



Chemistry 163 Laboratory Manual: Introduction and Appendices

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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

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ver 17.1



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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.

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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 Community College/UW Bothell 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
 - ❑ Eye Wash
 - ❑ Emergency Shower
 - ❑ “Emergency Procedures” flip chart
 - ❑ Telephone
 - ❑ Broken Glass Disposal Box
- First-Aid Kit is located in the Bio/Chem prep area next door. 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.
- Loose fitting clothing and long hair should be tied back when using Bunsen burners. Open-toed shoes, sandals, short shorts and short skirts 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.
- 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 in to 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 CCC lab property must remain in the laboratory facility.**
- **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 MSDS.**
- **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 review the Safety Protocol, then print this one page, and read and sign the following declaration.

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.

I _____ (Print Name) have read and understand all of the information presented in the Safety Protocol. I understand that these policies are in place to insure the safety of all Cascadia students, staff and faculty as well as the protection of CCC property. Failure on my part to follow the Safety Protocol may result in me being denied access to CCC laboratory facilities.

Signed: _____ Date: _____
Parent or guardian must sign if student is under 18

Class: _____

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.

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 complete, clear, organized, easy to read, and avoid unnecessary details
- 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:

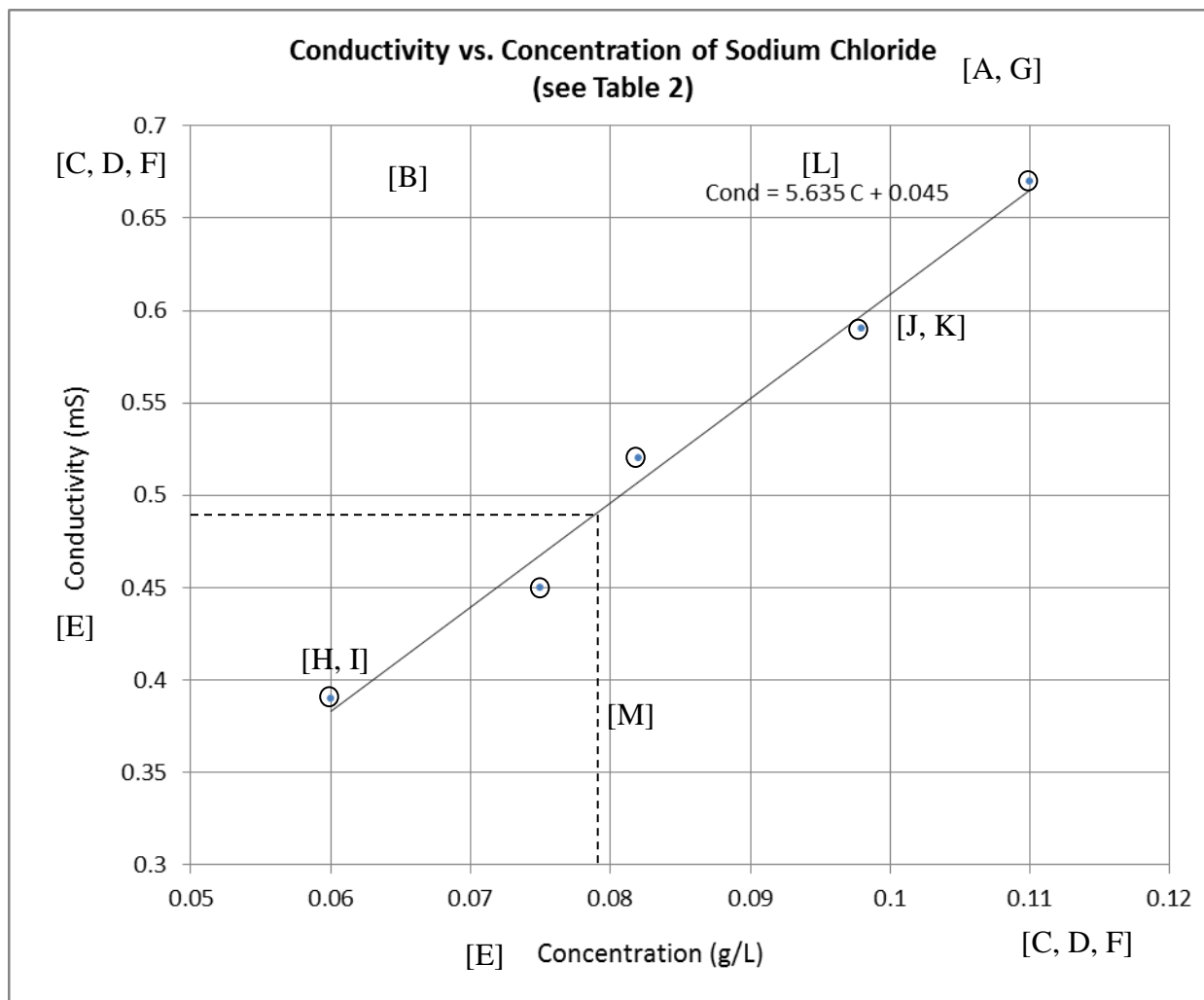
| | | | | | |
|------------------------|---|---|---|---|---|
| Score for: | 2.5 | 2.0 | 1.5 | 1.0 | 0.5 |
| Required Elements | Page, Date, Title, Purpose, Page reference, Prelab data, Partners are recorded, all in complete form. | Most elements always present, may occasionally miss one element. | Majority 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.

