

Chemistry 161 Laboratory Manual Introduction and Appendices

Table of Contents (this document)

Cascadia Learning Outcomes in the Chemistry Laboratory	3
CCC / UWB Safety Protocol	4
Safety Protocol Signature Page	6
The Laboratory Notebook	7 – 9
 Appendices:	
Volumetric Glassware: Use and care	10
Laboratory Glassware Tolerances	15
Making Use of Error Analysis	16
A Flow Chart for Calculating Errors	20
Features of A Good Graph	21
Formatting Scientific Graphs in Excel 2010	22
Linear Regression Routines	23

Individual experiments are contained in separate files which should be downloaded and printed for each lab period. The Table of Contents for these experiments follows on the next page of this document.

Learn Actively
Think Critically, Creatively and Reflectively
Communicate with Clarity and Originality
Interact in Diverse and Complex Environments

Tentative for Fall, 2018

ver 17.3

This manual was adapted from material developed, in part, under the National Science Foundation Division of Undergraduate Education, Applied Technology Grant DUE 9602403, Mary O'Brien and David W. Reichgott.



Table of Contents, continued in other documents

These documents are formatted for printing individual experiments two-sided on paper. Accordingly, there are occasional pages intentionally left blank, and PreLaboratory Assignments are always the last physical page and sheet of the printed document. These are to be completed and turned in at the start of each lab.

An Introduction to the Visible Spectrophotometer	26
Measurement and Error: Quality Control of Saline Solutions	35
Chemical Resource Lab	48
Spectrophotometric Analysis of Orthophosphate in Water	49
Reactions in Aqueous Solutions	57
Sodium Hydroxide Solutions: Preparation and Standardization	66
Heat of Reaction (Thermochemistry)	71
The Absorption and Emission of Light	85
The Titration of Vinegar: Design and Analysis	92

Cascadia Learning Outcomes in the Chemistry Laboratory

Chemistry laboratory provides a creative opportunity to put all the Cascadia Learning Outcomes to use at once. It also provides a setting which is very close to a workplace environment while still in an academic institution, and it illustrates how these Outcomes might be put into practice in the working world.

In this laboratory component of your General Chemistry course you will have opportunities to:

Learn Actively

- Learn experimental techniques and apply them to solve problems
- Apply “classroom” concepts in actual practice
- Learn to work safely

Think Critically, Creatively and Reflectively

- Work with appropriate preparation and attention to detail
- Evaluate your results at the same time they are being acquired
- Apply the techniques of error analysis to the selection of glassware and equipment, and to know when and when not to be accurate
- Make decisions about time and task management
- Make decisions about the management of wastes
- Reflect on the design of experiments, and where appropriate, create procedures of your own

Communicate with Clarity and Originality

- Communicate effectively with your co-workers in the laboratory
- Write clear entries in a laboratory notebook that would enable a co-worker to reconstruct your results
- Construct data tables in reports that communicate your measurements and results that communicate your findings
- Demonstrate sample calculations that clearly communicate your method
- Write clear summaries of findings in report form and in memorandum form

Interact in Diverse and Complex Environments

- Be an effective member of a group, contributing ideas, effort, and questions
- Listen to and consider the ideas and findings of others
- Maintain a concern for the safety oneself and of others
- Interact in a laboratory setting where students depend on each other, while coming from different personal cultures as well as different scientific cultures

The practice of Chemistry is fun. It combines science, art, technology, manual skills, insight, and creativity. There is a wonderful balance between attention to detail and creativity that will translate into skills in other scientific disciplines.

Welcome to the Cascadia College Chemistry Laboratory

All Chemistry students prior to beginning your work in these facilities must review the following Safety Protocol.

- No food or drink is allowed in laboratory facilities.
- Only students currently enrolled in Chemistry classes are allowed to use laboratory facilities.
- Protective splash-proof goggles must be worn at all times. Use protective gloves as directed by Instructor or Lab Technician. Avoid direct contact with chemicals.
- Sitting or lying down on the laboratory tables is not permitted.
- Locate the following safety items in the laboratory facility:
 - ❑ Fire Extinguisher
 - ❑ Fire Blanket
 - ❑ Eye Wash
 - ❑ Emergency Shower
 - ❑ “Emergency Procedures” chart
 - ❑ Telephone
 - ❑ Broken Glass Disposal Box
 - ❑ First-Aid kit
 - ❑ Evacuation procedures- know where to go in various emergency situations - ask instructor/lab technician
- A First-Aid Kit and Chemical spill response kits (Acid/Caustic/Solvent) are located in the laboratory. Report all accidents (e.g. cuts, spills or equipment damage) to your Instructor or Lab Technician immediately.
- Wear acceptable protective clothing which includes long pants or long skirts that cover the ankles, shirts that completely cover the torso and shoulders – front and back – preferably shirts with long sleeves or lab coats, and closed-toe shoes that fully cover the feet – tops of feet and back of heels. Clothing made of natural fibers is recommended. Loose fitting clothing and long hair should be tied back. Examples of non-acceptable personal protective attire: Open-toed shoes, sandals, “ballet” shoes, shorts, Capri pants, short skirts, nylons or tights, and tank tops are not permitted.
- Deposit all broken glassware in Broken Glass Disposal Box.
- Students must follow the specific lab procedures outlined by the Lab Instructor or Lab Manual. Additional experiments are not allowed.
- Prepare for lab in advance by studying experimental procedure and completing any pre-lab assignments BEFORE entering the laboratory. Always double check instructions when conducting experiment.

- **Check white-board for any specific instructions/safety information before starting procedure. Wait for the instructor's permission to begin lab work.**
- **Do not discard solid materials (e.g. paper towels, disposable pipets - use these items sparingly) or chemical waste in the lab sinks, and properly dispose of these materials as described by your Lab Instructor.**
- **Carefully read all labels on chemical reagents TWICE before dispensing and all labels on hazardous waste collection containers before discarding waste.**
- **To avoid contamination of reagents, never insert any implements into reagent bottles and do not return dispensed excess reagents to original container. Consult Instructor or Lab Technician for disposal instructions.**
- **Use fume extractor vents above lab station as directed by Lab Instructor.**
- **Do not leave flame or chemicals heating on hotplates unattended. Never evaporate chemicals to dryness over heat.**
- **Direct test-tubes away from self and others when heating.**
- **When observing odors, never place nose directly over sample. Instead, gently waft vapors towards nose using your hand.**
- **Lab materials are not to be removed from the laboratory facility. All chemical reagents, protective equipment or any other CC lab property must remain in the laboratory facility.**
- **SDS (Safety Data Sheets), formerly MSDS (Material Safety Data Sheet), for all chemical reagents are located in a labeled binder in the lab. Ask your Instructor or Lab Staff if you have any questions concerning chemical reagents or wish to have a copy of a particular SDS.**
- **Students are not allowed in the Bio/Chem Prep Room or Storage Areas unless accompanied by staff or faculty.**
- **When finished with experimental procedure:**
 - **Always check that gas valve is turned off completely and/or unplug hot plates.**
 - **Clean all glassware and wipe lab bench.**
 - **Wash hands thoroughly before leaving the laboratory facility.**

Please note that the above Safety Protocol is has been updated for Fall, 2017.

