CHEMISTRY 444/544

PHYSICAL CHEMISTRY LAB: WINTER, 2010.

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Email: rsimoyi@pdx.edu

Venue: **Room 439 SB2**

Text Book: **(a) P.W. Atkins and J. de Paula, *Atkins' Physical Chemistry*, 7th edition, Oxford University Press, Oxford, 2002.**
(b) Garland, Nibler and Shoemaker, *Experiments in Physical Chemistry*, Seventh Edition, McGraw-Hill, 2003.
(c) 'Physical Chemistry; A Molecular Approach' by Donald A. McQuarrie and John D. Simon. (University Science Books, Sausalito, CA). ISBN No. 0-935702-99-7.

LABORATORY RULES AND REGULATIONS

1 Students must be present ten minutes before the start of each scheduled laboratory session. Latecomers will be refused entry to the laboratory.

2 No student will be permitted to work in the laboratory outside of laboratory hours except by express permission of the staff member(s) responsible for the session. Never work in a laboratory on your own.

3 Smoking is strictly prohibited in all laboratories and instrument rooms.

4 Do not put anything into your mouth while working in the laboratory. **NEVER** taste a chemical or solution. Eating is totally **PROHIBITED** in all laboratories.

5 All students are required to wear a laboratory coat and no student will be permitted to work in the laboratory without one.

6 All students who do not wear conventional spectacles must wear eye protection. Safety glasses must be worn throughout all practical sessions. Students who wear conventional spectacles must have them on at all times when in the laboratory.

7 All students must wear closed shoes in the laboratory, unless permission has been obtained to wear sandals for some medical reason.

8 Apparatus and chemicals are **NOT** to be removed from the laboratory.

9 Students will find the laboratory bench clean on arrival in the laboratory. The bench at which you work must be left clean when you leave the laboratory at the end of the

practical session. Bench tops must be wiped clean. Glassware and other apparatus should be left clean and dry, unless otherwise indicated or instructed.

10 Work areas must at all times be kept clean, and free from chemicals and apparatus which are not required. All glassware and equipment must be returned to its proper place, clean and dry, and in working condition, unless otherwise indicated or instructed.

11 All solids must be discarded into the bins provided in the laboratory. Never throw matches, paper, or any insoluble chemicals into the sinks. Solutions and liquids that are emptied into the sinks must be washed down with water to avoid corrosion of the plumbing. Waste solvents must be placed into the special waste solvent bottles provided.

12 Before leaving the laboratory at the end of a practical session make sure that all electrical equipment is switched off, and that all gas and water taps are shut off.

13 Students who break or lose equipment allocated to them will be required to pay for replacements. All breakages or losses must be reported to the teaching assistant in charge.

14 Do **NOT** heat graduated cylinders or bottles because they will break easily.

15 Any apparatus or glassware which has to be heated must be heated gently at first, increasing the amount of heat gradually thereafter.

16 Balances, spectrophotometers and other expensive equipment must be treated with care and kept clean and tidy at all times.

17 Fumehoods must be used when handling toxic and fuming chemicals. Other operations, such as ignitions, are also carried out in fumehoods. The only parts of the human body which should ever be in a fumehood are the hands - never put your head inside a fumehood.

18 Never leave a laboratory experiment unattended without first consulting the TA in charge.

19 Reagent bottles must be re-stoppered immediately after use. It is **absolutely forbidden** to introduce anything into reagent bottles, not even droppers. Solutions and reagents taken from bottles must **NEVER** be returned to the bottles. Do not place the stopper of a reagent bottle onto an unprotected bench top.

20 Laboratory reagents and chemicals must be returned to their correct places immediately after use. Spillage must be cleaned off bottles/containers. Labels must face the front.

21 The use of reagent bottle caps as weighing receptacles is forbidden.

22 Liquids - whether corrosive or not - must be handled with care and spilling on the bench or floor should be avoided. Any spillage must be cleaned up at once - if the liquid is corrosive (acids or bases) call your TA or professor. **Never** hold a container above eye level when pouring a liquid.

23 When carrying out a reaction or boiling a liquid in a test tube, point the mouth of the test tube away from yourself and others in the laboratory.

24 Beware of hot glass and metal. Never handle any item which has been in a flame, a hot oven or a furnace without taking precautions. Use leather/asbestos gloves or tongs, or ask for advice on what to use.

25 Report all accidents, cuts burns, etc., **however minor**, to your TA or the professor. Eye-wash stations are located in various places in the laboratory. Ensure that you know where the nearest one to your bench is located.