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).

Self-Instructed Exercise Using Vernier “Graphical Analysis”

Vernier Software “Graphical Analysis” is a user-friendly scientific graphing program. It is installed on each of the Chemistry Lab (330) computers and all breakout area computers.

Graphical Analysis can be used with manual input of data, or data can be copied and pasted to and from Microsoft Excel. You can also paste data from the Vernier data acquisition program (Logger Pro) which we will use in the laboratory.

If you wish you can also install Graphical Analysis on your own computer. We have a site license that allows any Chemistry student to install the program on a personal computer. You can check out a reserve copy of Graphical Analysis on CD from the campus Library and install it on your own computer. (Be sure to return the CD as it is a Library Reserve item!)

The following exercise will take you through many of the features of the program. Using the following data set, in which the experimenter made various concentrations of a salt, and measured the conductivity, create a table and graphs as specified.

| Concentration (M) | Conductivity (uS) |
|-------------------|-------------------|
| 0.000 | 2 |
| 0.0100 | 20 |
| 0.0150 | 33 |
| 0.0600 | 122 |
| 0.120 | 238 |
| 0.150 | 285 |

- 1) Enter the data into the program, placing Concentration on the x-axis.
- 2) Label the table column headings, and specify abbreviations and units.
(Double-click the column heading and make changes under Column Definition.)
- 3) Specify three significant figures for concentration and zero decimal places for conductivity.
(Same as above, but change figures under Options.)
- 4) Try turning the connecting line on and off; leave it off. Give the graph a title.
(Double-click on the graph area; “check/uncheck” the Connect Points box; add Title.)
- 5) Use the Regression feature to fit a line to the data, but exclude the highest point from the fit while leaving it displayed on the graph.
(First select rows from the table, or “drag” your cursor around the data you want to include in the fit. Then click the [R=] icon.)
- 6) Display the concentration value for an unknown solution with a conductivity of 170 uS.
(Click Analyze then Interpolate and position the cursor to 170 uS.)
- 7) Print the graph and table on one page displaying all the above features, and include your name on the footer.
(File / Printing Options; make changes. Reposition the cursor; click Ctrl + P.)
- 8) Close the Interpolate box and the Linear Fit box, and fit a Natural Exponent function to the data.
(Analyze / Curve Fit; scroll through the equations, select Natural Exponent; click Try Fit, then OK.)
- 9) Print ONLY the graph with the Natural Exponent fit displayed. (File / Print graph.)

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

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.

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-85, TI-84, and TI-86:

SLOPES AND INTERCEPTS WITH “GRAPHICAL ANALYSIS”

Vernier Software’s “Graphical Analysis” is available for student use. See your instructor 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. 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.

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

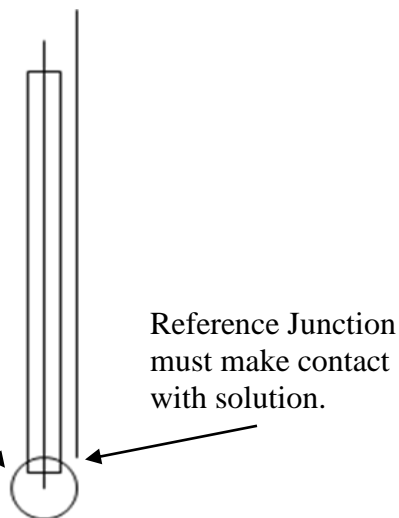
If you want to see a graph of this on your screen:

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

The Care and Use of pH Electrodes

A “combination” pH electrode has two chambers. The outer is an electrolyte that the manufacturer specifies and is usually refillable; the inner is normally HCl and is sealed. The inner and the solution to be tested are separated by a fragile glass membrane. The outer and the solution to be tested meet at the “reference junction,” a ground-glass joint or a pinhole.