- **You will be provided with a course-specific declaration form at the start of your course for you to sign.**
- **The signed declaration will be collected by either your Instructor or a Lab Technician and retained on file.**
- **Please keep the Safety Protocol for future reference.**

The Laboratory Notebook

Laboratory notebooks are kept in virtually all academic, commercial, and government laboratories. Each laboratory's management will have specifications for notebook keeping that will vary depending on the need. These may range from simple log books maintained by multiple users to detailed chronologies of every step that a researcher may carry out. New protocols are being established by laboratories that keep computer records along with, or in place of notebooks.

A laboratory notebook serves a variety of purposes. It serves the keeper of the notebook by providing a source of data and procedures, both recent and past. It serves the co-workers of the keeper as a reference to the data and procedures, as well as a cross-reference to container labels that identify stored preparations. It serves the institution as a chronological as well as a legal reference to when and how experiments were carried out. This is particularly important when patent or legal issues may depend on the time and the outcome of laboratory work, and in these cases notebook pages are signed and witnessed routinely.

Because a laboratory notebook serves several purposes, the way it is maintained is a compromise in order to satisfy all the needs, yet remain practical. Procedures common to virtually all laboratory notebooks include:

- Existence of established protocols for the notebook by the institution.
- A type of notebook that is durable, and makes the insertion or removal of pages impossible.
- Identification of the "custodian" (the person responsible for the notebook).
- A requirement that all entries be in ink and that any corrections allow reading of the original entry.
- Page numbers and dates.
- A way of identifying pages, or portions of pages intentionally left blank.
- References to other essential printed material (such as a standard method, or in a teaching laboratory, to the pages in the course lab manual).
- Sufficient information to enable someone else to understand and reproduce the results, or produce the calculated outcomes of the measurements.
- Correlation between the labels of preparations and notebook pages and entries.

In our teaching laboratories, our protocol includes all of the above. You will be required to purchase a bound (not spiral) notebook approximately 20 cm x 15 cm with lined pages. Maintain a Table of Contents on the first page. If the pages are not numbered you will need to number them as you go. In addition, before coming to lab each week, make an entry giving:

- Date
- Title and source (usually, the lab manual page number) of the experiment.
- A brief statement of the purpose of the lab.
- Any calculations that were specified in the pre-laboratory assignment to record in your notebook.
- If there is one specific chemical reaction being studied, then a balanced chemical equation.

During the lab, you should enter in your notebook:

- Changes made during pre-lab lecture that affect how you conduct the experiment.
- The name of your partner(s), if any.
- Notes, observations, and data. The best way to enter data is by using neat, labeled tables wherever possible. You do not need to copy printed procedures into your notebook. Instead, note the page and the step number from this lab manual in your lab notebook, then the data, so

that any co-worker would have the ability to understand what it stands for. **Never** record raw data on separate pieces of paper or on the report form. If you need to change an entry, make a single line through the incorrect entry (so the original is not obscured), and initial the change. Never use "white-out" in a lab notebook.

- Entries are made in time-sequence; after-the-fact and "rewritten" entries are never used.
- If the method is not in the lab manual, make a complete entry of what was done.

Do NOT enter data on the Report pages until **all** experimental work and calculations are complete.

You may need to make a reference to a method, an instrument, or a computer file that provides or stores data and calculations. For example, after a table of data, you might make an entry that reads: "Linear regression of the above using Excel gives $A = 0.00876 c + 0.00124$ ", meaning that you fit a line to the data set using the computer program Excel. When data are obtained from an instrument, the data should be entered in the notebook, and the instrument should be identified. If the data set is unusually large, then key pieces of data sufficient to support your conclusions should be entered.

"Extraneous material" refers to separate pieces of paper that are logically linked to the entries in a notebook. Each laboratory and each course will have its own protocol for handling extraneous material. In Chemistry 161 / 162 / 163, do not attach extraneous material to your notebook. If you produce a graph, it should not be pasted, taped, stapled or placed loosely in the notebook. A data table sufficient to reproduce the graph should be entered, and the graph may be stored elsewhere. If an instrument's output is a piece of chart paper, the existence of the chart should be noted with only key data or conclusions from the chart entered in the notebook. If the instrument's output is a computer file, note the file name and/or follow any special instructions you may receive concerning electronic record keeping.

At the end of each lab, show your notebook to a classmate who is not your lab partner.

That individual will use the following criteria, assign a score, and initial the notebook as a "witness."

This is **purely an advisory score** and is not part of your course grade.

Have your instructor initial your witnessed notebook before you leave the lab.

Laboratory notebooks will be graded by your instructor twice during the quarter, with a value of 10 points each time, as follows:

Required Elements (2.5 points)

- Table of Contents
- Pages are numbered; entries directly follow previous lab date on next page.
- Date
- Title
- Page reference to lab manual
- Brief statement of purpose
- Calculations or quantities specified by the Prelaboratory assignment.
- Lab partner(s) – full name(s)

Clarity and Organization (7.5 points)

- Page references and procedure numbers accompany data entries
- Data entries are clear, organized, easy to read, and avoid unnecessary details
- Data are entered in time-sequence
- Corrections have a single line strike-through so both the original and corrected value can be read
- Corrections do not detract from organization

- Handwriting is clear (this is not a score for “neatness”)

We will use the following guidelines for grading, and for your peer assessment for each lab:

Score for:	2.5	2.0	1.5	1.0	0.5
Required Elements	Page, Date, Title, Purpose, Page reference, Prelab data, Partners are recorded, all in complete form.	Most elements always present, may occasionally miss one element.	Majority of elements present, one element routinely missing.	Minority of the required elements present in each.	One of the required elements is present in each.
Score for:	7.5	7.0	6.0	5.0	0 to 4.0
Clarity & Organization	Data are complete, entered in sequence in clear, organized manner. All references to procedures given. Someone else could easily reconstruct results. Corrections do not impair clarity.	Data are complete, entered in sequence in clear, organized manner. All references to procedures given. Someone else could easily reconstruct results. Corrections impair clarity occasionally.	Data are entered in clear manner. Minor organization problems. References to procedures inconsistent. Someone else could easily reconstruct results. Corrections impair clarity frequently.	Data mostly complete, generally clear. Minor organization problems. References to procedures inconsistent. Possible problem to reconstruct results. Corrections or legibility impair clarity.	Data missing or are entered in unclear or disorganized manner. References to procedures absent. Probable problem to reconstruct results. Corrections or legibility impair clarity.