26 **A chemical laboratory is not a place for horse-play. Do not attempt any unauthorised experiments. Do not play practical jokes on your classmates. Such things are dangerous and can cause serious injury. Any student found indulging in such activities will be banned from the laboratory, with consequent grade of F for the lab course.**

OCCUPATIONAL HEALTH AND SAFETY

YOU are warned that all substances handled and all operations performed in a laboratory can be hazardous or potentially hazardous. All substances must be handled with care and disposed of according to laid down procedures. All operations and manipulations must be carried out in an organised and attentive manner.

In order to assist you in developing good and safe laboratory techniques, a set of Laboratory Rules and Regulations is attached. You are required to read these and to acknowledge that you have read and understood them. Additionally, in the laboratory manuals/practical books and/or pre-practical lectures your attention will be drawn to the correct and safe handling of specific chemicals/reagents/solvents, and to the correct/safe manner in which specified laboratory operations must be carried out. These specific instructions and/or warnings must never be ignored.

GENERAL FIRE ORDERS

Fire fighting instructions are exhibited in individual laboratories. However, the following orders must always be obeyed.

In the event of a fire

Attack it at once with the appropriate fire fighting equipment and **shout** for help.

On hearing a fire evacuation alarm

1 Stop normal work immediately.

2 Make safe any apparatus, and material in use, shutting off as necessary any local gas taps/valves, electricity and other potentially dangerous services under your control.

3 Immediately leave the building.

4 Go to the Fire Evacuation Area which for this CHEMISTRY BUILDING is outside to the south west entrance to the building, on the grassed area between the Hall of residence and Science Building 2 (which is the building you are presently).

DEPARTMENT OF CHEMISTRY

PORTLAND STATE UNIVERSITY

ACKNOWLEDGEMENT FORM

I, the undersigned (please print full name)

.....

Student No. .....

Identity No. .....

do hereby acknowledge having read and understood the documents headed "Occupational Health and Safety" and "Laboratory Rules and Regulations". Furthermore, I accept that contravention(s) of these rules and regulations will lead to my expulsion from the laboratory.

I agree to abide by any additional laboratory regulations or safety rules presented in writing in the practical manuals/books or issued verbally by the INSTRUCTOR-in-charge, or his/her responsible member of staff.

SIGNED ..... DATE .....

ARRANGEMENTS FOR LABORATORY SESSIONS

Dates and Times for Practical Sessions
Practical sessions will be held only on the specific timetabled day during the winter quarter.

Laboratory sessions will commence promptly. Students are expected to report punctually for each laboratory session. Those arriving late for a practical session may not be admitted to the laboratory. This will result in a mark of zero being recorded for that experiment.

Attendance
A register will be taken each day, and a grade of F may be awarded to students whose attendance records are regarded as unsatisfactory. Each experiment will be marked out of 100; a mark of 0 will be entered in the case of an uncondoned absence. Absences will only be condoned for medical reasons; in such cases a medical certificate must be provided at the next lab session.

Dress
Students must wear white laboratory coats and safety glasses at all times while in the laboratory. Open shoes and flip-flops are not considered acceptable dress. The wearing of any headgear in the laboratory is also unacceptable. Sunglasses are **not** to be worn as a substitute for safety glasses.

Accidents
Any accidents which occur during the laboratory session must be reported immediately to the professor in charge, who is required by law to write an accident report.

Breakages
Any breakages of equipment or glassware must be reported immediately to the TA in charge. The costs of replacement will be debited to the student's fee account.

Waste Disposal
Certain experiments generate hazardous waste which must be disposed of according to the instructions provided in the laboratory manual or given by the lecturer in charge.

Laboratory Notebooks
Students are required to have a hardcover notebook in which to write their laboratory reports. Some may prefer to have two lab notebooks so that they can still have one with them after submitting one for grading. These lab notebooks are available in the bookstore. All relevant data, measurements and observations, should be written directly

into the lab notebook, and not on some scrap pieces of paper and they transcribed into the lab notebook after the lab session. It is not essential that the lab notebook be clean and neat. Nothing wrong with well-arranged data presentation in a student's notebook, but this can be more of a product of preparation prior to the session.

Pre-practical Preparation and Report Writing

The key to doing these practicals correctly and expeditiously is your preliminary preparation before coming to the lab session!!

