Unknown Solid

Test of solubility of benzil: two spatulas of benzil added to ca. 1 mL cold 95% ethanol. Some solid dissolved with stirring. Heated in water bath—all solid dissolved.

Recrystallization of benzil: Used procedure described by Prof. (also in lab manual, see Figure 1). Half of the sample in vial put in 50 mL beaker. Heated about 100 mL of 95% ethanol on hot plate until too hot to touch. Added small amnts hot ethanol to sample, swirled constantly on hot plate. Took ca. 30 mL ethanol to completely dissolve. Filtered thru filter paper in short funnel in beaker heated with small amnt of ethanol. No residue on paper. Set yellow soln aside, scratching w stir rod formed crystals.

Recrystallization of unknown: unknown white, crystalline solid. Also soluble in hot ethanol. Used same procedure as for benzil with whole amnt of unknown (weight = 1.56 g). After filtration considerable residue left on paper (must not have used enough solvent or funnel was cold). Redissolved material in funnel in more ethanol and repeated. This time little residue. Cooled filtrate in ice bath and scratched with stir rod. Got long white needles. Filtered with suction using Büchner funnel, washed w ca. 2 mL ethanol. Continued suction 15 minutes, collected ppt in recrystallizing dish, put in locker to dry. Will weigh next week to calculate percent recovered.

Recording Significant Figures in your Notebook

When recording a numeric measurement, the number of significant figures conveys how accurate the measurement is.

- When massing, record all digits on the balance. The last place has uncertainty in it (you may even see the digit change).
- When measuring volumes using graduated glassware, estimate and record one digit beyond the markings, as shown.
GUIDELINES FOR LABORATORY REPORTS

EFFECTIVELY COMMUNICATING THROUGH A LABORATORY REPORT

Writing is ubiquitous in the sciences, and students primarily develop their writing skills through writing laboratory reports. As a scientist-in-training, you ultimately need to learn how to make a reasoned and articulate argument that is persuasive and based on scientific evidence; the foundation of that argument needs to be grounded in data that are presented in an organized and thorough manner. The laboratory report communicates to others the results and conclusions you obtained in performing an experiment. You should explain why the experiment was performed, how the experiment was performed, what results were obtained, how the results were analyzed, and what conclusions were reached.

WRITING STYLE AND AUDIENCE

Writing to the specified audience

The audience for the laboratory report is a student with the same general knowledge as yourself, but who has not done the particular experiment you are writing about. In other words, you are writing to a student who understands general techniques, but who does not know the details of the particular project. Here are some notable implications for what you should and should not include in the report:

- You should provide in the introduction necessary information, like chemical equations, so the reader understands why the experiment worked.
- In the experimental section, you do not need to explain general procedures. Your reader is someone who knows how to measure the mass of a sample and carry out a distillation.
- In the experimental section, you do need to explain the procedures specific to this project. Your reader does not know that you massed 0.2300 g of CuSO\(_4\) and dissolved it in 100 mL of water to generate a blue solution.
- In the results section, you must specify important details. If you label a column “Mass of sample” the reader does not know what the sample is or what units you used. A good label would be “Mass of CuSO\(_4\) (g).”
- In the discussion section, you must guide the reader through the logic behind your conclusions. (See “Making a Scientific Argument”)

Writing style

See appendix V.
REPORT SECTIONS

- Your laboratory reports for CHM 111,112 should have the following sections: Title; Introduction; Experimental Method; Results; Discussion/Conclusion.
- The Experimental Method, Results, Discussion, and Conclusion sections should be labeled. The Introduction should not be labeled.

Title
The title should reflect the report's content. It should be brief and grammatically correct. Avoid phrases like “on the”, “a study of”, “research on”, “report on”, “use of”, and so on. Choose terms that are as specific as the text permits: “The Densities of Copper-Zinc Alloys” is better than “The Densities of Metal Alloys”.

Introduction
The introduction is where you provide the context necessary for your reader to understand the rest of the report, particularly the data interpretation and derived conclusion. As discussed in the section on “Making a scientific argument”, key points are to identify the purpose of the lab and necessary experiment-specific background like chemical equations.

Experimental Method
Give the experimental procedure used. Do not repeat the directions that appear in this manual; use your own words for a brief summary of the procedure. Include the important information such as reagents used and weights, times, and temperatures. The results of a procedure should be reported in the results section.

For synthetic procedures, the format illustrated below is helpful because it provides both the amount of reagent in grams and the number of moles.

Sodium carbonate (4.27 g, 0.0403 mole) was added to 25 mL of 1.2 M CuSO₄. A blue precipitate formed immediately after addition. The mixture was heated at 60 °C for 30 minutes.

When a quantitative procedure is used, individual weights or volumes should be included only in a table in the Results section.

KHP was weighed by difference into each flask (Table I).

This statement would appear in the Experimental section; the actual masses would be given in the Results section. Typically, if a quantity is measured repeatedly or is used in a calculation, it is appropriate to include it in the Results section rather than the Experimental section.
Note that the Experimental section is not a set of directions, such as “distill the liquid to determine its boiling point.” As a description of what was done, it should be in the past tense, third person, and in passive voice.

The use of figures, such as a diagram for a distillation, is not necessary.

Results section
The results section is where data is presented. The strength of the report depends on clearly conveying the important data on which the argument will rely. This section contains tables and figures, as elaborated in the section on “Presenting data correctly and effectively.”

Discussion section
The discussion section is where data is discussed in the context of the purpose of the report and used to make an argument. This is where you use the background from the Introduction section and data from the Results section to build a case for your primary point. Use this to explain to the reader how you came to the conclusion you did. Exactly how does the data support your identification of an unknown substance or calculation of equilibrium constant? Be sure to discuss error and how it affects your conclusion.

The discussion section includes your findings and justification for the conclusions that you have drawn from them. Conclusions should be stated concisely and with specificity:

- The blue-green compound had an absorbance maximum in the red region of the electromagnetic spectrum at 645 nm;
- All of the compounds tested gave precipitates with the addition of chloride, but only the compound containing the silver ion turned purple when exposed to daylight;
- About 50% of the crude product was lost during recrystallization from hexane because the product is moderately soluble in cold hexane.

Do not use phrases such as “I believe”, “I concluded”, “I feel”; these are personal opinions.

Conclusions
A separate conclusions section may be requested. This is a short restatement of the main finding of the report.
References

Scientific journals have lengthy instructions for the format of references. For CHM 111 and 112, however, few references will be necessary, and we provide these guidelines:

1. References should be given to all literature sources of information given in the report.
2. Each reference must be indicated by a superscript number given in the text when that source is cited.

*The peak (m/e 87) representing the ion \([\text{CH}_2\text{CH}_2\text{COOCH}_3]^+\) is always more intense than its homologues.*

3. Number references in the order of appearance in the report. Once a citation has been assigned a number, the same number should be used again if reference is cited later in the report.
4. Put the text of all references at the end of the report.
5. Use the formats in the following table:

<table>
<thead>
<tr>
<th>Reference type</th>
<th>General format</th>
<th>Formatted example</th>
</tr>
</thead>
</table>
PRESENTING DATA CORRECTLY AND EFFECTIVELY

Data is primarily presented in the results section in the form of tables and figures.

Significant figures rapidly convey the number of digits to which a value is confidently known.
• Ensure that measurements are presented with the correct number of digits, as described in the section on note taking [page 9].
• Calculated values should be presented with the correct number of digits based on the significant figure calculation rules [Appendix III].

Statistical analysis (see Appendix III for more detail) is used for rapid comparison of measurements.
• When multiple trials have been run, report the average value and a measure of precision, like the standard deviation.
• When a “real” value is known, report a measure of the accuracy.

Tables
The data obtained in the experiment should be presented in tabular form whenever possible. Tables are highly recommended for presentation of data for multiple samples or repetitive experiments. Table I illustrates the presentation of data in tabular form.
• Provide only that data essential for your calculations, determination of identity, or measurement of a property. For example, buret readings before and after delivery are not important—the volume delivered is important. Likewise, give the weights of samples, but do not give weights of beakers or weighing bottles.
• Present data for multiple samples or repetitive experiments in tabular form and give the appropriate statistical information.

For example:
Table I. The molar heat capacity of CO as a function of temperature for two trials.
<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Heat Capacity, 1 (cal deg⁻¹ mol⁻¹)</th>
<th>Heat Capacity, 2* (cal deg⁻¹ mol⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10.05</td>
<td>6.436</td>
<td>6.431</td>
</tr>
<tr>
<td>20.49</td>
<td>6.454</td>
<td>6.448</td>
</tr>
<tr>
<td>24.33</td>
<td>6.460</td>
<td>6.439</td>
</tr>
<tr>
<td>29.97</td>
<td>6.469</td>
<td>6.460</td>
</tr>
<tr>
<td>34.68</td>
<td>6.477</td>
<td>6.475</td>
</tr>
<tr>
<td>42.77</td>
<td>6.490</td>
<td>6.504</td>
</tr>
</tbody>
</table>

* The temperature bath was not regulated electronically; temperatures were read as the bath cooled.
Note the following:

- Tables are numbered with *Roman* numerals.
- Each table must have a heading that briefly but accurately describes the contents.
- Data are given in columns or rows. Each column and row must have a heading; include the units in parentheses.
- Decimal points should be vertically aligned for ease of reading.
- Very large or very small numbers should be given as powers of ten; two ways are shown below. The column on the right is preferred because it is easier to read and requires less type.

<table>
<thead>
<tr>
<th>Table II. Data shown with proper decimal alignment.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentration (mol L⁻¹)</td>
</tr>
<tr>
<td>-------------------------</td>
</tr>
<tr>
<td>1.1 x 10⁻³</td>
</tr>
<tr>
<td>1.2 x 10⁻³</td>
</tr>
<tr>
<td>1.3 x 10⁻³</td>
</tr>
</tbody>
</table>

- Do not use ditto marks.
- Be sure that all columns are necessary. If there are no data in most of the entries of a column, it should probably be deleted and replaced with a table footnote. If all of the entries of a column are the same, the column should be replaced with a table footnote.
- Data from a literature source should be properly cited.
- Unreliable data should be noted with a table footnote.