The Laboratory Report

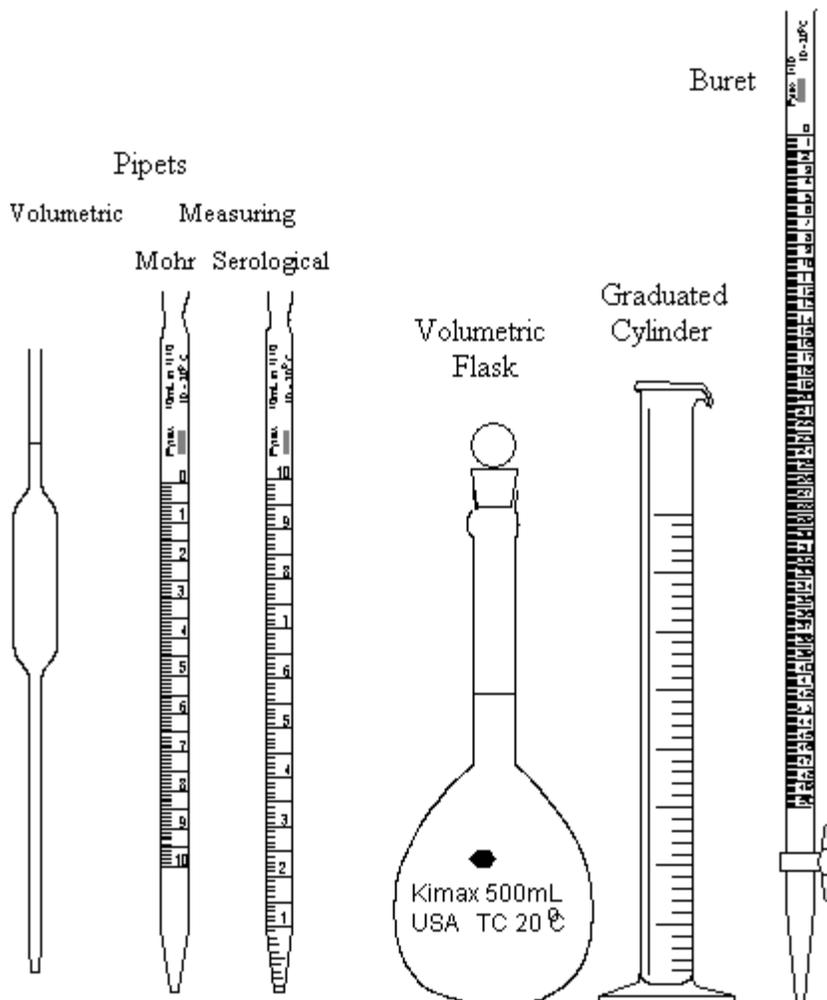
Your laboratory notebook should contain sufficient information, along with the instructions in the laboratory's procedure, to complete your laboratory report. Carefully follow the instructions in the laboratory's procedure concerning the report and any supplemental instructions your instructor may have given. The laboratory report will sometimes be completed on pre-printed sheets accompanying the procedure, sometimes on separate pieces of paper, and sometimes a combination of the two. It is **not** written in your notebook. If there are attachments (such as graphs) they should be **stapled** to the report.

VOLUMETRIC GLASSWARE: USE AND CARE

A. What is volumetric glassware?

Volumetric glassware is glassware designed to accurately measure volumes of pure liquids or solutions. There are four types of volumetric glassware that we will be using: graduated cylinders, volumetric flasks, pipets, and burets. Other laboratory glassware, such as beakers and Erlenmeyer flasks, is not considered volumetric glassware because it is not designed to accurately measure volume, but, instead, is used for measuring approximate volumes and for mixing reagents.

Typical examples of each piece of volumetric glassware are shown below:



Each of these pieces of volumetric glassware is available in various sizes. Pipets are available in various types, as well as various sizes. The types of pipets that you will likely encounter are the volumetric (transfer) pipet and the measuring pipet (Mohr or serological). Automated, digital pipets also exist, but will not be used in this class.

The obvious distinction between the volumetric and measuring pipet is that the volumetric pipet has only one calibration mark and will, therefore, deliver only one volume, whereas, the measuring pipet has multiple calibration marks and can deliver a wide range of volumes. The distinction between the Mohr and serological pipets is that the Mohr pipet is not calibrated to the tip and the serological pipet is calibrated to the tip. The serological pipet is, therefore, designed to be blown out and the Mohr pipet is not designed to be blown out. Pipets calibrated to be blown out will have a frosted band at the top or a double ring to emphasize this feature.

Volumetric glassware of two classes is available: Class A and Class B. Class A glassware is manufactured under more stringent calibration requirements than Class B glassware and is, therefore, more accurate. Class A glassware will have an "A" on the label. All volumetric glassware in our laboratory is Class A, with the exception of graduated cylinders. If there is no A on the label of the piece of glassware, assume that it is Class B glassware.

The label on volumetric glassware will generally also include "TD" or "TC". "TD" means that the glassware has been calibrated "To Deliver" the volume indicated. The "TC" designation means that the glassware has been calibrated "To Contain" the volume indicated. Volumetric flasks and graduated cylinders typically have the "TC" designation and pipets have the "TD" designation. A temperature will also be included in the label of volumetric glassware. This refers to the temperature at which the glassware was calibrated and is usually 20 °C. If the temperature of the materials being used is significantly different from the temperature indicated on the glassware, a correction would have to be applied. This will generally not be needed for our work.