Before coming to the laboratory you should have stated the **aim** of the experiment you are about to perform that afternoon in your laboratory notebook. In addition, you should have prepared the tables (with the columns appropriately headed) into which you will enter the data you collect during the practical.

The crude **data** collected during the laboratory session must be entered directly into the notebook, in ink, in tabular form where appropriate. Each table should contain the appropriate columns of data, each column consisting of a particular physical property. As a physical property is the product of a number and a unit, the heading of each column should consist of the property divided by its unit, so that the entries in the column below are pure
numbers, as shown in the example below.

Concentration/mol dm^{-3}	Osmotic Pressure/atm
0.098	2.59
0.192	5.06
0.282	7.61
0.370	10.14
0.453	12.75
0.533	15.39

Likewise, graphs should be included in the report where appropriate. In plotting graphs, students should use graph paper (provided at the front desk) and utilise the full area of the sheet of graph paper, adjusting the scales on the axes to maximize the space available. Measurements of the gradients of graphs should be performed by using the largest possible spread of values along the horizontal and vertical axes, in order to minimize

errors in determining the slopes. The axes should be labelled clearly with the properties being plotted, along with their units, in the same way as for the column headings in tables, as shown in the attached example (page ix). The same applies to graphs prepared with a computer package such as Microsoft Excel®.

For the example of a typical graph shown in Figure 1;

$$\text{slope} = \frac{(15.4 - 2.6)\ \text{atm}}{(0.54 - 0.105)\ \text{mol dm}^{-3}}$$

$$= 29.4\ \text{atm dm}^3\ \text{mol}^{-1}$$

Calculations must be shown in full; for repetitive calculations of the same type, it is only necessary to include one full set of workings.

Each report should end with a **discussion**, in which students are expected to remark on the significance and meaning of their results, and explain any unexpected observations which may have been made. For several experiments the laboratory manual sets a number of **questions** which should be answered as part of the discussion in the report. This is the most creative part of the report, and presents the opportunity for students to score marks for demonstrating their understanding of the experiments performed and the relevance of their results. A copy of the Handbook of Chemistry and Physics is provided in the laboratory, and students are expected to consult this reference work in order to compare their results with those in the literature, and to cite a **reference** for any value quoted. Relevant comments on possible causes for significant deviations of their results from those in the literature should also be included in this section of the report.

Note: Read the first 89 pages Garland, Nibler and Shoemaker, *Experiments in Physical Chemistry*, Seventh Edition.

READING REFERENCES

The references given in the detailed instructions in this laboratory manual refer to the following two texts:

P.W. Atkins and J. de Paula, *Atkins' Physical Chemistry*, 7th edition, Oxford University Press, Oxford, 2002.

Garland, Nibler and Shoemaker, *Experiments in Physical Chemistry*, Seventh Edition, McGraw-Hill, 2003.

