

INTRODUCTION

This manual is intended as a supplement to the textbook, Shoemaker and Garland, "Experiments in Physical Chemistry". The manual contains extra material and helpful hints for several of the experiments, together with an outline of course requirements and other general information. The student must have access to a copy of the text, which gives a complete description of most of the experiments and provides additional hints and suggestions for topics of discussion.

COURSE REQUIREMENTS

Lecture/Tutorial

The weekly one hour lecture/tutorial has three purposes:

- 1) Exercise in low-stake writing; students will be asked to write in ~five minutes some short notes (1 page maximum) on a subject related to the Lab; the subject will be announced at the beginning of the lecture. These notes will be collected; they will not be marked individually but will indirectly make some contribution to the final grade as part of the instructor's assessment of the student's performance. The content of this writing may be discussed with the student during the next lab session.
- 2) The laboratory grades will be returned during these sessions and the reason for the grading obtained will be discussed.
- 3) As time permits, lectures/seminars will be offered on technical subjects relevant to the Physical chemistry laboratory, chosen from the chapters supplemental to the experiment descriptions of the text book, or on special subjects requested by students.

Experimental Work

Each student must complete eight experiments from the list next page.* At least one experiment must be chosen from each group.

Each student must maintain a laboratory notebook in which all experimental observations and results obtained during the laboratory session are recorded. At the end of each laboratory session, this notebook must be seen and signed by the instructor (see next section under *LABORATORY NOTEBOOK*).

* New experiments which are in development may be offered during the term. In this case, supplemental written material will be provided in due course.

List Of Experiments

Group 1	I	Langmuir–Blodgett Trough Experiments
	II	Surface Tension
Group 2	III	Scanning Tunnelling Microscope (STM) (Special hand-out)
	IV	C _p /C _v Ratio
Group 3	V	Charge Transfer Complexes
	VI	Kinetics of a Fast Reaction
Group 4	VII	Dipole Moment of Polar Molecules in Solution
	VIII	Dissociation of Weak Acids
Group 5	IX	Differential Scanning Calorimetry
	X	Polymer Viscosity
Group 6	XI	Infrared Spectrum of SO ₂
	XII	Low Temperature Heat Capacity

Laboratory Reports

A formal report (see below) must be written for each experiment.

Report submission dead-lines

For the first three reports, the deadline for submission is the next lab day following the week of performance of the experiment. The report handed in will be graded and returned at the following laboratory or lecture/tutorial session. Then, for the final next five report the deadline is relaxed to two weeks from the date of performance of the experiment (however students are strongly encouraged to hand in their reports weekly; also be aware that reports corresponding to experiments performed during the last week of classes are due *one* week later).

Late reports

There will be penalties for late reports and a bonus for overall punctuality (See marking scheme below). In addition, *reports late by more than two (2) weeks may be assigned a failing grade*. This system is an attempt 1) to allow for some feedback to the student on her/his performance, and 2) to prevent too much postponing in writing reports to the end of the term, at which time other things are happening (like “finals”).

Course Grading

The final grade for the course will be based on:

- a) grades obtained for each report - broken into approximately 60% for quality of report writing (clarity of presentation and relevance of discussion), 30% quality of experimental content,
- b) laboratory notebook, technical ability, degree of preparedness as judged by the instructor from submitted writing material and discussion during the laboratory session, and punctuality in handing in reports within the deadline (10%).

LABORATORY NOTEBOOK

This must be a hard- or wire-bound notebook — loose-leaf is not acceptable. The notebook is used to record what you do in the laboratory and the results of your experiments. Here are some important features expected for a lab notebook.

Owner's name

The name of the owner must appear on the cover.

Table of Content

Reserve a few of pages at the beginning for information which may be added at later time, like a Table of Content.

Entries dated

For each experiment, the title of the experiment and the date of performance should be written (name of partner if applicable).

Complete record

As a rule, enter data and information on only one side of the open book; the other side is left blank or may be used later for extra notes or for other scribbles. Results are to be entered in the book as they occur. The original data must be entered without transcription or processing; for example, if you measure a pressure by reading the left and right sides of a U-tube manometer, both readings should be recorded, not just the resulting pressure found by subtracting them. The object is to always have a complete record of the original data as collected. Make a note of any mishaps during the experiment or changes in the procedure.

Units

When entering data, make sure to record the units.

Do not delete mistakes

Nothing should ever be erased (no white-out either) in the notebook. A single line should be drawn through mistakes and the correct information re-entered. If you are

really unhappy about a page, just cross out the whole page and re-enter the data in a new page; do not tear off the offending page.

Identify digital information

If data are collected in digital form (*ie*, by a computer), make a note of the identity of the computer and the name of the relevant data storage medium and/or files.

Notebook assessment

It is not expected that a laboratory notebook will be a thing of beauty or that it will have any artistic merit. It must, however, be sufficiently well organized that everything can be found at a later date. This notebook is to be shown to the instructor at the end of each laboratory session and at the end of the term for assessment; it will be checked for completeness and accuracy of the information contained. As mentioned above, the instructor's judgement of the laboratory notebook will make some contribution to the final grade.