**Figures**

Graphs are treated as figures and numbered sequentially with other figures. See Figure 1 for the names applied to the various aspects of graphs.

- Every graph must have a caption that includes the figure number and a brief description. The caption should be understandable without reference to the text.
- The dependent variable is normally the vertical axis and the independent variable is normally the horizontal axis.
- Graphs should be generated using a program such as Excel (unless your instructor specifies otherwise)
- Graphs should have appropriate axes, legends, and captions.
- Figures are numbered with Arabic numerals.
**Figure 1.** The various aspects of a graph defined.

**MAKING A SCIENTIFIC ARGUMENT**

Establish the question to be answered and provide necessary background

In order for your reader to understand the argument you will make based on the data, they need to understand the point and the background. This is all established in the introduction section. The necessary background depends on the audience. The audience for the lab report is another student who has the same basic knowledge as you, but who is unfamiliar with the particular experiment.

- Include a clear statement of the purpose of the experiments.
- Do include specifics of the particular report, like chemical reactions and techniques used.
- When reporting chemical equations, use a space between a stoichiometric coefficient and a formula in chemical equations:

\[
2 \text{H}_2 + \text{O}_2 \rightarrow 2 \text{H}_2\text{O}
\]
• Do not include descriptions of general chemical knowledge.

Describe the rationale for the conclusion, citing the supporting data

The discussion section is where you present your interpretation of the data and how it comes together to prove a point.

• Clearly state the conclusion at the beginning of the discussion section.
• Explain how the data supports the point. Remember that this is not obvious to your audience.
• Refer back to the appropriate data in the results by citing the Table or Figure.
• Refer to all Tables and Figures. If it does not seem necessary to refer to a particular table or figure, an important point is likely missing or the table/figure is not necessary.

Discuss the error present realistically (see Appendix V for more about error)

Discussion of error is an important part of your argument. There is nearly always evidence of error, and you should discuss whether or not it could discredit your conclusion. Identify the evidence for error, describe its cause, and how it affects the conclusion.

• Identify evidence of error. Examples include:
  o Multiple trials that are not identical, especially is there is a large standard deviation. The percent sulfate found for sample 1 was several percent lower than the percent sulfate found for sample 2 because a significant amount of solid lead (II) sulfate was lost through a small crack in the filter crucible.
  o Values that are different than expected based on a known value or chemical theory. The sharp melting range of 134-135 °C indicates a pure compound. The small discrepancy between this value and the literature value is not surprising because the thermometer was not calibrated.
  o Experimental errors recorded in the laboratory notebook.

• Describe a plausible cause of the error. “Human error” is not descriptive. Use the observations in your notebook and understanding of the experiment to determine what most likely went wrong.

A note on “human error”: As an example of a possible source of error, do not include examples related to your operation of instrumentation such as “the buret may have been misread”. So-called “human error” is part of any scientific measurement and in many cases accounts for much of the error in a result. If three people read the level of liquid in a graduated cylinder they may each obtain a different reading—primarily a result of trying to obtain the maximum amount of information from a piece of apparatus. If the graduated cylinder has graduations at each mL, each observer will try to estimate the volume to the closest tenth of an mL and in the process the estimations will differ slightly, probably by ±0.1 mL. This type of error is referred to as indeterminate error. On the other hand, if one observer consistently reads the cylinder at a 45° angle (rather than perpendicular to the cylinder), this will produce a faulty reading usually called a determinate error. Your textbook and the Appendix should be consulted...
for a more detailed discussion of errors. In any event, the term “human error” should not be used. In addition, performing calculations incorrectly is not a source of error.

- Describe how the error affects the result.
Purification and Identification of an Unknown Solid Compound

Aaron Williams
September 23, 2012

The objective was to purify an unknown solid compound by recrystallization and to identify it using its melting point. The compound was identified by mixing it with other known substances and observing the changes in melting point ranges. The melting points of impure mixtures will have a greater range and will be lower than that of the pure compound.

Experimental Method
Ethanol was heated in a beaker with boiling chips on a hotplate. The hot ethanol was mixed with the unknown and swirled to dissolve until a saturated solution was formed. The solution was filtered by gravity to remove impurities, and the filtrate was set aside to cool. Crystallization was induced by scratching the bottom of the beaker with a stirring rod. The compound was then collected by suction filtration in a Büchner funnel. The crystallized unknown was set aside in a recrystallizing dish to dry.

After the crystals had been air-dried for a week, the melting point range of the unknown solid compound was determined, twice, using the MEL-TEMP melting point apparatus. Then, a quarter of a spatula full of the unknown compound was mixed with each of six compounds that had melting points within six degrees of the observed melting point. During the determination of the melting point of each mixture, the temperature was regulated to increase at a rate of approximately 2 °C per minute beginning at about 10 °C below the melting point.

Results
The results of the melting range determinations are given in Table I.

<table>
<thead>
<tr>
<th>Compound Tested</th>
<th>Melting Point Range (°C) when Mixed with Unknown</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unknown (Trial 1)</td>
<td>132-135</td>
</tr>
<tr>
<td>Unknown (Trial 2)</td>
<td>133-135</td>
</tr>
<tr>
<td>Cinnamic acid</td>
<td>110-120</td>
</tr>
<tr>
<td>2-furoic acid</td>
<td>105-110</td>
</tr>
<tr>
<td>Sebacic acid</td>
<td>120-126</td>
</tr>
<tr>
<td>Benzoin</td>
<td>131-136</td>
</tr>
<tr>
<td>Benzamide</td>
<td>109-117</td>
</tr>
<tr>
<td>Maleic acid</td>
<td>121-132</td>
</tr>
</tbody>
</table>
Discussion
The melting points of the mixtures of the compounds were obtained in order to determine the identity of the unknown. Impurities within a compound or mixture cause the melting point to decrease and also cause the substance to melt over a larger range than it would if it were pure. Thus, if two different compounds are mixed together, they will act as impurities within each other, causing them to melt at lower temperatures and over longer ranges.

Based on the melting points (see Table I), the unknown solid compound is most likely benzoin. When mixed with the unknown substance, benzoin had the closest range to the melting point range of both the unknown and benzoin’s known melting point, 136°C.¹ No other mixture of compounds came as close to matching the melting point range of the unknown. The somewhat larger range of the melting point of the unknown relative to that of pure benzoin may be the result of impurities remaining in the unknown.

Reference
GRAVIMETRIC ANALYSIS

Chemical analysis is the general term applied to procedures used in determining the composition of matter. The determination of what elements or compounds are present is called qualitative analysis. Quantitative analysis involves the determination of the proportions in which the various elements or compounds are present. Both qualitative and quantitative analyses are required when a compound is synthesized. Although a synthetic procedure is designed to produce a particular compound, that compound may not have been formed in the procedure. Quantitative analyses are also used to investigate environmental systems, in the reconstruction of old artistic methods (art conservation), in the analysis of crime scenes (forensic chemistry), and for the control of the quality of pharmaceuticals, commercial chemicals, and food stuff. In general, analytical techniques require great care, consistency, patience, and an understanding of underlying concepts.

One general method of quantitative analysis is called gravimetric analysis (from the Latin gravis = heavy, and metricus = measurement) so named because the only measurements required are weighings.

GRAVIMETRIC PROCEDURE

Most gravimetric analyses involve the following procedures:

1. A sample of the matter under investigation is accurately weighed (usually to ±0.1 mg).
2. The weighed sample is dissolved to form an aqueous solution. (Depending on the nature of the sample, water alone may dissolve it, or other reagents may have to be added.)
3. To the solution is added some other substance that reacts with the constituent in such a way as to form a precipitate. All of the constituent in the original sample must end up in this precipitate.
4. The precipitate is removed from the liquid by filtration using either a filter crucible or an “ashless” filter paper in a funnel. In either case, the liquid passes through and the precipitate is retained.
5. The precipitate is washed to remove adhering impurities and is dried to constant weight. Some precipitates can be thoroughly dried at relatively low temperatures. In those cases a filter crucible is used for the filtration, and the crucible and its contents are heated in a laboratory oven until no further weight loss occurs.
GRAVIMETRIC TECHNIQUES

In carrying out the procedures described above, the following techniques are employed to minimize errors.

Drying the Sample

Finely divided solids contain water vapor adsorbed on the surfaces of the particles. The amount of this loosely bound water is dependent upon temperature, humidity, and the history of the sample, as well as the size of the sample. Consequently, the weight of the sample is not reproducible unless this adsorbed moisture is removed. This drying is usually accomplished by heating the sample in an oven at 105-110°C for at least one hour. The material to be dried is placed in a weighing bottle and the weighing bottle and its lid are placed in a crystallization dish, as shown in Figure 8. Initials are written (pencil or ink) on the frosted area of the crystallization dish. No labels are used because they char and flake off in the oven.

![Figure 8. Setup for drying the sample.](image)

Use of the Desiccator

Samples that have been dried can be kept dry by storing them in a desiccator. The common desiccator (Figure 9) is a covered glass container designed for storage of small objects in a dry atmosphere. It must be pointed out that the efficiency of an ordinary desiccator as a method of drying is very poor, since it takes a long time for the moisture to diffuse from the object through the air to the desiccant. The purpose of a desiccator is to maintain an already dried object indefinitely in a dry condition.

![Figure 9. Desiccator components](image)
A desiccator is prepared for use as follows (use anhydrous calcium chloride as desiccant). Care must be taken when dealing with finely divided solids, particularly solids that can remove water. For example, these materials can irritate the lungs and desiccate mucous membranes and eyes.

1. Carefully remove the old desiccant in the hood, discarding it as directed by the instructor.
2. Wipe off the old grease with a paper towel, wash with soap and water, and dry with a clean towel. No lint should remain after drying. The porcelain plate should also be cleaned.
3. In the hood carefully add desiccant to the desiccator to a depth of one inch only (pour carefully to minimize dust in the air and on the sides of the desiccator). Roll an 8-1/2 by 11-inch sheet of paper into a wide-mouthed funnel and prevent it from unrolling by fastening it with two paper clips. Pour the desiccant down this funnel, holding its bottom against the bottom of the desiccator. While pouring, hold the desiccator and funnel in an inclined position, so that the desiccant slides down gently and does not fall through any appreciable height. When a sufficient amount is added, level the desiccant and wipe off any dust from the upper inner walls of the desiccator. Then insert the porcelain shelf.
4. Smear a thin band of Vaseline® around the ground-glass area, put on the cover, and rotate it back and forth through a small angle (without lifting and breaking the seal) to distribute the Vaseline®. If the proper amount of lubricant is used, the contact area is clear. Carefully wipe excess Vaseline® from the inner and outer edges with a paper towel without smearing.