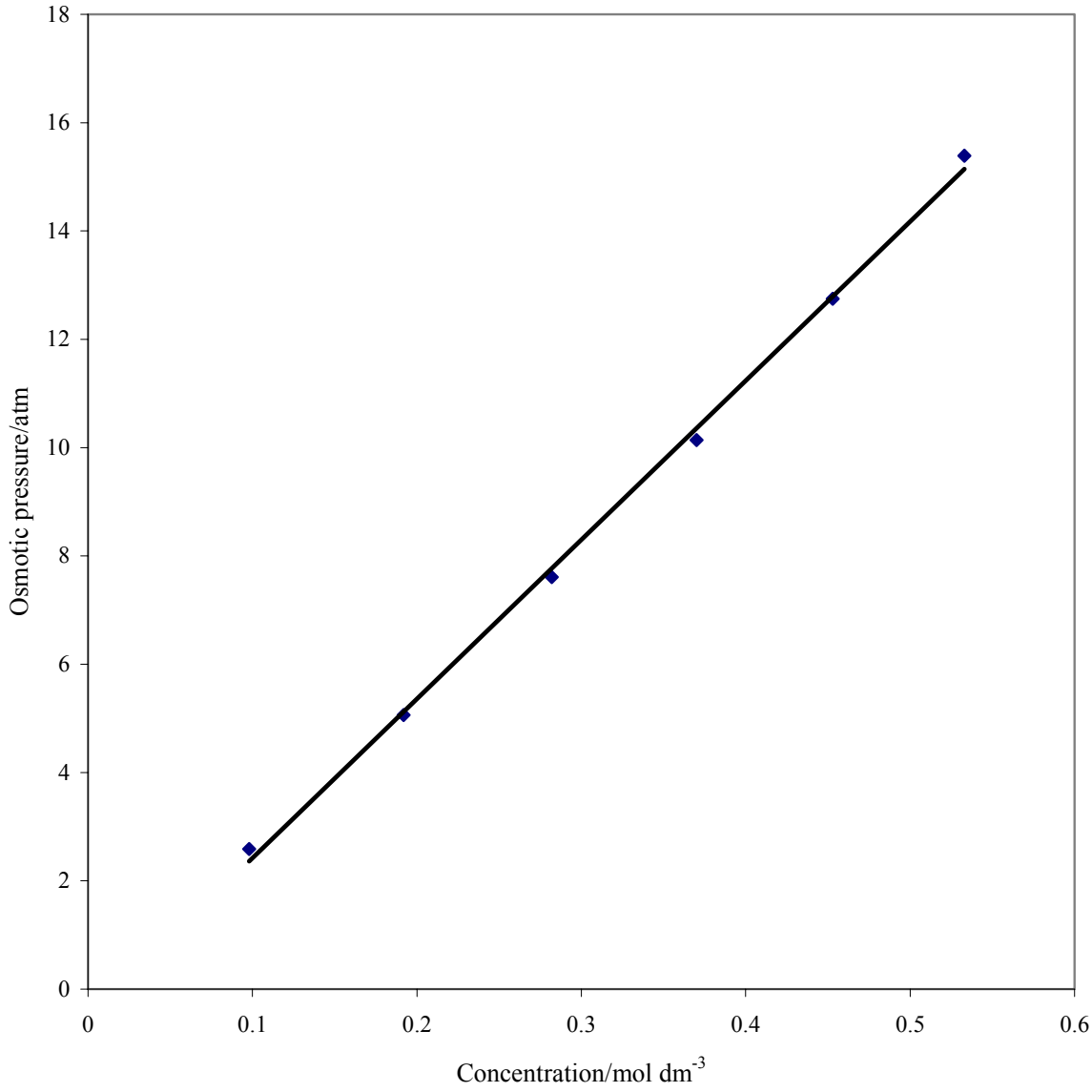

Figure 1: Osmotic pressure of aqueous solutions of sucrose plotted as a function of concentration at 20 °C.

PLAGIARISM

Plagiarism is defined as the submission or presentation of work, in any form, which is not one's own without acknowledgement of the source(s). It is an attempt to deceive the reader that the work or ideas presented are your own, whereas, in fact they are the words/ideas of others.

With regard to essays, reports and dissertations, a simple rule should be used when deciding if it is necessary to acknowledge sources. If you obtain information from an outside source, that source must be acknowledged. Another rule to follow is that any direct (verbatim) quotation must be placed in quotation marks and your wording should clearly indicate that the item is not your own work and the source immediately cited. The mere inclusion of the source in a bibliography shall not be considered sufficient acknowledgement.

This applies to all work contributing to assessment, including laboratory reports and projects. All assessed work must be your own individual effort. Copying of laboratory reports, for example, is plagiarism. You may share data, where appropriate, but the calculations, answers to assignment questions and the discussion of results must be your own work.

Work referred to from Internet sources must also be acknowledged as above, with the web address (URL) of the source included and the date it was accessed.

Lab Reports:

Lab reports must be written **INDIVIDUALLY**, must be of journal quality, and must follow the JACS format. Online you can also find guidelines on http://www.rose-hulman.edu/~tilstra/ .

Important Dates: To be advised

Before you start an experiment, ask the following questions:

1. Do I understand the principle of the experiment?
   - If not, please go back and read about the experiment. The following resources are available: Experiments in Physical Chemistry by Shoemaker, Garland, Nibler; the web; any physical chem. text.
2. Do I know the experimental setup?
   - If not, go to the lab and find out, ask instructor, web/library search, read the instrument manual.
3. Do I have my lab notebook ready?
   - The simplest way to keep all the important data is to have a notebook. You are allowed to use the PC's in the physical chemistry lab to do all the searches and keep good documentation.
   - Writing all the relevant information about how you are going to do the experiments will help you reduce the time it takes to finish the lab report.