B. How to Clean Volumetric Glassware?

We use volumetric glassware in applications where accuracy is important to the laboratory analysis. The accuracy of a piece of volumetric glassware may, however, be seriously compromised if it is not clean. When filled with water and emptied, a clean piece of volumetric glassware will have a uniform film of water on the inside of the glassware; dirt and grease will cause water to form beads instead of a thin, uniform film. You can assume that volumetric glassware issued to you in this laboratory will be clean and usually dry. If you use a piece of glassware several times with different solutions or if your glassware is wet, you should rinse it with a small amount of the new solution to be used. Typically, one rinse will be sufficient for our purposes, although most analytical procedures will recommend three rinsings with small volumes of solution.

All glassware should be rinsed with water and deionized water **immediately** after use. If glassware is allowed to dry out, cleaning is much more difficult. Should this occur, the glassware should be washed with a detergent solution whose concentration is no greater than 2%. Consult with your instructor if this is necessary. More concentrated detergent solutions should be avoided. Do not allow detergent to soak in the glassware for longer than 20 minutes at a time. Volumetric glassware should not be dried in an oven. If it is still wet, but needed for use, rinse with the solution to be used.

These comments on cleaning volumetric glassware are general. Certain analytical procedures will specify how the glassware is to be cleaned, and obviously, then those directions should be followed.

C. How to Use Volumetric Glassware?

Since volumetric glassware is used to accurately measure volumes of reagents, to accurately prepare solutions of a given concentration, and to accurately dilute solutions of known concentration, it should never be used to store solutions for longer than a few minutes since certain solutions actually attack glass. If a solution is to be stored for longer than a few minutes, it should be placed in a clean, dry bottle and immediately labeled. The label should include the name of the solution, its concentration, your name, the date of preparation, and the principal hazard category of the solute.

Using volumetric glassware requires that the liquid level in the glassware be read. This reading should always be performed in a consistent way to minimize errors. For aqueous solutions, the liquid level will not be flat, but will instead form a concave surface called the meniscus. The bottom of the meniscus should always be read and the bottom of the meniscus should always be aligned with the calibration line when that is appropriate. You should position your head eye level with the meniscus to avoid parallax errors. To assure that you are reading the meniscus in a consistent way, view the liquid level and meniscus against a background of contrasting color.

When using volumetric glassware with multiple divisions, such as graduated cylinders, measuring pipets, and burets, it is important to always read to the limit of the calibration. This implies that you read all the certain digits dictated by the calibration lines and that you estimate between the lines.

1. Graduated Cylinders

The graduated cylinder may be used to perform simple dilutions in which the accuracy of a pipet and/or volumetric flask is not required. While graduated cylinders are not as accurate as pipets or volumetric flasks, they can usually be read to three significant digits with precision. Because of the shape of graduated cylinders, they are not good mixing devices. Instead, solutions should be measured in the graduated cylinder and then transferred to a beaker or Erlenmeyer flask for mixing. Strongly basic solutions should be not stored in graduated cylinders for longer than a few minutes.

2. Pipets

Liquids should **never** be pipetted by mouth; instead, a pipet bulb should always be used. Some pipet bulbs and dispensers are designed to attach over the end of the pipet; the type we will use is designed to be placed firmly on the end without attaching it.

- Before placing the bulb on the pipet squeeze most of the air from the pipet bulb.
- The pipet bulb should then be placed firmly on the end of the pipet and the pipet placed in the liquid to be pipetted, as close to the bottom of the container as possible, to avoid drawing air into the pipet.
- Carefully allow liquid to be sucked up into the pipet. Liquid should be drawn past the calibration mark (but take care not to draw liquid into the bulb), and then quickly remove the bulb and place your finger over the end of the pipet. **Allowing liquid into the pipet bulb may destroy it.**
- Release the pressure of your finger to drain the liquid down to the mark.
- Before allowing the exact volume of liquid to be pipetted into the new container, touch the pipet tip against the wall of the container containing the liquid, and ensure that there is not an air bubble in the tip of the pipet. If any of these situations occur, the process must be repeated.
- Drain the liquid into the receiving container without forcing it out, holding the pipet vertically. During this entire operation be certain that the pipet is held vertically.
- Touch the tip of the pipet against the wall of the container. To insure consistent and complete drainage, continue touching the tip of the pipet to the inside wall of the container for 4-5 seconds after it appears that all the liquid has been delivered.

For pipets designed to be blown-out, force the liquid out only after the pipet has drained completely by gravity. (Blowing to increase the rate of delivery will change the volume of liquid delivered.)

Blow out the pipet using the pipet bulb, **NOT with your mouth!**

3. Volumetric Flask

Volumetric flasks are used for preparing solutions of a single, known volume and they are calibrated to contain that known volume of liquid. Solutions may be prepared either by diluting a known volume of a more concentrated solution or by dissolving a given amount of the solid solute in the solvent. In either case, it is critical that the final solution be well mixed. The shape of the volumetric flask makes it difficult to mix solutions well, so special care must be taken. To insure complete mixing, the volumetric flask should be inverted (keep finger on the cap) and swirled several times.

When preparing a solution from a solid, there are two methods that could be employed. The first requires weighing the solid directly into the volumetric flask, dissolving the solid with solvent with about the 2/3 of the flask full, and then filling to the mark once the solid has dissolved. This requires inverting and swirling as many as 12 times. The second method involves weighing the solid into a beaker, totally dissolving the solid in the minimum amount of solvent (a much smaller volume than the volume of the flask), and then quantitatively transferring the solution to the volumetric flask and filling the flask with solvent to the final volume. A quantitative transfer involves very carefully transferring the dissolved solute into the volumetric flask, then rinsing the beaker several times with a small amount of solvent, each time pouring that rinse into the volumetric flask. Special care must be taken with each transfer to insure that all the solute is transferred to the volumetric flask. Volumetric flasks should never be used to store or heat solutions.

4. Buret

Before beginning a titration, burets should be rinsed two times with a small amount (5 to 10 mL) of titrant. With the stopcock closed and the open end above a waste container, hold the buret at an angle and contact all the inner surfaces with the titrant. Then drain the rinse liquid through the tip of the buret into the waste container, stopping with the liquid level just above the stopcock. Repeat the process with a second small portion of titrant. Use a specially designed buret clamp to clamp the buret to the ring stand.

Fill the buret past the 0 mL mark and drain enough liquid into the waste container to bring the meniscus below the 0 mark. This should also fill the tip of the buret with liquid, which has a tendency to develop an air bubble. If the tip still has an air bubble, carefully jerk the buret with stopcock open, allowing some liquid to flow into the waste container.