Some tips on the proper handling of a desiccator follow:

1. To open the desiccator place it squarely upon the desk top, anchor it to the desk top with one hand, and slowly slide the cover to one side with the other hand. If the pressure inside is less than atmospheric, the sliding should be done very slowly, stopped as soon as air begins to hiss into the vessel, and not continued until pressures are equalized.
2. To carry the desiccator place the palms of the hands on opposite sides, with index fingers under the lip. The thumbs should hold the lid on so that it will not pop off if the inner pressure is accidentally greater than atmospheric.
3. Objects should be put into the desiccator when hot. Wait about a half-minute after any red glow disappears and then put the object on the porcelain shelf, being very careful not to touch the glass which might crack. Put the cover on but leave a small opening to the air for another half-minute. Then slide the cover into place and seal. If the desiccator is closed too soon, pressure will build up inside and may pop the lid off. As the contents cool, a slight vacuum develops inside, pulling down the lid and giving a good seal.
4. The desiccator should be opened as infrequently as possible, kept open only as long as necessary, and objects should be lowered into it with as little disturbance of the air as possible. When closing the desiccator, slide on the top and rotate it slowly in both directions to produce an even seal.

**Weighing**

The most convenient method of weighing samples for analysis is “weighing by difference”. Remember that samples and their containers must be allowed to reach room temperature before being weighed. This method is carried out as follows:

1. The weighing bottle with excess sample is weighed to the nearest 0.1 mg.
2. The sample is transferred from the bottle to the vessel in which the next step of the analytical process is to be performed. Everything that goes out of the bottle must go into the receiving vessel. It is better to underestimate the amount to be transferred because a second portion may always be added if the first is not large enough; it is poor practice to transfer from the receiving vessel back to the weighing bottle if the first portion is overestimated.
3. The weight of the weighing bottle after removal of the sample is determined to the nearest 0.1 mg.
4. The weight of the sample is determined as the difference in weight between the bottle with sample and the bottle minus sample.

![paper strip](image)

**Figure 10.** The correct method for handling weighing bottles.

After being cleaned, containers to be weighed should be handled as infrequently as possible with the fingers. Weighing bottles may be conveniently handled as shown in Figure 10.
Dissolving the sample

Care must be taken in adding solvents to dissolve the sample. Because of the danger of loss by splashing, water or reagents are never allowed to fall into a container, but are added by the methods illustrated in Figures 11 and 12.

Tip of glass rod held close to, but clear of, beaker contents, unless rod is to be immersed in solution after pouring.

Figure 11. One method of adding water to a sample without splashing.

Beaker held inclined, so that liquid trickles slowly down side of beaker without splashing.

Figure 12. Another method of adding water to a sample without splashing.

Filtering the precipitate

If the precipitate can be dried at temperatures below 150°C—low enough to permit drying in an oven—then the most convenient device for collection of the precipitate is a filter crucible, which is a glass crucible with a porous disc in the bottom. It is mounted on a filtering flask using a crucible holder and the filtration is accomplished by suction (Figure 13).

As much of the mother liquor as possible is decanted through the filter without disturbing the precipitate in the beaker, so that the major portion of the liquid may be filtered rapidly and before the precipitate begins to clog the filter. Figure 14 illustrates the pouring operation. It is good to keep the filter crucible always fairly full of liquid. After the filter crucible is filled, it may be necessary to interrupt the pouring and to set the beaker on the table while waiting for the crucible to empty. When pouring is interrupted, the beaker is not turned upright immediately, because there is a tendency for a drop to adhere to the outside of the spout and to run down and be lost. Instead, the beaker is tipped back only slightly and the clinging drop is transferred to the rod by touching the rod to the beaker spout. The drop adhering to the end of the rod is removed by touching it to the side of the crucible. The rod is laid across the top of the beaker with the wet end resting on the spout.
It is advantageous to wash some precipitates by decantation because better contact may be achieved between precipitate and wash liquid. To wash by decantation, the beaker is set upright on the table, and the bulk of precipitate adhering to the sides is washed onto the bottom with a stream from the wash bottle. The precipitate and the liquid are swirled, the precipitate allowed to settle for a few minutes, and the supernate then poured through the crucible, leaving most of the precipitate behind. This process is repeated once or twice more using enough wash liquid per portion to give a depth of 1-2 cm. The filter crucible is allowed to drain before the next wash portion is added.

After the last washing by decantation, much of the precipitate still remains in the beaker. The loose part of this precipitate is transferred to the filter as shown in Figure 15. One hand holds the beaker and rod while the other hand manipulates the stream of wash water.

After the loose part of the precipitate is removed, the adhering particles are dislodged with a policeman (Figure 16) fixed to another clean glass rod. Any precipitate adhering to the original rod is also dislodged, and the rod is rinsed so that the washings fall into the beaker; the original rod is laid aside.

The dislodged precipitate is transferred to the crucible. The policeman should be wet with distilled water before contacting the precipitate. When a rod with a policeman is used for pouring, the beaker spout is placed against the policeman; if the spout is placed against the glass, solution may be lost as it flows over the shoulder of the policeman. The loosening and transfer process is repeated until inspection of the beaker shows no precipitate remaining.

After the precipitate is quantitatively transferred to the crucible, the final washing is begun immediately. A precipitate is never allowed to dry before the final washing, nor even to drain for a long time, for it might cake or channel, and wash liquid would then drain ineffectively through the cracks.
Drying the precipitate

A precipitate collected in a filter crucible may be brought to constant weight in a drying oven. Drying temperatures and time periods vary with the nature of the precipitate.

GRAVIMETRIC CALCULATIONS

The computations involved in gravimetric analyses are simply applications of stoichiometry. The only information required for calculation of the percentage of the constituent in the sample is:

a. the accurate mass of the sample being analyzed,

b. the accurate mass of the precipitate being weighed, and

c. the correct formula of that precipitate. Implicit in the method of calculation is the assumption that two conditions have been met in the analytical procedure:

1. the constituent being determined was completely precipitated, and

2. the precipitate is a single, pure substance.
VOLUMETRIC ANALYSIS

Volumetric analysis is a widely used quantitative analytical method. As the name implies, this method involves the measurement of volume.

VOLUMETRIC PROCEDURE

1. A solution is prepared from an accurately weighed sample of the material to be analyzed.
2. A substance is chosen that will react rapidly and completely with the constituent that is to be determined. A standard solution of this substance is prepared. A standard solution is one of accurately known concentration, usually expressed as molarity with a precision of ± 0.0001 M.
3. Some of the standard solution is poured into a buret. The buret is graduated (usually in tenths of a mL) so that the volume of solution that passes through the stopcock may be accurately measured.
4. Standard solution is added slowly from a buret to the “unknown” solution, allowing the reaction to occur. This process is called titration, and the solution in the buret is known as the titrant. Ideally, the titration is continued until the reaction is complete; that is, until the amount of reactant added is exactly the amount required to react with all of the constituent that is being determined. This point is called the equivalence point. The equivalence point is detected by adding an indicator to the “unknown” solution before the titration is begun. An indicator is a substance that gives a color change at or near to the equivalence point. The point at which this change occurs is called the endpoint. The particular indicator that is used depends on the specific reaction involved. The titration is stopped when the endpoint is reached.
5. The exact volume of standard solution required can be measured, from buret readings before and after the titration. Since the molarity of the standard solution is known, the number of moles of titrant can be calculated. Furthermore, from a knowledge of the equation for the reaction, the number of moles of constituent present in the sample can also be calculated.

STANDARD SOLUTION

The most accurate and convenient way of preparing a standard solution is to weigh the reagent accurately, dissolve it, and dilute the solution to a definite volume in a volumetric flask. This method can be employed only if the reagent is a primary standard. In order to qualify as a primary standard, a substance must meet the following requirements: it must be obtainable in pure form; it must be stable both in pure form and in solution; it must be easy to dry and keep dry; and it must be soluble in a suitable solvent.

Unfortunately, many useful reagents do not meet these requirements. In such a case, the reagent is dissolved and made up approximately to the concentration desired. This solution is then standardized by titrating it against a primary standard. A solution standardized in this fashion is a secondary standard.
TITRATION PROCEDURE

1. Carefully clean the buret with Alconox solution and a brush to remove all dirt and grease. Rinse the buret with several portions of tap water by partially filling it, draining a small portion through the tip, and pouring the bulk of the rinse from the top of the buret while rotating it. If any drops of water collect on the walls when the rinse is poured from the buret, the cleaning is unsatisfactory and must be repeated. Finally, rinse the buret with two or three portions of distilled water.

2. Before filling the buret, rinse it with the titrant solution two or three times, using about 10 mL portions.

3. Place the buret in a buret clamp attached to a large ring stand. Using a funnel, fill the buret with titrant to a level above the zero mark. Place a beaker under the buret and open the stopcock for a few seconds to remove all air from the tip. The top of the solution should now be below the zero mark.

4. **Read the buret to ± 0.01 mL.** (Because the buret is graduated to 0.1 mL, the second decimal place must be estimated.) To make this reading, it is necessary to locate the meniscus (that is, the surface of the liquid) with respect to the markings. The reading is considerably affected by the position of the eye and by the color of objects behind the buret. The variation of the apparent position of the meniscus is called **parallax.** To minimize errors due to parallax, the meniscus should be level with the eye (Figure 18).