In the Lab: A Checklist

- Do I have all the equipment necessary to setup the experiment?
- How do I hook it up and start the experiment?
- Safety first!!
  - Use common sense.
  - With radiation or high voltage make doubly sure that you are following the appropriate procedure.
  - Do not play with open electrical connections or liquid nitrogen.
- Prior to collection of data
  - Check if calibration run needs to be performed.
  - Check how the data will be saved.
  - Note the exact instrument model/serial numbers in your notebook.
  - Take a few minutes to look at the instrumentation manual.
  - Spend a few minutes to assess sample preparation/purity.
  - Determine the precision of the instrument.
- Data collection
  - Decide which variables are fixed and the uncertainties involved in their measurement.

    - See how minor perturbations of the experimental conditions affect your data.
    - Repeat…Repeat…REPEAT…to improve the accuracy/precision of the data.
    - Record everything in your notebook.
- After the experiment
    - Shut down the power and clean up the surroundings.
    - Write down brief notes in the logbook.
    - Notify instructor if there is any problem.

Analysis of Data

- Identify the relationship between the independent/dependent variables.
- Determine both precision/accuracy of measurements
- Rejection of data: Students test.
- Fitting the data
    - Selection of equation.
    - Perform linear and/or nonlinear least squares analysis using proper weighting function.
- Compare your data to what is expected in the literature.
- Summarize your conclusions.

Suggest how you might be able to improve the experiment

Laboratory Report Guidelines

Technical reports have several features that are consistent between various fields of study. Below is a list of sections typically found in a technical report. They may exist with slightly different names in different fields. The order in which these are presented may also vary but for the purposes of these guidelines we will use this order, which is the same as the one used in the Journal of the American Chemical Society (JACS).

Abstract

Introduction

  Equations

Procedure

Results

  Tables

  Figures and Plots

Discussion

The following pages include a more detailed description of each of the sections. Be aware that this is a living document. If some portion of it is inconsistent with your experience, please relay that information.

1.) ABSTRACT

The abstract may be the hardest section of a paper to write. Although it appears at the beginning of an article and is usually the first thing the reader looks at, the abstract should be written last, after the article is complete.

In professional journals, the abstract is often used to identify key features for indexing and so it should contain words that other professionals would use in a literature search. The abstract may appear by itself in a separate publication, and so it must be self-contained. On the other hand, because other professionals read the abstract to get a quick feel if the rest of the article will be of interest to them, it must be concise.

The abstract should contain a brief statement of the problem or purpose of the research. It should indicate the experimental or theoretical plan, summarize the principal findings, and state the major conclusions. It should not add to, evaluate, or comment on conclusions in the text.

The abstract should not cite tables, figures, or sections of the paper. Abbreviations and acronyms should be used sparingly, and should be defined at their first use.

2.) INTRODUCTION

The opening sentence of a paper should state the problem or the purpose of the experiment. Subsequent sentences should provide a concise background and identify the scope and limit of the work.

In professional journals the introduction should also contain the background and/or history of the research project that would be presented. This background should include the citation of pertinent literature, and identify how this work is different or related to the cited literature.

The concepts and their related equations must be developed from an accepted starting point. This means that terms must be defined early in the section, and that the concepts are presented in a logical order such that--when appropriate--they build on each other.

Discussion between equations should connect the equations conceptually. Completeness and clear thought are required in this section. The reader should be convinced that the author(s) know and understand the principles of the experiment.

► EQUATIONS

Equations should be offset from the text in some manner, either by indentation or by centering; and numbered. Equations should be numbered sequentially in order of initial appearance; this makes it easy to refer to them at some later point in the text. The terms of equations should be defined the first time those terms appear. It is not necessary to redefine a term every time it appears in an equation. It is not appropriate to use the same notation for different terms in different parts of the text, nor is it correct to use different notation for the same term in different parts of the text.

3.) **EXPERIMENTAL PROCEDURE**

This section of a report should have sufficient detail about the materials and methods that the audience could repeat the work and obtain comparable results. Identify the materials used, giving information about the purity. Give the chemical names of all compounds and the chemical formulas of compounds that are new or uncommon. Describe your apparatus if it is not standard or commercially available. Describe the procedures you used. Always use third person past tense, e.g. "Acid was added to the solid" and NOT "I added acid to the solid".

4.) **RESULTS**

Summarize the data collected. Include only relevant data, but give sufficient detail to justify your conclusions. Use equations, table, and figures for clarity and conciseness. It is often convenient to connect various pieces of information with some discussion. In this case, this section would be called RESULTS AND DISCUSSION

►TABLES

One of the most efficient methods used to communicate technical information is by means of a *data table*. While you have all seen examples of well-organized, legible data tables, few of you have had a great deal of practice constructing one from scratch. The construction of a good data table requires knowing what the important features are.