Read the initial volume of titrant in the buret to the nearest 0.01 mL, noting that the buret is calibrated with 0 mL at the top, 50 mL at the bottom, and volumes increase going downward. Perform the titration by operating the stopcock with your left hand and continually swirling the Erlenmeyer flask with your right hand (if left-handed, vice versa). At the end of the titration, read the final volume of liquid in the buret to the nearest 0.01 mL. When using an indicator to detect the equivalence point, titrate against a white background. Use a card with a black mark to read the initial and final volumes of liquid in the buret, always reading the bottom of the meniscus.

At the end of a set of titrations, the buret should be emptied into a waste container, rinsed several times with deionized water and placed upside down in the buret clamp with the stopcock open.

Laboratory Glassware Tolerances

	Burets Class A	Flasks (Volumetric) Class A	Graduated Cylinders Class B	Pipet (Mohr) Class B	Pipet (Serological) Class B	Pipet (Volumetric) Class A
	Tolerance, ± ml	Tolerance, ± ml	Tolerance, ± ml	Tolerance, ± ml	Tolerance, ± ml	Tolerance, ± ml
capacity (mL) less than & including						
0.1				0.005	0.005	
0.2				0.008	0.008	
1		0.010		0.02	0.02	0.006
2		0.015		0.02	0.02	0.006
3		0.015				0.01
4		0.020				0.01
5	0.01	0.02	0.08	0.04	0.04	0.01
10	0.02	0.02	0.10	0.06	0.06	0.02
15						0.03
20						0.03
25	0.03	0.03	0.30	0.10	0.10	0.03
50	0.05	0.05	0.40			0.05
100		0.08	0.60			0.08
200		0.10	1.4			0.10
250		0.12	1.4			
500		0.15	2.6			
1000		0.30	5.0			
2000		0.50	10.0			
4000			18.0			

Tolerances are established on the basis of capacity only and are independent of subdivisions.

Tolerances of Class B glassware are twice as large as Class A glassware where not otherwise specified.

Volumetric glassware not labeled as Class A is assumed to be Class B.

Beakers and Erlenmeyer flasks have approximate volumes, about $\pm 5\%$ of measurement.

MAKING USE OF ERROR ANALYSIS

A. OBJECTIVES - The two most important objectives of error analysis are:

Communicating the range of values within which the true value of the answer is most likely to be found. This is usually written as a “confidence interval”: [the answer] \pm [the absolute error].

The calculated concentration was 0.023 ± 0.005 gram/liter.

Determining which step in a sequence of measurements most severely impacts the error in an experiment, and which steps do not. To make this determination, you compare relative errors. This helps you decide how to improve an experiment, if needed. It also impacts what kind of glassware you choose, where you take care in obtaining measurements, and where it is reasonable to work quickly or to take short-cuts.

*In my density calculation, the relative error in mass was 0.0002.
The relative error in volume was 0.004. I need a better pipet!*

B. WHAT ARE ABSOLUTE AND RELATIVE ERRORS?

1. Absolute Errors and Confidence Intervals

Absolute errors communicate the size of the error using the same units as the quantity being expressed.

*If the error in a 10.44 mL burette reading is 0.02 mL, the absolute error is 0.02 mL,
and the quantity would be expressed as $10.44 \text{ mL} \pm 0.02 \text{ mL}$.*

The \pm format is called a “confidence interval”. The error should have only 1 significant figure, and the same number of decimal places should be used for the quantity and its absolute error.

If a mass is determined to be 3.5685 g and the absolute error associated with that mass is ± 0.02 g, then the mass should be rounded and expressed as $3.57 \text{ g} \pm 0.02 \text{ g}$.

If scientific notation is needed for a confidence interval, the value and the absolute error should have the same power of ten, and the decimal places should match.

*If the error in a concentration of 2.35×10^{-4} g/L is 7.4×10^{-6} g/L,
the confidence interval should be expressed as $2.35 \times 10^{-4} \text{ g/L} \pm 0.07 \times 10^{-4} \text{ g/L}$.
This may also be expressed as $2.35 \pm 0.07 \times 10^{-4} \text{ g/L}$.*

Absolute errors may represent either accuracy or precision.

2. Relative errors

Relative errors are always dimensionless and are calculated by dividing the absolute error by the quantity. Relative errors may also be reported in % format, which is relative error $\times 100\%$.

*In the above example, the relative error would be $(0.02 \text{ g} / 3.57 \text{ g}) = 0.006$.
The % error is $.006 \times 100\% = 0.6\%$.*

Relative errors are often expressed as “parts per” to compensate for the lack of units. For example, our relative error of 0.006, which equals six thousandths, could be expressed as six parts per thousand or 0.6 parts per hundred (0.6%).

Relative errors may represent either accuracy or precision.

C. HOW ARE ABSOLUTE ERRORS OBTAINED?

When you take a measurement, or when you calculate an answer, there are several different ways in which absolute errors can be known. There is a definite hierarchy, which is outlined below:

1. Deviation from the true value (accuracy)

If a true value is known, the absolute error is the difference between a measurement and the true value.

If a length is known to be 2.72 cm (the true value) and you measure 2.70 cm, then the absolute error (or deviation) is the absolute value of the difference between the two, $|2.70 \text{ cm} - 2.72 \text{ cm}| = 0.02 \text{ cm}$.

2. From the manufacturer’s claim (accuracy)

Certain pieces of laboratory equipment will come from the manufacturer supplied with a tolerance for that piece of equipment. That tolerance is the absolute error for that item and it indicates the accuracy of the particular item because the manufacturer knows the true capability of the equipment. Glassware tolerances are indicated on the chart provided.

If you measure 5 ml of water using a volumetric pipet, you would record that volume as $5.00 \text{ mL} \pm 0.01 \text{ mL}$. The value 0.01 mL is the absolute error for the 5 mL volumetric pipet.

3. From an average value (precision)

a. Calculate the individual deviations:

If multiple measurements have been made of a single quantity, then the absolute error in a single measurement (a deviation) is the absolute value of the difference between that measurement and the mean.

A set of absorbance readings was 0.423, 0.426, and 0.429. The mean is 0.426. The deviation of the first reading is $|0.423 - 0.426| = 0.003$.

b. Calculate the average deviation (precision):

If multiple measurements of a single quantity have been made, then the average deviation may be obtained by averaging all the deviations. The steps to calculate the average deviation are:

- Calculate the average. Include non-significant digits.
- Subtract each value from the average and express the difference as an absolute value (+ sign). These are the deviations.
- Take the average of the deviations. Round the average deviation to one significant figure.
- Express the average (round) using the same decimal place as the average deviation.
- Write the average and the average deviation as a confidence interval.