   ![Figure 18. Avoid parallax error by using correct eye position.](image)

The variation of colors behind the buret can be eliminated by mounting a piece of black tape on a white card and holding it behind the buret as shown in Figure 19. If a highly colored liquid is used, it is more convenient to read the position of the top of the meniscus.
5. Place the solution that is to be titrated in an Erlenmeyer flask and add the appropriate indicator. Position the flask under the buret. Add the titrant slowly from the buret while swirling the contents of the flask to assure adequate mixing. As the endpoint is approached, the titrant must be added very slowly—a drop at a time. Usually there is warning as the endpoint is approached. If the endpoint is a color change, the change is produced momentarily where the reagent drops into the solution, but fades with stirring into the bulk of solution. This fading occurs more slowly as the endpoint is approached.

6. If the indicator change is a very sharp one, it may be desirable to add standard solution only a half drop at a time near the endpoint. This may be done by opening the stopcock slightly until a drop begins to form on the buret tip. When the droplet has grown to a few hundredths of a mL (one drop is about 0.05 mL), it is touched to the side of the titration vessel and rinsed down with a little distilled water from the wash bottle.

7. If an endpoint is not distinct, or if it is unfamiliar, it may be difficult to decide when the endpoint has actually been reached. In this case, record the buret reading, add another drop, and note the change produced. If the observer is still uncertain, another reading should be recorded, and another drop added. When a series of such readings have been recorded it is easier to select the endpoint in retrospect than by direct approach.

8. When the endpoint has been reached, subtract the initial buret reading (step 4 above) from the final reading to obtain the volume of titrant used.
QUALITATIVE ANALYSIS TECHNIQUES

Qualitative analyses are generally performed on the semi-micro scale (total volumes of a few milliliters). The equipment is ordinary laboratory equipment, but small sizes. For example, 3” test tubes and 50 mL beakers are favored.

All equipment must be kept clean and should be rinsed several times with distilled water before use. The inner surfaces of glassware should not be dried with towels. Stirring rods and capillary pipets should never be placed on bench tops.

REAGENTS

Reagents are stored in several places in the laboratory:

- Concentrated acids and bases are in the hood.
  
  HCl (12 M)
  HNO₃ (16 M)
  H₂SO₄ (18 M)
  CH₃COOH (17 M)
  NH₃ (15 M)

  These reagents must be handled very carefully in the hood.

- Dilute acids and bases are on the shelf above your lab bench (in small dropper-bottles):
  
  HCl (3 M)
  HNO₃ (3 M)
  H₂SO₄ (3 M)
  NaOH (3M)
  NH₃ (3 M)

- Other reagents used for identification tests and known solutions of cations and anions are stored on the side-shelf. Do not take these reagents to your lab bench. Use these reagents at the side-shelf and return them to their proper place (the bottles are numbered).
MIXING AND HEATING SOLUTIONS

Most chemical reactions take place in test tubes by dropwise addition of reagents (stored in dropper-bottles). Solutions are mixed with stirring rods or by flicking the test tube with a finger (Figure 20). Heating should be done in a water bath. A 250 mL beaker with a perforated aluminum ashtray to support test tubes makes a suitable water bath (Figure 21). Solutions in test tubes should not be heated directly with a flame from a Bunsen burner.

Figure 20. Mix solutions with a small stirring rod or by flicking.

Figure 21. Setup for heating solutions
TESTING THE ACIDITY OR BASICITY OF A SOLUTION

Instructions sometimes call for addition of acid or base to make a solution acidic or basic. Be certain to mix the added acid or base into the solution thoroughly before testing. The most common indicator is litmus paper, which is red when acidic and blue when basic. If the acidity of a solution is to be tested, remove a drop of the solution with a stirring rod and touch the rod to a piece of litmus paper (Figure 22). Do not dip the paper into the solution.

![Correct method for testing the acidity or basicity of a solution. Incorrect method](image)

Figure 22. Testing the acidity of a solution.

CENTRIFUGATION

Precipitates are separated from their mother liquors by centrifugation. Keep the centrifuge balanced by placing a counterbalancing test tube, filled with an equal volume of water, directly opposite to the test solution (Figure 23). The solution should be centrifuged for several minutes to pack the precipitate in the bottom of the tube. The mother liquor can be withdrawn carefully with a capillary pipet.

![Counterbalance the solution to be centrifuged](image)

Figure 23. Counterbalance the solution to be centrifuged.
QUANTITATIVE PRECIPITATION

The precipitate must be washed to remove traces of the mother liquor. Washing is a very important part of the separation procedure. Add a few drops of water to the precipitate and gently stir the precipitate with a stirring rod. Centrifuge the mixture and remove the wash water with a capillary pipet. Repeat the washing procedure several times.

Figure 24. Test for complete precipitation.

When precipitation is part of a chemical separation, the precipitation must be quantitative; i.e., the precipitation must remove all of the ions to be precipitated from the solution so that they do not interfere with subsequent tests. The correct method (Figure 24) is to add the precipitating reagent dropwise, stir thoroughly, and centrifuge the solution. Add another drop of reagent. If more precipitate forms, stir, centrifuge and test again. When precipitation is complete, add a few drops of reagent in excess to make use of the common-ion effect.
APPENDIX II - INSTRUMENTATION PROCEDURES

MAKING MEASUREMENTS WITH THE XPLORER GLX

During the course of several lab projects, you will be using the Xplorer GLX datalogger. This handheld device, coupled with the appropriate sensor, provides measurements of pH, temperature, conductivity, and light absorbance. In the following sections, measurements of pH and absorbance will be discussed; instructions for using each sensor with the Xplorer unit are at the end.

MEASURING pH:

Perhaps the most common measurement made in chemistry is the determination of the pH of a solution. The instrument used to make this measurement, a pH electrode, is an electrochemical cell that measures the electrical potential difference between an internal solution of constant hydrogen ion concentration and an external solution (the sample) of unknown hydrogen ion concentration.

The pH sensor has a thin glass membrane shaped into a bulb that is filled with a solution of constant pH; this is the pH-sensitive part of the electrode. Only $H^+$ can bind significantly to the glass membrane. Although $H^+$ ions can’t cross the glass membrane, $Na^+$ ions can. Inside the pH electrode are both glass and reference electrodes, forming a single combination electrode. This cell can be represented as follows:

\[
\begin{align*}
\text{Glass membrane} & \quad \text{selectively binds } H^+ \\
\text{Outer reference electrode} & \quad \text{H}^+ \text{ in the sample solution} \quad \text{H}^+ \text{ inside the glass electrode} \\
\text{H}^+ \text{(aq, outside)} & \quad \text{H}^+ \text{ (aq, inside), } Cl^- \text{(aq)} \\
\text{Inner reference electrode} & \quad \text{Ag} | AgCl | Ag
\end{align*}
\]

where a single vertical line denotes the phase boundaries and a double vertical line represents the salt bridge. The electrode described on the left is typically a coiled $Ag | AgCl$ electrode, whereas the electrode on the right is often a straight $Ag | AgCl$ electrode down the center of the electrode.

The exchange of $Na^+$ ions results in a potential difference across the glass membrane. The two electrodes measure the potential difference across the glass membrane that develops when the sensor is in contact with a solution with a different $[H^+]$. The potential difference between the two electrodes depends on the $[Cl^-]$ and the potential difference across the membrane; since $[Cl^-]$ is relatively constant due to its large excess in concentration and $[H^+]$ is fixed inside the glass membrane, the only variable is the pH of the sample. It has been found experimentally that the potential ($E$) across the membrane follows a Nernst-like relationship at 25°C and over a pH range of ~0 to 12 as follows:

\[
E = \text{constant} + 0.0592 \cdot \text{pH}_{\text{outside}}
\]
Therefore, the voltage of an ideal pH electrode would change by 59.16 mV for every unit change in pH.

**MEASURING LIGHT ABSORBANCE:**

Spectrophotometry is an analytical technique that depends on a compound absorbing light and can be used for the quantitative determination of certain ions or molecules. When the procedure is focused only on the adsorption of visible light, the procedure is called colorimetry. In a spectrophotometric procedure, light is passed through a monochromator to produce one wavelength. This monochromatic (or “one color”) light, with irradiance $P_0$, passes through the sample across distance ‘b’. Some of the light will be absorbed by the sample, and the remaining (unabsorbed or transmitted) light, $P$, emerges from the other side of the sample (Figure 32).

![Figure 32. A simple schematic for a spectrophotometer.](image)

The amount of light absorbed or transmitted will depend on the wavelength of light. The relationship between transmittance ($T$), or the fraction of light that is not absorbed by the sample, and absorbance ($A$) is:

$$A = \log \left( \frac{P_0}{P} \right) = -\log T$$

When no light is absorbed by the sample, $P=P_0$, $T=1$, and $A=0$. If 90% of the light is absorbed, then $T=0.10$, and $A=1.0$.

Absorbance is important because it is directly proportional to the concentration, $c$, of the absorbing species (Figure 33). The mathematical relationship between absorbance and concentration is called the Beer-Lambert Law or Beer’s Law:

$$A = \varepsilon bc$$

where $A$ is the absorbance, $b$ is the pathlength (cm), and $c$ is the sample concentration (M). Because the molar absorptivity, $\varepsilon$, has units of $\text{M}^{-1} \text{cm}^{-1}$, the absorbance will be unitless. The molar absorptivity, a characteristic of the light-absorbing substance, may be different at different wavelengths, and changes with temperature. Therefore, the molar absorptivity is typically determined experimentally.
Figure 33. Increasing absorbance with increasing concentration of the copper ammonia complex at 660 nm. How might you determine the molar absorptivity from this data?
Key Functions and Ports

- **Temperature Ports**
- **Voltage Port**
- **Signal Output Port**
- **Function Keys**
  - Execute the menu or function above each key
- **Arrow Keys**
  - Navigate up, down, right, and left
- **Exit and cancel changes**
- **Record a point in manual sampling mode; Annotate data with text or voice**
- **Numeric and text input**
- **Microphone**
  - For sound input
- **USB Ports**
- **AC Adapter Port**
  - Activate and Accept changes
- **Return to the Home Screen**
- **Start and Stop data collection**
- **Delete selected item**
- **Power**
  - Press and hold for 1 second to turn unit On or Off
- **Speaker**
  - For sound output
XPLORE GLX INSTRUCTIONS

How to measure pH

1. When you are finished with the GLX please place it back on the charger, which plugs into the right side of the handle.