Glass membrane with HCl inside and tested solution outside. The membrane is fragile. It also dissolves slightly at very high pH.



Use

Always use beaker-bumper.

Always keep membrane wet.

Rinse membrane and reference junctions when changing solutions. Dry only when necessary. Keep electrode immersed when not in use: tap water (short-term), buffer, or acidified KCl (long-term). Avoid prolonged exposure to solutions with pH > 11.

A good electrode requires about 30 - 45 seconds to stabilize.

Our meters screen-blank after a while – press POWER to restore; calibration is saved.

Suppress this when turning on by pressing MODE and POWER together.

Calibration

Measures over 10^{14} change in M concentration; calibration near working range needed.

1, 2, or 3-point procedures (we will consistently use 2-point: pH 7 and 4 or 7 and 10).

Follow procedure on instruction sheet carefully. An overview is:

Go to Calibration mode

Immerse in 7, meter will beep when stable. Press Yes. Rinse

Place in 4, watch speed, meter will beep when stable. Press Yes and observe slope value (best 95 -102; ok 92-95).

Rinse, place in solution to be tested or tap water.

Calibrate to start and re-calibrate any time electrode has been exposed to pH > 10.

Storage

Short-term: tap water

Long term: pH 4 or 7 buffer; preferably pH 3-4 KCl storage solution

Error Analysis in Large Data Sets

Error calculations for data sets with greater than 4 individual measurements (or greater than 4 pairs) should be done with a statistical “standard” error analysis rather than with average deviation. There are several styles of calculating and communicating a standard error, all of which begin with the Standard Deviation.

The standard deviation of a data set measures the spread of the data (which is indirectly related to the standard error). It can be obtained from many calculators and also from Excel. It represents the interval, above and below the mean value, which includes 68% of the data. If you extend the interval to \pm twice the value of the standard deviation, above and below the mean value, it will contain 95% of the data. The standard deviation is often inappropriately reported as an error interval. In large data sets, the uncertainty of the location of the mean value may be a much smaller interval than this.

The Standard Error can be defined as the standard deviation divided by the square root of the number of data points. This is the interval, above and below the mean value, where there is 68% probability of including the mean value from an infinitely large data set. (The symbol is usually σ , sometimes s , and sometimes there is a subscript “n-1” or “n”. Given an option, choose “n-1”.) If you extend the interval to \pm twice the value of the standard error, you increase the probability to 95%. This is the most commonly used appropriate error for large data sets.

Calculation:

- Enter your data into a calculator or Excel.
- Compute the mean.
- Compute the standard deviation.
 - The Excel function call is: =STDEV().
 - On a TI-83 Plus: Enter your data in a list using STAT / 1:EDIT / Enter. Then use STAT / CALC / 1:1-Var Stats, Enter; select your list; Enter.
- Divide by the square root of the number of data points (n).
- This is the Standard Error, i.e., $\frac{\sigma}{\sqrt{n}}$
- Multiply by 2
- Report the mean \pm twice the standard error, i.e.,

$$\text{mean} \pm \frac{2\sigma}{\sqrt{n}}$$

Example:

For {2, 2.1, 2.1, 2.3, 1.9, 2.2, 2.0} mean = 2.0857 σ = 0.1345

$$\frac{\sigma}{\sqrt{n}} = \frac{0.1345}{\sqrt{7}} = .0508 \quad \frac{2\sigma}{\sqrt{n}} = .1016$$

$$\text{mean} \pm \frac{2\sigma}{\sqrt{n}} = 2.0857 \pm 0.1016 = 2.1 \pm 0.1$$

(Note use of 1 significant figure for the error term, and the same number of decimal places in the answer.)

The Q Test for the Rejection of Data

http://orion1.paisley.ac.uk/marco/Statistics_level2/Statistics_05.htm

The best statistical test available for objectively handling data rejection when the number of observations is **small** is the Q test: This is used by first calculating an observed Q value (Q_{obs}) as shown below.

$$Q = \frac{|\text{suspect value} - \text{nearest value}|}{|\text{highest value} - \text{lowest value}|}$$

Where nearest value refers to the value numerically closest to the suspect value. For calculations of means and standard deviations, the suspect value should be rejected if the modulus of Q_{obs} exceeds the tabulated value at a particular confidence level. For example, the Q_{tab} values for a small number of measurement

($3 < n < 10$) at the 90% level is given below.

| n | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|------------|----------|----------|----------|----------|----------|----------|----------|-----------|
| $Q_{90\%}$ | 0.94 | 0.76 | 0.64 | 0.56 | 0.51 | 0.47 | 0.44 | 0.41 |