This result gives the absolute error in the average of a set of measurements (as opposed to the absolute error in a single measurement as described in #3a).

*The deviations in #3a are 0.003, 0.000, and 0.003.
The average deviation is $(0.006 / 3) = 0.002$
The answer is written as 0.426 ± 0.002 .*

*If the mass of an object is measured five times and the mean is found to be 5.6732 g and the average deviation is calculated to be 0.003g, then the mass should be reported as $5.673 \text{ g} \pm 0.003 \text{ g}$.
The 0.003 g is the absolute error in the precision of the measurements.*

In larger data sets, the standard error (based on the standard deviation) will be used in place of the average deviation. (Confidence intervals based on the standard deviation are not appropriate for small data sets for two reasons: the distribution of the data is not “Gaussian”, and the “t-value” on which the standard error’s confidence interval is based, is very large for small data sets. It is appropriate to use standard error for data sets with greater than about five values.)

4. From experimenter’s estimate of error (precision):

If a true value of a measurement, or a manufacturer’s claimed tolerance, or multiple measurements are unavailable, then the experimenter must estimate the absolute error for a given measurement. This estimate takes into account the experimenter’s judgment in making the measurement, their practice in doing so, and their ability to read between the graduations on a scale in a reproducible manner. (Since it represents reproducibility, it is an estimate of precision.) When estimating values between graduations on a scale, it is typically possible to estimate one-fifth the distance between markings and with practice it can be possible to estimate to one-tenth of a division.

*In measuring the length of a line using a centimeter ruler with marked divisions of 0.1 cm, one could report the length as $5.32 \text{ cm} \pm 0.02 \text{ cm}$.
The 0.02 cm is the absolute error in this measurement, estimated by the measurer.*

It is also related to the measurer’s ability to reproduce this measurement.

*I measured a mass using a balance that is rated by the manufacturer as $\pm 0.001 \text{ g}$.
However, when I took my reading the balance was fluctuating about $\pm 0.002 \text{ g}$.
I estimated my error (precision) as $\pm 0.002 \text{ g}$.*

D. HOW DO WE USE RELATIVE AND ABSOLUTE ERRORS?

If we are reporting error from a single measurement or a set of measurements of a **single** quantity, the preceding information should allow you to state the absolute error in a measurement and the relative error in a measurement. If, however, you are reporting an experimental result which was calculated from several measurements of different quantities, then the relative error in each measurement must be considered when expressing the absolute error in the final quantity reported. The largest relative error controls the absolute error in your experiment. The method that we will use for determining absolute error ensures that the quality of information in your answer can be no better than in the “worst” value of experimentally determined quantities.

(In the future you may learn that the statistically correct term is the square root of the sum of the squared errors. You can probably show that this is approximately equal to the value of the largest single error.)

Simply stated,

- identify the largest relative error in the variables used in your calculations;
- multiply this relative error by your answer to convert it to an absolute error;
- write down your answer and its error using consistent significant digits.

*I calculated that a concentration is 0.023184 gram/liter.
I also calculated that the largest relative error in my measurements was 0.0042.
My answer's absolute error is $(0.023184 * 0.0042) = .000097$ which rounds to .0001.
My answer is 0.0232 ± 0.0001 gram/liter.*

The overall method is shown in the attached flowchart.

The following example shows how relative errors in different measurements allow you to calculate the absolute error in a final answer. This example shows how to evaluate the data obtained from a density experiment.

One measurement of mass and one measurement of volume were taken to calculate a density.

Mass = $3.877\text{g} \pm 0.001\text{g}$ (the $\pm 0.001\text{g}$ is the manufacturer's claim of accuracy.)
Volume = 5.00 ± 0.04 mL (the ± 0.04 mL is the experimenter's estimate of precision.)

Density calculation (using consistent significant digits):

$$D = (m / V) = (3.877\text{g} / 5.00 \text{ mL}) = 0.775 \text{ g/mL (rounded to 3 s.d.)}$$

Relative errors:

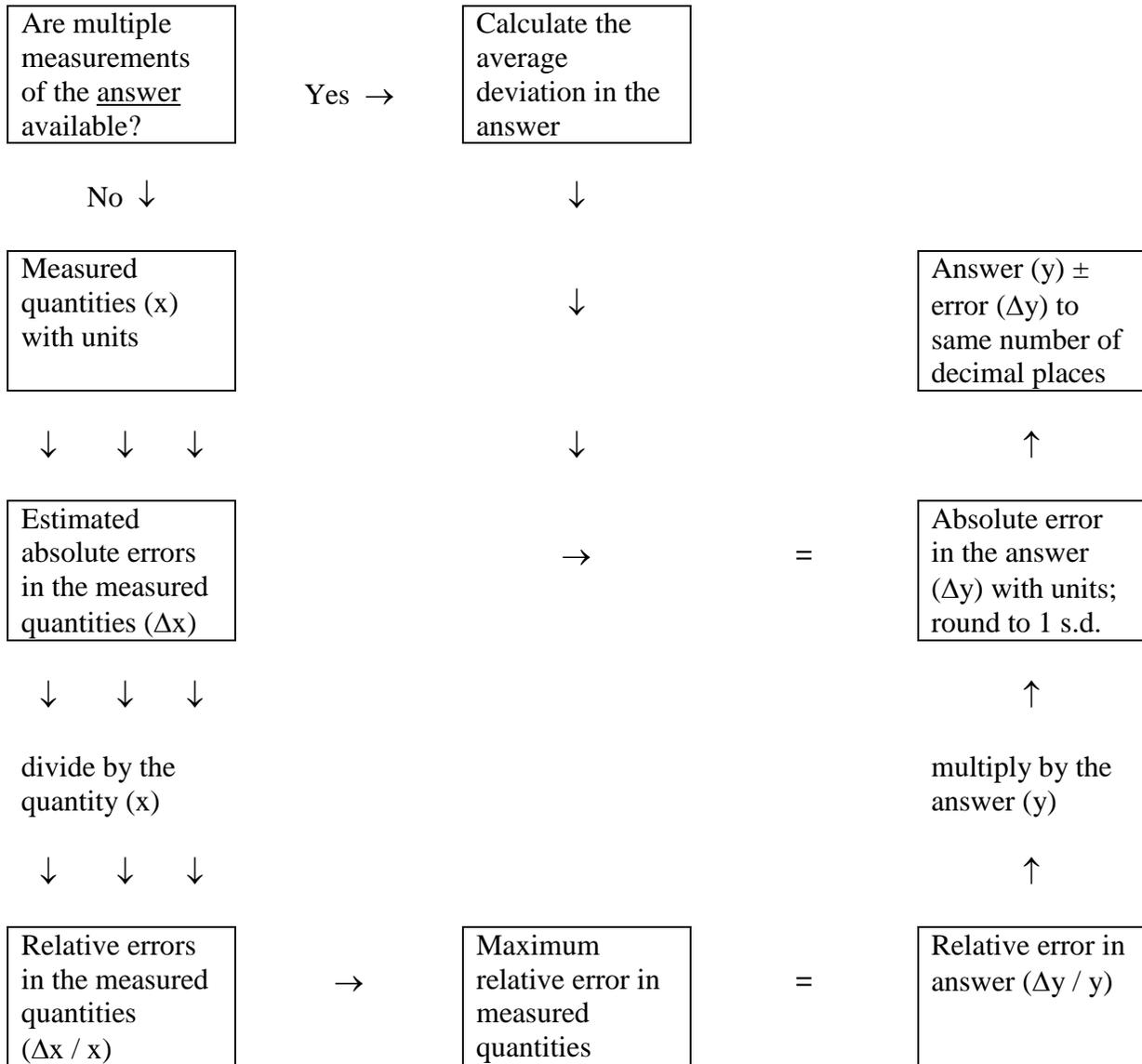
mass: $(0.001 \text{ g} / 3.877 \text{ g}) = .00026$ (keeping one extra "s.d." since we will round off later)
volume: $(.04 \text{ mL} / 5.00 \text{ mL}) = .0080$ (one extra "s.d.")