2. Insert the pH probe into the first port on the end of the unit

3. Insert a USB drive into the slot on the right side of the datalogger.

4. Turn on the GLX by pressing

5. Press and then press F4 for sensor.

6. Press F1 for mode, use the arrow keys to select “manual” and press .

7. Press to select “keyboard data” and type “volume” and press .

8. Use the arrow keys to select “measurement unit” and press . Type “mL” and press .

9. Use the arrow keys to select “number of digits” and use +/- to change it to 2. Press F1 for ok.

10. Use the arrow buttons to select “pH” and press F2 for properties. Set the number of digits to 2 using the + button. Press F1 for ok.

11. Press .

12. Press F2 for table.

13. Press to begin recording data. Press to record the pH, enter the data for volume, and press F1 for ok. Continue this for as long as instructed.

14. Press to stop recording.

15. To export your data to a USB drive, press F4 for “tables” and select “export all data”. Press and enter the file name. MAKE SURE THE NAME ONLY CONTAINS LETTERS OR NUMBERS AND ENDS WITH .TXT OR IT WILL NOT OPEN IN EXCEL. Press again and press F1 for ok.

16. Start the next trial by repeating steps 13-15. Make sure each trial has a unique name.
**How to measure temperature**

1. When you are finished with the GLX please place it back on the charger, which plugs into the right side of the handle.

2. Insert the temperature sensor into position 1 on the left side of the datalogger.

3. Insert a USB drive into the slot on the right side of the datalogger.

4. Turn on the GLX by pressing  

5. Press  and then press F4 for sensor.

6. Press  to select “sample rate unit” and use the arrow button to change to seconds. Press  .

7. Use the arrow keys to select “sample rate”, press  and change to 10. Press again.

8. Use the arrow keys to select “temperature” and press F2 for properties.

9. Use the arrow keys to select “number of digits” and use +/- to change it to 2. Press F1 for ok.

10. Press  .


12. Press  to begin recording data. Record for as long as the laboratory instructions indicate.

13. Press  to stop recording.

14. To export your data to the USB press F4 for “tables” and select “export all data”. Press and name your file appropriately (MAKE SURE THE NAME ONLY CONTAINS LETTERS OR NUMBERS AND ENDS WITH .TXT OR IT WILL NOT OPEN IN EXCEL) and press again. Press F1 for ok.

15. Start collecting the next set of data by pressing  and continue from instruction #11.
How to measure absorbance

1. When you are finished with the GLX please place it back on the charger, which plugs into the right side of the handle.

2. Insert the sensor into the first port on the end of the unit.

3. Turn on the GLX by pressing .

4. Press . Use arrows to move to “digits”. Then press .

5. Press F2 to display four color absorbances.

6. If the desired absorbances and transmittances are not displayed, press once to select the titles, and again to change the title. Use the arrow buttons to select the bottom or top wavelengths, and then press .

7. Fill one cuvet with at least 6 mL of deionized water, and screw the cap on tightly. Handle the cuvet by the cap and wipe the glass clean with a KimwipeÔ. Avoid touching the glass.

8. Insert the cuvet into the colorimeter, and close the lid.

9. Press the green button on the colorimeter

10. Remove the deionized water sample when the light on the green button goes out. The colorimeter has now been calibrated for 0% absorbance (100% transmittance) with only water.

11. The test solution may then be added to a new cuvet and placed in the colorimeter. The absorbance can be recorded by hand directly into your lab book. Record the absorbances for all of the standard solutions and any samples.

12. When finished, the colorimeter may be disconnected from the Xplorer GLX.
SPECTRONIC 20

INSTRUCTIONS

1. Turn instrument ON — wait 15 min.

2. Set to desired wavelength.

3. Adjust meter to zero (0% T).

4. Wipe outside of blank tube (containing distilled water) and insert the blank into the cell compartment. Make sure that the white line on the tube lines up with the line on the compartment.

5. Set meter to 100%.

6. Remove blank. Wipe outside of sample tube and make certain there are no bubbles in the solution. Insert sample tube into the cell compartment. Make sure that the white line on the tube lines up with the line on the compartment.

7. Read % T and Absorbance on the meter.
INFRARED SPECTROMETER

INSTRUCTIONS

The wavelengths of infrared (IR) radiation extend from ~750 nm, at the edge of red in the visible range, to ~40,000 nm. When organic or inorganic molecules are exposed to IR radiation, the strength of the radiation is not energetic enough to break bonds or to excite the electrons in molecules. Instead, the absorption of IR radiation results in changes in the vibration of molecules. The number of ways a molecule can vibrate is related to the number of atoms, and therefore the number and type of bonds that it contains (Figure 34).

![Figure 34. Adsorption of IR radiation for a range of atoms and a variety of bonds.](image)

Note that IR radiation is measured in wavenumbers (cm\(^{-1}\)).

For example, 4000 cm\(^{-1}\) = 2.5 mm = 2500 nm.

Each molecule produces a distinct and characteristic adsorption pattern based on the IR radiation that the molecule absorbs, which results in an IR spectrum (Figure 35).

![Figure 35. IR adsorption spectrum for an organic molecule (left) and an inorganic carbonate. By looking at the adsorption bands, can you figure out what atoms and bonds might be present?](image)

Sources of IR light are not lamps, but are instead inert solids that are heated to high temperatures by an electric current (Figure 36). The sample is then exposed to IR radiation that is generated by the hot inert solid. In this case, the sample is placed on a crystal, which is part of
the ATR (attenuated total reflectance) platform. The incoming IR radiation is reflected from the surface of the sample, with a penetration depth of ~0.5 to 2 µm into the sample. The resulting radiation passes through a monochromator (from monochromatic or “one color”) to remove stray or scattered light. The resulting IR spectrum will show the IR absorption bands that are indicative of the functional groups present on the sample placed on the ATR surface and are characteristic of that molecule.

Figure 36. Schematic drawing of an IR spectrometer.

HOW TO COLLECT IR SPECTRA
1. Clean the crystal with several drops of ethanol, wipe dry with a Kimwipe™, and allow to dry for several seconds.
2. Record the background spectra by pressing the “Background” button at the top of the screen.
3. Place your sample on the crystal. If solid, press down with the pressure accessory or clamp.
4. Change the sample name.
5. Record the sample spectra by pressing “scan”.
6. Print your spectra by going to “File”, then “Print” and clicking OK. To save your spectra on a flash drive, go to “File”, then “Send To”, then “Word Pad”, then “New Word Pad Document”. The spectrum will open in Word Pad. You can then go to “File”, then “Save As”, select your flash drive, name your spectrum, and click “Save”. Note that the saved document should be opened with Microsoft Word.

CHANGING SPECTRA APPEARANCE
1. To add gridlines, right click, choose “properties”, then “appearance”, then click gridlines option.
2. To change spectra color, right click, choose “properties”, then “appearance”, then click color option under curves.
3. To change from overlay/stacked options, go to “view”, then “overlay/split”.
4. To change the scale, click “View”, then “format graph”. This will allow you to modify the top, bottom, left, or right scales.
APPENDIX III. MEASUREMENT

In general, measurements are designed to obtain and communicate information about a system under investigation. A measurement will generate a datum or set of data (datum is singular, data plural) and that set of data is often used as a basis for making some type of decision. An experiment is a well-thought-out method designed to obtain the data necessary to answer a question or make a decision. We read a thermometer (an experiment or measurement) in the morning to obtain the temperature (a datum), which allows us to decide whether or not to wear our heavy jackets (a decision). If we do not have a thermometer, we can listen to the local weather to find out the current temperature. In this case, someone else has taken the measurement and is communicating the results to us, and we will make a decision based on our understanding of these results. The more effective the communication, the more sound our decision will be.

MEASUREMENTS

The information obtained by a measurement may be grouped into two categories, qualitative and quantitative. Qualitative measurements are general in nature, subjective, and open to interpretation and bias. They generally do not include numerical values or units. Examples like “the sky is blue” and “the water is hot” allow the recipient of the information to subjectively define “blue” or “how hot”, and these definitions may not be at all close to the definition intended by the person having done the experiment, resulting in a failure to communicate. These types of measurements may serve a purpose and are generally easier to perform, but the information communicated is less detailed and of less value than data from a quantitative measurement.

A quantitative result consists of both a number and a unit of measurement. The number provides a relative value for the measurement, while the unit of measurement precisely defines the quantity, or dimension, which provides the basis on which the relative value is defined. For example, a qualitative measurement of temperature may be “it is cold”, whereas a quantitative measurement would be “it is 1.2 °C”, where the 1.2 provides a relative value and the °C provides the units which precisely define the relative basis for the number. Notice how the meaning of “it is cold” can be significantly different if we simply change the units on 1.2 from °C to Kelvin, since 1.2 K is near absolute zero and much, much colder than 1.2 °C. Clearly, if you were told the temperature is 1.2 with no units provided, you would not be able to make any decisions based on this value until the question “1.2 what?” is answered.

ERRORS IN MEASUREMENT

Although data in the form of a number with the appropriate unit convey the most information, these data are still subject to errors and uncertainties, which must be identified, corrected if possible, and communicated. Errors associated with scientific measurements are normally classified into two types, indeterminate and determinate errors.
**Indeterminate Errors**

*Indeterminate errors* are always present, cannot be eliminated or their sources determined (hence the name), and become important when pushing the measurement technique to its limit. They are random, with an equal probability of being positive or negative, meaning above or below an average. These errors arise from the many uncontrollable variables present in a measurement and are a reflection of the natural limitations on our ability to physically take a measurement. Most often, the individual size of these errors is small and their sources are hard to isolate and identify, but the accumulation of many small indeterminate errors will affect the **precision** of measurements.

Precision communicates the reproducibility of the measurement, or how closely the data from several identical experiments are to one another. A common example of an indeterminate error, which affects precision, is the subjective visual interpolation of measured data, such as in reading a ruler or a buret, where the last number in the measurement is an estimate. If we measure the length of a stick more than one time, we may find our results to vary slightly due to a slight difference in the way we see the last figure on the ruler. These differences will be greater or less than some “average” value and these +/- deviations represent the indeterminate error. The more finely divided the divisions on the ruler are, the greater the limit of the measurement technique will be, and the indeterminate error associated with reading the ruler will be smaller. This relationship is illustrated in Figure 37 which shows ruler C to be the most precise and ruler A to be the least precise and most subject to indeterminate errors. If we average enough individual measurements, we can expect the random +/- fluctuations to cancel one another and reduce the total indeterminate error associated with the experiment.