I. **When to use Tables**

Tables are to be used when the data are precise numbers, when there are too many to be presented clearly in the narrative, or when relationships between data can be more clearly

conveyed in a table than in the narrative. Tables should supplement, not duplicate, text and figures. If data is not treated theoretically in the report or if the material is not a major topic of discussion, do not present it in tables.

II. **How to Construct Tables**

A table should consist of at least three columns, and the center and right columns must refer back to the left column. If there are only two columns, the material should be written as narrative. If there are three columns, but they do not relate to each other, perhaps the material is really a list of items and not a table at all.

Tables should be simple and concise, but many small tables may be more cumbersome and less informative than one large one. Combining is usually possible when the same column is repeated in separate tables. Use symbols and abbreviations that are consistent among tables and between tables and text.

Numbering Tables: Number tables sequentially with Roman numerals, in order of discussion in the text. Every table must be cited in the text.

Title: Every table must have a brief title that describes its contents. The title should be complete enough to be understood without referring to the text, and it should not contain new information that is not in the text. Put details in footnotes, not in the title.

Column Headings: Every column must have a heading that describes the material below it. Keep headings to two lines, use abbreviations and symbols. Name the parameter being measured and indicate the unit of measure after a comma. A unit of measure is not an acceptable column heading.

Columns. The leftmost column is called the stub column. All other columns refer back to it. Main stub entries may also have subentries that should be indented. Be sure that all columns are really necessary. If there are no data in most of the entries of a column, it probably should be deleted. If the entries are all the same, the column should be replaced with a footnote that says "in all cases, the value was . . . " Do not use ditto marks or the word ditto. Define nonstandard abbreviations in footnotes.

► FIGURES AND PLOTS

Often the most concise and precise way to present data is to plot it. The challenge is knowing what to plot on which axis. An understanding of the theory behind the experiment should provide clues so that the author can determine how to design a plot. Some general rules apply regardless of the content of the report.

- The figure, graph or plot should have an appropriate title. Restating the axis labels is NOT an appropriate title.
- All axes should be labeled with the parameter being plotted and the units of that parameter. The number of values on the axis and the number of tic marks should

be sufficient to make it easy for the reader to identify the x and y values of each data point.

- The x-axis is always the independent axis and the y-axis is the dependent axis; that is, the value of the y variable depends on or changes *because of* a change in the x variable. For example, the concentration of reactants changes *because* time has passed. It is not true that time has passed because of a change in the concentration of reactants. Consequently, time will be on the x-axis and concentration of reactants will be on the y-axis. By convention, a plot of A versus B means that A is on the y axis and B is on the x-axis.
- The plot should not have a legend if there is only one set of data being plotted.
- Individual data points should be obvious.
- DO NOT CONNECT THE DOTS!!! It is not appropriate to connect the dots with straight lines (unless it's a linear regression) because this implies that, between the points for which you took a measurement, the function follows the straight line that you've drawn. This is usually not true. It is appropriate and good to draw a curve on your plot that is the best fit of your data *to the functional form that theory predicts*. It is appropriate to try to determine the functional form of the data you've presented, however if the theory provides a functional form, use it.
- There should be a minimum of emptiness on a plot. If your data covers only a small portion of the plot, expand the axis so that the data fills the plot. There is one exception to this. If part of the purpose of the plot is to identify the value of the y-intercept, the y-intercept should be on the plot.
- Plots should be as large as is feasible because if you make a plot too small, all data points will fall on a line. Everything appears linear; even really bad data can be made to look artificially good.

5.) **DISCUSSION**

After clearly presenting results of your experiments, either as tables or figures, it is necessary to discuss the meaning of those results. The discussion section of a report should be objective. The results of the experiment should be interpreted and (where appropriate) compared with each other. They should be related to the original purpose of the project. In the discussion section, it should be clearly stated whether or not the problem has been resolved. The logical implications of the results should be stated and further study or applications may be suggested. Conclusions should be based on the evidence presented.

6.) **CONCLUSION**

The conclusion should begin with a restatement of the results. The results should be compared with literature values whenever possible. Any error in the results should be addressed, for example why a plot that--according to theory--should be linear isn't linear. Any possible sources of error should be identified.

7.) **REFERENCES**

Reference citations come in different styles depending on where the article is being submitted to. For these guidelines we are following the JACS format so familiarize yourself with this and try to use it for your reports. Other formats are also acceptable but it is extremely important that whatever style is used should be used consistently.