The largest relative error in the data is .0080;
therefore the relative error in the calculated quantity is 0.0080.

Since relative error = (absolute error) / (quantity)
absolute error = (relative error) * (quantity)
absolute error = $(0.0080) * (0.775 \text{ g/mL}) = 0.0062 \text{ g/mL}$
 $= 0.006 \text{ g/mL (rounded to 1 s.d.)}$

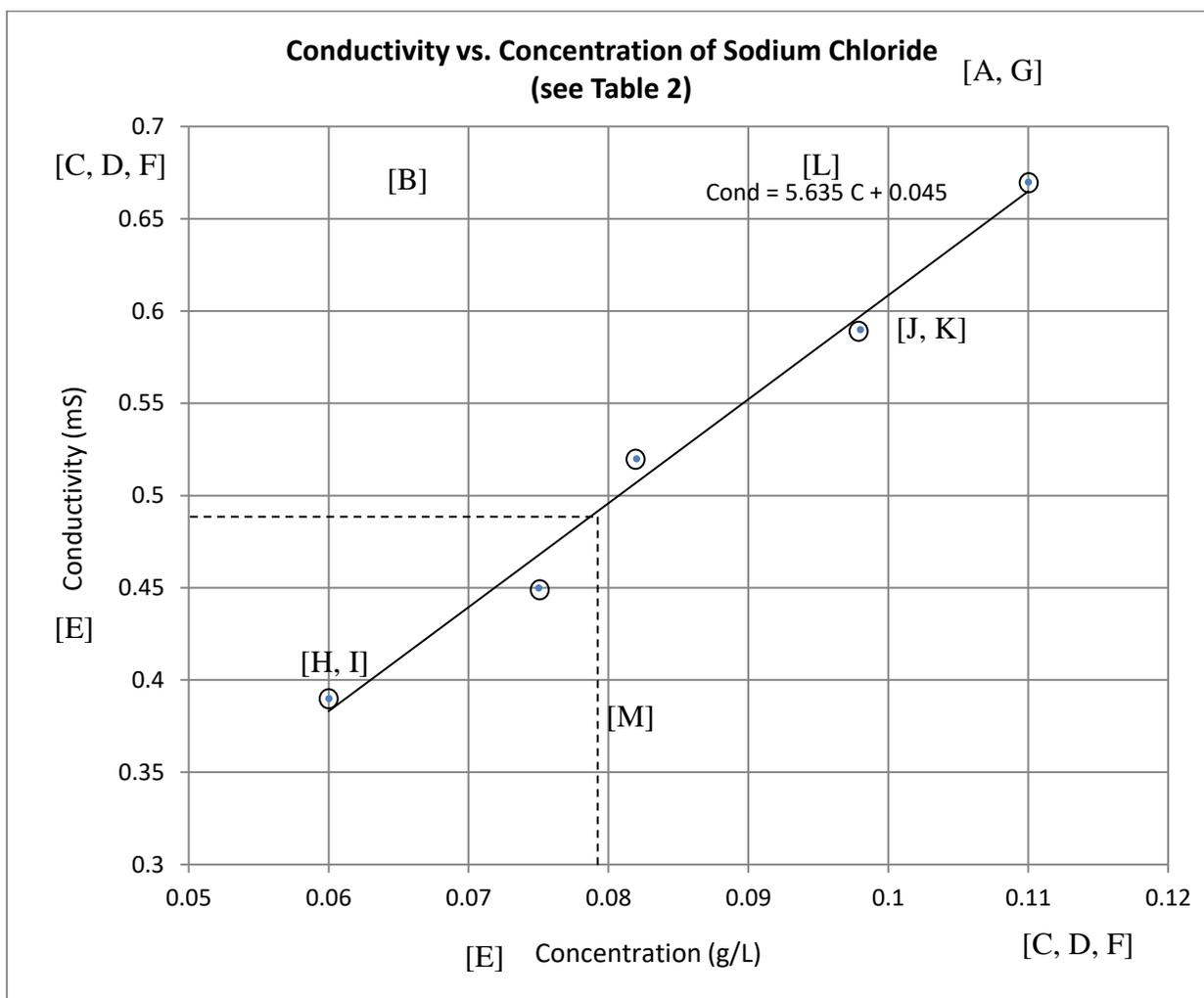
Report the density as $0.775 \text{ g/mL} \pm 0.006 \text{ g/mL}$.

A Flow Chart for Calculating Errors



Features of a Good Graph

Features indicated by letters in brackets; see key below.