![Figure 37](image)

**Figure 37.** Three rulers with different graduations and therefore different levels of precision, with A the least precise and C the most precise.
Determinate Errors

Determinate or systematic errors occur in the same direction (always positive or always negative in a systematic fashion), can be determined (hence the name), and impact accuracy. Accuracy is a measure of how close the experimental results are to a known “true” value, defined as a standard. In theory, these errors can be identified and compensated for in the results through a calibration procedure. Determinate errors are generally classified into three categories depending upon their source; instrument errors, method errors, or personal errors.

Instrument errors are inherent to the measuring device. For example, the markings on volumetric glassware are valid only for a certain temperature. If the glassware is used at a higher temperature, there is a slight expansion of the glassware and the volume increases slightly, introducing an error. In theory, we could calculate the expansion of the glassware at the new temperature and determine its effect on the volume, allowing us to correct for the error. Therefore, this error would be a determinate instrument error. Other common examples of instrument errors include using an electronic device that is not properly set to zero before use, pH meters that are not calibrated with an appropriate buffer solution, thermometers that do not read correctly, and electronic balances that always weigh either too high or too low. All of these errors could be eliminated by proper calibration.

Method errors are more difficult to identify and correct as they arise from nonideal chemical or physical behavior of the system under investigation. For example, the number of moles of a gas could be determined by measuring the temperature, pressure, and volume of the gas and employing the ideal gas law. However, if the gas under investigation deviates from ideal behavior, an error would be introduced. In theory, with some difficulty we could determine the extent of nonideal behavior and correct for this error. Other common examples of method errors include solubility loss when precipitating a solid for subsequent weighing, errors due to indicators in a titration because an indicator changes color only after the addition of an excess of reagent, and errors in procedures that assume reactions to go to completion when they do not. As in the case of instrument errors, all method errors could be identified and eliminated.

The third type of determinate error is one with which we are all familiar—personal errors. These determinate errors result from mistakes or errors in judgment made by the person doing the experiment. Errors of this type are exemplified by writing down the wrong data value, errors in calculations, sloppy laboratory work, and personal bias in reading data to give the most precise or accurate value, especially if you think you know what the answer should be. Personal errors are easy to correct by being careful, thoughtful, and disciplined when carrying out the laboratory procedure.

Identification, correction, and communication of indeterminate and determinate errors take different forms and these forms are discussed in the following sections.
**PRECISION**

The precision present in a set of data may be communicated in a number of ways, from the simple application of significant figure conventions to more complex considerations of statistical analysis. Several of these methods are reviewed in the following sections.

**Significant Figures**

The number of significant figures in a measurement conveys an indication of the relative reproducibility of the measurement. For example, assume we are to repetitively measure the length of the piece of wood shown in Figure 38 using the two rulers shown.

![Figure 38. The measurement of a piece of wood.](image)

When we use ruler A, the graduated divisions on the ruler allow us to clearly measure the length of the wood to be least 4 cm. We are not sure of the next digit in the measured value, but we can approximate that digit to be less than halfway to the 5, perhaps somewhere around 0.2. Although our approximation may not be exactly correct and will introduce some random error to the measurement (we could have guessed too high or too low), including the value provides more information than if we chose to ignore it (4.2 cm vs. 4 cm). The next time we perform the same measurement with ruler A, we again clearly see the wood is at least 4 cm in length, only this time we approximate the next digit to be 0.3, for a total length of 4.3 cm. As we perform more and more of these measurements, we always find the length to be 4.2 ± 0.1 cm. We could then write the average length as 4.2 cm. The final digit in the measurement is implied to be uncertain by ±1, and this is the last significant figure in the measurement. If we tried to add another significant figure and write out the length as 4.23 cm, we would realize that the final number 3 has no meaning because we never measured the wood stick to that degree of reliability, and in fact, we are not even completely sure about the preceding digit. Therefore, this number is not valid and should not be included in the value for the measurement.
If we use ruler C, we can see the length of the stick is clearly at least 4.2 cm but the next digit is not clearly delineated on this measuring device. Therefore, we approximate the next number and write down 4.21 cm for the measured value. The next time we take the measurement, we approximate the value as 4.23 cm. After several attempts at measuring the stick with this ruler, the average length is reported to be 4.21, with the 1 the last significant figure. With the more finely divided graduations on this ruler, the measured value can be reported to more significant figures.

In this example, we can clearly see that the number of significant figures is related to the precision, or reproducibility, of the measurement. The precision is dependent upon the measuring device, with ruler C providing more precision, and dependent to an extent on the person taking the measurement. If we wanted to measure the length of the stick to \( \frac{1}{1000} \) of a centimeter, we would need to invest in an expensive micrometer whose graduations are very carefully determined to \( \frac{1}{1000} \) of a centimeter. If we wanted to reduce the uncertainty associated with visually interpreting the micrometer, we could purchase an instrument that provides a digital (numerical) readout, although it is important to remember that the digital readout still retains uncertainty associated with the last digit in the measured value.

In obtaining data care should be taken to insure that all measurements and the calculations involving measurements include the proper number of significant figures. Too many significant figures communicate a higher degree of precision and reliability than is justified, leading to decisions made from data that is not valid. On the other hand, too few significant figures will needlessly throw away information that may have been obtained at a high cost. In general, the more significant figures associated with a measurement, the higher the cost of the instrumentation and the greater the effort required to obtain the data. The general rules for handling significant figures are provided in the next several sections.

To determine the number of significant figures present in a number, we simply count all the nonzero digits, all the zeros that are between nonzero digits, and zeros to the right of the decimal place. Zeros which precede the first nonzero digit simply hold the decimal place and are not significant, as is obvious when the number is written in scientific notation. Several examples are shown below.

\[
\begin{align*}
1.07 \times 10^5 & \quad \text{three significant figures} \\
10.45 & \quad \text{four significant figures} \\
0.045 \text{ or } 4.5 \times 10^{-2} & \quad \text{two significant figures (zeros to the left are not significant)} \\
12.000 & \quad \text{five significant figures}
\end{align*}
\]
Occasionally integers and precisely defined numbers are used in calculations. These numbers are assigned an infinite number of significant figures and so will not influence the determination of the number of significant figures in a resulting calculation. For example, there are exactly 12 inches in 1 foot as defined, so the 12 inches would have an infinite number of significant figures. The integer 2 could be considered to be 2.00000000000. . . . ., which also contains an infinite number of significant figures.

**Rounding Off**

When one or more digits must be dropped from a calculated result to give that result the proper number of significant figures, the following rules apply:

1. When the first digit to be dropped is less than 5, the last digit retained remains unchanged.

2. When the first digit to be dropped is greater than 5, the last digit retained is increased by 1.

3. When the first digit to be dropped is 5, the last digit retained remains unchanged if it is even, and is increased by 1 if it is odd. (This arbitrary rule is based on the assumption that on the average as many digits will be increased as will remain unchanged. Thus, any errors introduced in rounding off will tend to compensate for one another.)

These rules are illustrated by the following examples, in which the numbers are rounded off to three significant figures:

- 1.6723 rounds off to 1.67
- 1.677 rounds off to 1.68
- 1.665 rounds off to 1.66
- 1.675 rounds off to 1.68

**Multiplication and Division**

As a general rule, the answer to a calculation involving multiplication or division should contain the same number of significant figures as the factor with the smallest number of significant figures in the calculation. For example, in the problem:
the least precisely known factor is 5.62, which has three significant figures. The answer to the problem therefore contains three significant figures. There are some exceptions to this general rule. For example, consider the calculation

\[
\frac{34.4 \times 9.5}{252.7} = ?
\]

The solution to this problem to four figures is 1.218. The least precise factor in the calculation is 9.5, which contains only two significant figures. Thus, according to our rule the solution should have two significant figures; that is, we should round off this particular answer to 1.2. But our least precise factor of 9.5 has a precision of 1 part in 95, whereas our answer of 1.2 implies a precision of only 1 part in 12. If we retain an additional digit in our answer, it becomes 1.22 and indicates a precision of 1 part in 122. We are justified in doing this because a precision of 1 part per 95 is closer to 1 part per 122 than to 1 part per 12. The correct answer to the calculation, then, is 1.22. This example illustrates the important point that the rules for significant figures are not hard and fast, but often involve a personal judgment as well.

**Addition and Subtraction**

In addition and subtraction, the rule for determining the proper number of significant figures in the answer is different from the rule that is applied in multiplication and division. When numbers are added or subtracted, the units of those numbers must all be the same; that is, we cannot add pounds and inches or subtract grams from milliliters. The least precise number is not necessarily the number with the fewest significant figures; instead, it is the number with the fewest digits to the right of the decimal point.

The basis for this statement is clarified in the following illustration. Assume that we have determined the weights of a number of objects in grams, and we wish to total these to find the combined weight of the objects. The individual weights and the precision of each are:

\[
\begin{align*}
1.02 \text{ g} & \pm 0.01 \text{ g} \\
107.3 \text{ g} & \pm 0.1 \text{ g} \\
14.273 \text{ g} & \pm 0.001 \text{ g} \\
0.12 \text{ g} & \pm 0.01 \\
122.713 \text{ g} & = 122.7 \text{ g} \pm 0.1 \text{ g}
\end{align*}
\]

The least precise weight in the column is 107.3 g (although it is not the number with the fewest significant figures). Thus, the sum of these weights must indicate the same precision that is indicated in the 107.3; that is, \( \pm 0.1 \) g.
The rule for addition and subtraction, then, is that the solution must have the same number of digits to the right of the decimal point as the number in the problem with the fewest digits to the right of the decimal point.

**Addition/Subtraction with Multiplication/Division**

The proper significant figures for the results of a mathematical procedure involving both addition/subtraction and multiplication/division may be found by carrying out all additions/subtractions with proper significant figures, followed by the multiplication/division operations. These types of calculations are illustrated below:

\[
\frac{(12.34 + 1.2) \times 2.19}{9.0134} = \frac{13.5 \times 2.19}{9.0134} = \frac{29.6}{9.0134} = 3.29
\]

\[
\frac{14.894 \times 1.3 \times 10^7}{(2.189 - 0.91)} = \frac{14.894 \times 1.3 \times 10^7}{(1.28)} = \frac{1.9 \times 10^8}{(1.28)} = 1.5 \times 10^8
\]

Because calculators generally show as many digits as their display field will hold, there is a tendency to provide too many significant figures. Figure 39 shows a calculator display that results when determining the number of moles of copper present in a 0.2439-gram sample, which is calculated by dividing the mass of copper by the atomic weight of copper (63.546 g/mol).

**Figure 39.** Calculator showing too many significant figures in its display, given the number of significant figures in the original data.

The proper number of significant figures in this calculation is 4, and the value you should report would be 0.003838 or 3.838 \times 10^{-3} moles copper. Remember, just because your calculator provides you with many digits, it doesn’t mean they all have meaning and should be reported.
MEAN AND MEDIAN

After expressing each of our results with the proper number of significant figures, we still need a method for expressing a final result from a series of identical measurements, each of which has some indeterminate error. We would like to communicate the “central” value of our series of measurements because this value represents the best single description of the total data set. There are two common methods for obtaining a single value from a series of measurements—taking the mean and taking the median.

The mean value of a set of measurements is simply the average of all of the measured values. The mean provides a good estimate of the central value for data sets that are large or for small sets whose values are all relatively close. However, if the data set is small and contains a value that is very different from the others, the mean value becomes distorted in the direction of the outlying value and the measurement that is very different has an impact on the mean that is unduly large. For example, if we have the following four measured values, 4.0, 3.0, 1.0, and 10, the mean would be 4.5. Now, consider how well the mean of 4.5 represents this data set. Of the four measurements, three are below the mean. Only the measured value of 10 exceeds the mean value. Clearly, the value 10 has had the greatest influence on the data set and has shifted the mean to an artificially high value that is not a very good representation of the overall result of the four measurements. Under these circumstances, an alternative method of expressing the final result, the median, would be useful.

The median of a set of data is defined as the middle measurement value in an odd number of measurements, or the average of the middle two measured values in an even number of measurements. Exactly half of the measured values will be greater than the median and half will be less than the median. In our previous example with measured values of 4.0, 3.0, 1.0, and 10, the median would be the average of 3.0 and 4.0, or 3.5. Now, two of our measured values are less than the median and two are greater. With this set of measurements, the median provides a better single representation of the data than the mean. Use of the median value tends to minimize the influence of a single measurement that is much different than the others and is normally used when reporting the results from small data sets.

The difference between each measurement and the mean or median of a data set can be expressed in terms of absolute or relative deviation. These values provide a method for communicating the extent of indeterminate error associated with the procedure used for the measurements. The lower the deviation, the greater the precision and the lower the indeterminate errors. The absolute deviation is the difference between an individual measurement, $x_i$, and the mean or median value $\bar{x}$ and would have the same units as the measured quantity.

$$\text{Absolute deviation} = x_i - \bar{x}$$
Relative deviation is defined as a ratio of the absolute value of the absolute deviation over the average value and is often expressed in terms of a percentage or in terms of parts per thousand (ppt).

Relative deviation (percent)  =  \( \frac{|x_i - \bar{x}|}{\bar{x}} \times 100\% \)

Relative deviation (parts per thousand)  =  \( \frac{|x_i - \bar{x}|}{\bar{x}} \times 1000 \) ppt

The relative deviation is a more effective means of communicating the indeterminate error associated with different measurement procedures because the quantity of deviation is not dependent on the size or units of the measurement. For example, assume that the absolute deviation of a measured volume was 10.0 mL in two experiments. In the first, the total average volume was 100 mL while in the second it was 1000 mL. Although the absolute deviations in the two experiments are the same, the relative deviation for the second experiment is ten times less (1% vs. 10%) than the first experiment and it is clear the second experimental procedure has less indeterminate error associated with it than the first. Clearly, use of the relative deviation more effectively communicates the errors in the two experiments.

Because data sets frequently contain many values, average deviations and average relative deviations are often determined. These values are simply the average of the absolute or relative deviations for each of the individual measurements.

Another, more sophisticated, method used to express precision is by the **standard deviation**. The standard deviation for a large set of measurements is termed the population standard deviation, \( \sigma \), and is defined as

\[
\sigma = \sqrt{\frac{\sum_{i=1}^{N}(x_i - \mu)^2}{N}}
\]

where \( \mu \) is the “true average” value (the average value if we were able to take an infinite number of measurements), \( x_i \) is the value for each individual measurement, and \( N \) is the number of measurements. The term “standard deviation” arises from empirical studies of the frequency of occurrence of deviations (indeterminate errors) of a certain size as the same measurement is repeated over and over.

For example, assume that we measure the lead concentration in a municipal water supply 22 times and obtain the data listed in Table XIII, which has a mean value of 18.2 parts per billion (ppb). Due to random errors, each individual measurement deviates by some amount from the mean and these deviations are also provided in Table XIII.
### Table XIII. Data for the determination of lead in water.

<table>
<thead>
<tr>
<th>Value (ppb)</th>
<th>Deviation from Mean (ppb)</th>
<th>Value (ppb)</th>
<th>Deviation from Mean (ppb)</th>
</tr>
</thead>
<tbody>
<tr>
<td>18.1</td>
<td>-0.1</td>
<td>18.4</td>
<td>+0.2</td>
</tr>
<tr>
<td>17.9</td>
<td>-0.3</td>
<td>18.0</td>
<td>-0.2</td>
</tr>
<tr>
<td>18.5</td>
<td>+0.3</td>
<td>18.2</td>
<td>0.0</td>
</tr>
<tr>
<td>18.2</td>
<td>0.0</td>
<td>17.9</td>
<td>-0.3</td>
</tr>
<tr>
<td>18.3</td>
<td>+0.1</td>
<td>18.2</td>
<td>0.0</td>
</tr>
<tr>
<td>18.3</td>
<td>+0.1</td>
<td>17.8</td>
<td>-0.4</td>
</tr>
<tr>
<td>18.6</td>
<td>+0.4</td>
<td>18.1</td>
<td>-0.1</td>
</tr>
<tr>
<td>18.2</td>
<td>0.0</td>
<td>18.4</td>
<td>+0.2</td>
</tr>
<tr>
<td>18.1</td>
<td>-0.1</td>
<td>18.0</td>
<td>-0.2</td>
</tr>
<tr>
<td>18.6</td>
<td>+0.4</td>
<td>18.2</td>
<td>0.0</td>
</tr>
<tr>
<td>18.1</td>
<td>-0.1</td>
<td>18.3</td>
<td>+0.1</td>
</tr>
</tbody>
</table>

We can plot a bar graph of the frequency of occurrence of each deviation versus its magnitude (Figure 40). Figure 40 shows an experimental Gaussian or normal error curve beginning to take shape. However, due to the limited number of experimental measurements, this error distribution is not smooth and has not yet reached its theoretical shape. If, instead of taking only 22 measurements we took an infinite number, the frequency versus magnitude curve would become smooth and symmetrical as shown in Figure 41. (Remember that when discussing graphs, the dependent variable, plotted on the y-axis, is normally listed first and the independent variable, plotted on the x-axis, is given second. Thus, a plot of A vs. B implies A is on the y-axis and B is on the x-axis). The standard deviation for this set of measurements is calculated to be 0.2 ppb.

![Figure 40](image-url)
Gaussian curves have a number of properties of interest. First, they are symmetrical, centered about a deviation of 0, which means that the indeterminate errors are most likely to cancel one another to provide zero deviation. Second, the width of the curve is directly proportional to the standard deviation, providing the mathematical basis for the term's definition. Experiments with greater indeterminate error provide results that show a greater deviation from the mean, and hence a broader error curve and a larger $\sigma$ value. Clearly, then, the magnitude of $\sigma$ is a measure of the precision of the measurement.

If we slightly modify the manner in which we plot the data, another interesting and useful property of normal error curves may be visualized. We can convert the magnitude of deviation values plotted on the x-axis in Figure 42 into relative number of standard deviations. For example, a deviation of $-0.2$ ppb becomes $-1\sigma$ (the standard deviation of the data is 0.2 ppb so $-1 \times 0.2$ ppb = $-0.2$ ppb) and a deviation of $+0.6$ is equivalent to $+3\sigma$. If we then plot the frequency of occurrence values as a function of the number of standard deviations from the mean, a universal error curve results which is no longer dependent on the specific experiment performed or the specific measured value's units, but rather is valid for any set of data with the same standard deviation. The same curve would result if we measured parts per million, grams, ounces, or whatever, as long as the standard deviation of each data set was 0.2 whatever. It turns out that 68% of all measured value will be contained within the region of the curve bounded by $\pm 1\sigma$, while 95% of all values are within $\pm 2\sigma$. Such a curve for this data set is shown in Figure 42.
Figure 42. Error curve plotted with x-axis in units of $s$.

If the number of measurements in the data set is small (i.e., <20), the standard deviation term is slightly redefined as the sample standard deviation, $s$, which is given by,

$$
s = \sqrt{\frac{\sum_{i=1}^{N} (x_i - \bar{x})^2}{N - 1}}
$$

where $\bar{x}$ is the measured average of the measurement set. This definition provides a slightly broader error curve to compensate for the fact that with fewer measurements the expected error is somewhat larger than that expected with many measurements. As $N$ increases, the difference between $N$ and $N-1$ becomes less pronounced and $s$ approaches $\sigma$. Both terms provide a measure of precision, with the larger the standard deviation the less precise the measurement. More thorough treatments of the development of the standard deviation term are available in textbooks on statistics and advanced analytical chemistry texts.