- [A] Title.
- [B] Graph paper used or a computer graph with x and y grid lines.
- [C] The "X" axis is for the controlled (independent) variable; the "Y" axis is for the result (the dependent variable).
- [D] Axes are scaled so that the data "fit" and most of the area is used.
(The origin of the axes need not be at 0,0.)
- [E] Axes are labeled and have units.
- [F] Axes have evenly spaced "tic" marks on the outer edges, with labels having the same power of 10.
- [G] Data are included in a separate table, with a reference to where it is.
- [H] Data are not written next to the points on the graph.
- [I] Data points have "point protectors" such as a small circle or square
(unless there are many closely spaced data points).
- [J] A straight line of best fit or a smooth curve is drawn through the points
(not a point-to-point connection).
- [K] The line does not obscure any data points (drawn as a broken line if necessary).
- [L] The line has an equation written, if known, using the variables of the axes.
- [M] If the result for an "unknown" is shown, use a different format (such as a dashed line).

Formatting Scientific Graphs in Excel 2010

Excel is a powerful spreadsheet tool, but the “default” graphs it produces (called “Charts”) require several formatting steps before they take on the appearance of a good quality scientific graph. Most important, the “Scatter” graph option must be selected in order to obtain a properly spaced x-axis. The title is usually too big and the graph’s “legend” usually takes up too much space, so the graph is compressed. The default version also does not place x-axis grid lines on the graph space.

The following steps are pertinent to Excel 2010:

In spreadsheet, put x-data in first column, y-data in second column.

Select (highlight) these data.

Click Insert

Scatter

Choose the icon with no lines

From top bar, Chart Tools, click Layout, then select (as needed)

Chart Title (after typing a right click will be needed to reduce the font)

Axis Title (horizontal and “rotated” vertical)

Legend (usually select None)

Axes (for Horizontal and Vertical, choose More Axis Options)

Gridlines (Horizontal are present by default; need to add Vertical

Try either Major or Major and Minor, depending on appearance)

To add a regression line, click Trendline

Click Linear Trendline

Under More Trendline options, check Display Equation on chart

To print, have Chart selected (double outline) then File / Print

LINEAR REGRESSION ROUTINES

The purpose of linear regression is to obtain the single, best-fitting linear equation for a set of data. Linear regression routines are available on many advanced scientific calculators as well as in Excel and Vernier Graphical Analysis. (While these give slope, intercept, and correlation coefficient, they do not give error terms for slope and intercept. You may need to consult an advanced text to obtain these parameters.)

The following are the routines for Graphical Analysis, Excel, TI-82, TI-83 TI-84, TI-85, and TI-86:

SLOPES AND INTERCEPTS WITH “GRAPHICAL ANALYSIS”

Vernier Software’s “Graphical Analysis” is available on Cascadia’s computers for student use. You may also see the Library Reserve Desk for a copy to install at home under our site license. Enter your data in the X and Y columns and click on the regression icon (R=) at the top. (Data in scientific notation must use E instead of 10^). You can also select a smaller range of data by clicking and dragging over the range before performing the regression. In the output box, a capital E means 10 to the following power. If you use the program for a finished graph, double-click the “X” and the “Y” in the table’s column headings to format and label the axes; double-click on the graph to uncheck the connect line box and to add a title.

SLOPES AND INTERCEPTS WITH EXCEL:

SLOPE and INTERCEPT are recognized as functions in Excel. The format and an example are:

	A	B	C	D
1	1	1.1		
2	2	1.9		
3	3	2.9		
4	4	3.9		
5			0.94	
6			0.1	

In this worksheet, cell C5 would be entered as: =SLOPE(b1:b4, a1:a4)

C6 would be entered as: =INTERCEPT(b1:b4, a1:a4)

The slope and intercept will be computed using b1 through b4 as “y” values,
and a1 through a4 as “x” values.

Regression equations with slope and intercept values are also accessible through the “Trendline” option with Excel charts, but you may not obtain the desired significant figures.

LINEAR REGRESSION WITH TI-82:

1. STAT
2. Toggle to ClrList
3. ENTER
4. 2nd Function L1 (above #1)
5. ENTER
6. STAT
7. Toggle to ClrList
8. Enter
9. 2nd Function L2 (above #2)
10. Enter
11. STAT, EDIT
12. Enter
13. L1 (enter x data, toggle down after each entry)
14. Press ENTER, ENTER after last entry
15. Toggle to L2 (enter y data)
16. Press ENTER after last entry
17. WINDOW- set xmin and xmax and ymin and ymax
18. 2nd Function, STAT PLOT (above y =)
19. Plot 1, ENTER
20. Turn ON
21. TYPE: 1st box for scatter plot, then ENTER
22. Toggle to x list: L1 and y list: L2
23. Mark Lists
24. GRAPH
25. STAT, toggle to CALC
26. Toggle down to Lin Reg ($ax + b$), ENTER, ENTER
27. Read values for a, b

LINEAR REGRESSION WITH TI-83 or TI-84:

1. 2nd Mem #4 ClrAll lists
2. ENTER, ENTER
3. STAT
4. EDIT
5. ENTER, L1, L2, L3 should appear (if they do not appear, then use up toggle to get to the top of the display, then 2nd L2, Enter, toggle to middle of top display, 2nd L2, Enter)
6. Enter paired data into two lists (L1 for x's, L2 for y's); enter all x values, hitting ENTER after each value, then enter all y values in the same order, hitting ENTER after each value)
7. STAT
8. CALC (toggle to)
9. #4 LINReg ($ax + b$)
10. ENTER, ENTER
11. Record equation of line of best fit

LINEAR REGRESSION WITH TI-85:

1. STAT
2. F2 (Edit), ENTER, ENTER
3. F5 (Clr x y)
4. Enter x and y values
5. When x and y values are all entered, EXIT
6. DRAW (F3)
7. SCAT (F2) Set range before graphing
8. EXIT
9. CALC (F1) ENTER, ENTER
10. LinR (F2)
11. Read a, b ($y = bx + a$)

LINEAR REGRESSION WITH TI-86:

1. Press 2nd STAT
2. F2 (Edit)
3. Enter data in x and y stats columns. In fstats column enter a 1 for each row of entries you have in x and y stats columns (You should have a 1 in fstats for each xy coordinate).
4. Exit
5. Press 2nd STAT
6. Choose F1 (Calc)
7. Choose F2 (Two vars)
8. Press enter. Two variable fact screen will show at this time.
9. Choose F3 (LinR)
10. Press enter (linear regression shows)
11. Read a and b

If you want to see a graph of this on your screen:

12. Press exit once
13. Choose F4 (Draw)
14. Choose F3 (xyline) Graph will show at this point.