Looking at either definition of standard deviation, we can see that the larger the errors ($|x_i - \mu|$), the larger the standard deviation. We can also see that as more measurements are taken, $N$ will increase and the size of the standard deviation must decrease. This relationship makes sense because as we take more measurements we are increasing the likelihood that the random errors will cancel one another and reduce the overall indeterminate error. As $N$ approaches $\infty$, the standard deviation will approach 0 and no indeterminate error will be present. Therefore, we can always reduce the impact of indeterminate errors on a measurement set by increasing the number of measurements. However, the standard deviation is dependent on the $\sqrt{N}$, so there comes a point of diminishing returns when increasing the number of measurements. For example, going from 1 to 4 measurements should reduce the standard deviation by a factor of 2, a fairly substantial improvement for only 3 more measurements. Going from 1 to 9 measurements
increases precision by a factor of 3, and going from 1 to 100 improves precision by a factor of 10. It took 96 more measurements to improve the precision from a factor of 2 to a factor of 10. If we wanted to improve precision by another factor of 2, to a factor of 20, we would need 400 measurements, and if we wanted to improve precision by a total factor of 50 relative to 1 measurement, we would need 2500 measurements. Clearly, for each subsequent improvement in precision, we need to take increasing numbers of measurements. Thus, there comes a point at which the increased precision available by taking more measurements is not worth the time, effort, and cost of the additional measurements required.

Each set of measurement data will always have some indeterminate error associated with it. The extent of the error will be determined both by the fundamental indeterminate error associated with the measurement procedure, described by $\sigma$, and by the number of measurements taken. The “true” mean value with no indeterminate error present will lie within some range of our measured mean, with the range determined by (1) the standard deviation, (2) the number of measurements, and (3) the statistical likelihood of finding the true value in this range. The important points to recognize in our discussion are that the measured average is not necessarily the “true” average, therefore the results of measurements are often expressed in terms of a range which takes into account the indeterminate error. By minimizing the sources of indeterminate error or by taking more measurements this range can be narrowed. The narrower the range, the more precisely we can communicate the results of our measurements, and the more useful the data become. Keep in mind that data can be no more precise than the instruments used to obtain them. If our thermometer only reads to ± 1 °C, reporting a temperature of 23.4591 °C is nonsense, and it should be reported as 23 °C. In addition, if we have not calibrated the thermometer, it may not be reading accurately and we cannot assure that the temperature is actually 23 °C.

**ACCURACY**

Even if our measurements are very precise and we get the same result every time we perform the measurement, they may not be very accurate. As you may recall from our earlier discussion, accuracy is defined as the difference between the experimental result and some known “true” value and is impacted by the presence of determinate errors. The **accuracy** of a measurement is normally expressed in terms of either an absolute or relative error, much like absolute and relative deviations express indeterminate error. An absolute error is defined as the difference between the “true” value ($\mu$) and the measured quantity ($x$) and has the same units as the measured quantity.

\[
\text{Absolute error} = x - \mu
\]

**Relative error** is defined as a ratio of the absolute error over the true value and is often expressed in terms of a percentage or in terms of parts per thousand (ppt).

\[
\text{Relative error (percent)} = \frac{x - \mu}{\mu} \times 100\%
\]
Relative error (parts per thousand) = \( \frac{x-\mu}{\mu} \times 1000 \text{ ppt} \)

The “true” value for a measurement is determined by using an appropriate standard whose measured quantity is well-known. For example, a thermometer can be tested for determinate errors by immersion in a beaker of boiling water at 1.0 atm. Because the boiling point of pure water at 1.0 atm is a well known quantity of 100.0 °C, the boiling water can serve as our standard with a “true” temperature. If, after equilibration in the boiling water, the thermometer reads 97.8 °C, we know there is a determinate error of 2.2 °C

\[
\text{Relative error} = \frac{2.2 \degree C}{100.0 \degree C} \times 100\% = 2.2\%
\]

associated with the measurement when using this thermometer. This is an example of an instrumental determinate error. We could correct this determinate error by adding 2.2 °C to all temperature values obtained with this thermometer. Of course in correcting the readings of our thermometer using error data obtained at 100.0 °C, we are assuming that the error in the thermometer remains constant over our range of application. This may not be a very good assumption. To test the assumption, we can prepare a mixture of pure water and ice. We know that at 1.0 atm a mixture of ice and water will have a temperature of 0.0 °C so we have a second standard to test our thermometer against. If our assumption was correct and the error is constant regardless of temperature, the thermometer should read –2.2 °C. However, if the temperature now reads 0.9 °C, the error associated with the thermometer is not constant but is temperature-dependent. In order to correct this error, the magnitude of the error for each measured temperature would be necessary. We can obtain this information by taking the temperature of a series of standards with known temperatures (possibly prepared by using a thermometer known to be very accurate) and developing a mathematical relationship between the measured temperature and the known temperature. The mathematical relationship between a measured quantity and a known value is often determined by graphical plotting of a “calibration curve” and the entire procedure is one method of calibration.
APPENDIX IV. EQUIPMENT/GLASSWARE

CHEMISTRY 111/112 EQUIPMENT LIST

1. 2 Beakers, 50 mL
2. 1 Beaker, 100 mL
3. 2 Beakers, 150 mL
4. 2 Beakers, 250 mL
5. 2 Beakers, 400 mL
6. 2 Beakers, 600 mL
7. 1 Bottle, screw cap, 30 mL
8. 2 Bottles, screw cap, 500 mL
9. 2 Bunsen burners
10. 1 Buret, 50 mL, teflon stopcock
11. 1 Condenser, 400 mm
12. 2 Crucibles, porcelain, size 0
13. 2 Crucible lids
14. 1 Crucible holder, Walter
15. 1 Desiccator, with porcelain plate
16. 1 Dish, crystallization, 90x50 mm
17. 1 Dish, evaporating, porcelain
18. 1 Flask, distilling, 50 mL
19. 1 Flask, Erlenmeyer, 50 mL
20. 3 Flasks, Erlenmeyer, 250 mL
21. 1 Flask, Erlenmeyer, 500 mL
22. 1 Flask, filter, 500 mL
23. 1 Flask, volumetric, 100 mL
24. 1 Flask, volumetric, 500 mL
25. 2 Funnels, filtering, 65 mm
26. 1 Funnel, stemless
27. 1 Funnel, Büchner, 5.5 cm, with rubber stopper
28. 1 Graduated cylinder, 10 mL
29. 1 Graduated cylinder, 100 mL
30. 2 Medicine droppers
31. 1 Pipet, volumetric, 5 mL
32. 1 Pipet, volumetric, 25 mL
33. 2 Rubber policemen
34. 1 Spatula
35. 2 Stirring rods, glass
36. 8 Test tubes, 10x75 mm
37. 4 Test tubes, 16x150 mm
38. 1 Test tube block, large
39. 1 Test tube block, small
40. 1 Test tube holder
41. 1 Thermometer
42. 1 Tongs
43. 3 Tubing, rubber, 2' lengths
44. 1 Wash bottle, polyethylene, 500 mL
45. 4 Watch glasses, 2-90, 2-100 mm
46. 2 Watch glasses, 125 mL
47. 2 Weighing bottles, 16 mL
LABORATORY GLASSWARE
(listed alphabetical left to right)

BOTTLE, WEIGHING
BURET
CONDENSER
CRUCIBLE, FILTER
CRUCIBLE HOLDER, WALTER
CRUCIBLE, PORCELAIN, WITH LID
APPENDIX V: WRITING STYLE

WORD USAGE

- Use of the passive voice and avoidance of the first person lend impartiality and objectivity to the report

  Instead of “I distilled the colorless liquid” use “The purple liquid was distilled.”; instead of “The average percent oxalate in my unknown is 14.73%” use “The average percent oxalate in the unknown was 14.73%.” Notice that in both examples the past tense indicates that you are reporting an observation or determination that has already occurred.

- In general, a sentence should not begin with a number.
- Use the proper subordinating conjunctions.
- “While” and “since” denote time. Do not use them where you mean “although”, “because”, or “whereas”. For example, it is better to say “Because only changes of absorbance are of interest, …” rather than “Since only changes of absorbance are of interest,…”
- All pages should be numbered with Arabic numerals for easy reference by you or by your instructor.
- The report should be generated by your word processing program and be printed and stapled.
- The subject and its verb must agree in number.

  Be aware of collective nouns that take a singular verb. Collective nouns take a singular verb when the group as a whole is meant; they take a plural verb when individuals of the group are discussed. “Data” is the most common collective noun in report writing. The following are examples of collective nouns:

| contents | dozen | none | range   |
| couple   | group | number | series  |
| data     | majority | pair | variety |

  For example, if you have explored four compounds you probably want to refer to each compound and therefore might say “a series of compounds were tested” rather than “a series of compounds was tested.” On the other hand if some property of the whole group is being compared to another group, you might say “the volatility of the series was compared to that of the series reported in the literature.”

  Units of measure are treated as collective nouns and therefore take a singular subject. Thus it would be correct to say “five mL of water was added” not “five mL of water were added.”

- Do not capitalize any chemical name or nonproprietary drug name unless it is the first word of a sentence or in a title or heading.
- Do not capitalize the common names of equipment (spectrophotometer, mercury lamp, gas chromatograph).
FORMATTING OF NUMERIC DATA

Units
Most numbers cited in a laboratory report are measurements that require proper units. Commonly used abbreviations are listed in Table XIV:

<table>
<thead>
<tr>
<th>Unit</th>
<th>Abbreviation</th>
<th>Unit</th>
<th>Abbreviation</th>
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<td></td>
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</table>

Numeral Usage
1. Use numerals with units of time or measure. Use a space between the number and the unit (except %). For example:

30 mL  150 mg mL⁻¹  0.30 g  25 °C  50%

2. With items other than units of time or measure, use words for numbers less than 10; use numerals for 10 and above, except as the first word in a sentence.

3. Do not begin a sentence with a numeral. Recast the sentence if possible. The sentence—Twenty milliliters of the mixture was added to the reaction vessel—does not provide an unambiguous indication of significant figures. Does twenty mean that the volume was measured to ±1 mL, to ±0.1 mL, or perhaps to four significant figures? It is therefore better to write it as: “A 20.0 mL aliquot of the mixture was added to the reaction vessel.”

4. Use an initial zero before a decimal; for example, 0.25.

5. In ranges and series, retain only the final unit of measure (for example, 25-30%).
# Periodic Table of the Elements

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*lanthanides

**actinides

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