STORAGE AND SUBMISSION OF DATA IN DIGITAL FORM

For several experiments the data are collected and stored via a computer. For other experiments, most of the calculations can be (and should be) performed with the help of a spreadsheet program. In addition, for most experiments you are expected to submit your raw data (data as collected during the experiment, before any processing) in a specific digital format stored on a diskette, memory key or other convenient forms, or as an email attachment sent to the instructor; details are provided for each experiment in the lab manual.

GUIDELINES FOR REPORTS

A formal report must be submitted for each experiment performed. These should generally follow the suggestions in Shoemaker and Garland (S&G) keeping in mind that the report is meant to be informative and easy to follow by the reader.

The report is to be written in proper English, in sentence form using the third person (should not contain first person pronoun, *I, me, we*).

The following sections must appear in the report: Title Page, Abstract, Theory, Experimental Procedure, Results, Calculations, Discussion, Conclusion and References. Other sections may be added if it seems appropriate.

At the same time you hand in your report, hand in the completed "Report check-list" (found in this manual, at the end of each experiment description). A report will not be accepted without it.

There is no mandatory format for the report; however a suggested format is shown in one of the appendices and the corresponding Microsoft word template can be downloaded from the course web site.

Title page

Include the title of the experiment, then name of course, semester, your name, student no. (name of partner if applicable), date of performance of experiment and date of submission of report.

Abstract

The abstract should summarize the principal findings, give the *actual final numerical results*, the relevant conditions under which these results were obtained (eg, temperature, pressure, etc...) and the major conclusions. It should be concise (200 words max) with a few sentences. It should be self-contained, *ie*, no references to tables, figures or sections of the report.

Introduction and theory

This section contains a brief statement about the purpose of the experiment. Then the theoretical basis and the principle of the experiment should be explained fully but *concisely* (no more than one page); long passages copied directly from a text book or manual, will be frowned upon. Equations or formula to be used to analyse and interpret the data need not be derived but literature reference to the derivation must be indicated.

Experimental

Simple reference to the manual and/or text book in which the detailed procedure are given is usually sufficient; it is not necessary to recopy the whole description of the procedure. However,

- Present the various important experimental parameters which would be necessary to repeat the experiment (*eg*, temperature, pressure, solution concentrations, etc...),
- *do* indicate clearly changes from the written procedures and the reasons for such changes such that the experiment could be repeated under identical conditions.
- Indicate also unusual happening or failure of equipment which occurred during your session.
- A diagram of the apparatus may be included — again indicate deviations from manual description. In particular, if you feel that a diagram is warranted, make sure that the drawing you include in your report is a good representation of the actual apparatus encountered in the lab. The external aspect of the set-up used may be quite different from the picture found in the text book, although both do the same thing.

Results

You should present the actual results of your experiment.

- These should be neatly organized into tables or graphs, as appropriate.
- All results should be presented to the precision justified by experimental uncertainties and an estimate of these uncertainties should be given.

- The raw data (the actual data collected during the lab session, whether in tabular form or plots from a chart recorder) *must be included or attached*; they may appear as an appendix at the end of the report, may be found on some digital media or send to the instructor by email in the format indicated in the manual (see additional note below). For data collected by a computer, DO NOT include a printed output of the numerical results; hand in a diskette or other convenient digital media containing the data (memory key, email attachment) or indicate on which computer the data can be found and the corresponding file name. Accessibility to the original data is the only way for the grader to trace back the reason for mistakes or strange results.

Note: a report may not be graded if the raw data are not available to the instructor.

- If it is felt that some data should be discarded, indicate clearly this, the reason why and the justification; one must have very good reasons (other than aesthetics or convenience) to reject experimental data.
- Eventually the final results should be summarized in a table, including the uncertainties (see below) and the literature or accepted values.

In addition, for some experiments, raw data (ie, unprocessed data as collected during the lab session) are to be submitted in a specific format. In this situation a MS-Excel template is available from the course web site; this is mentioned in the lab manual. The corresponding file is to be submitted either on a diskette or other convenient digital media, or as an email attachment to the instructor and teaching assistants.

Calculations (may be included as an Appendix to the body of the report)

You should document how quantities derived from your original experimental data are obtained.

If you submit typewritten reports, the "Calculation" section can be very tedious to type; it is totally acceptable to submit this section hand-written.

- Give the literal formula to be used, followed by the value of each member of the formula, then the results (with units); DO NOT retype (or rewrite) the formula with numbers substituted in. For example, suppose one needs to calculate the mole fraction x which is given by

$$x = \frac{n_s}{n_s + N_{\text{Solvent}}} \text{ where } n_s \text{ and } N_{\text{Solvent}} \text{ are number of moles of solute and solvent}$$

respectively.

Then, for example, using some actual numbers, after showing the literal formula, write, $n_s = 0.011 \pm 0.001$ moles,

$$N_{\text{Solvent}} = 9.91 \pm 0.01 \text{ moles, thus } x = (1.04 \pm 0.09) \times 10^{-3},$$

But please *do not write*

$$x = \frac{0.011 \pm 0.001 \text{ moles}}{0.011 \pm 0.001 \text{ moles} + 9.91 \pm 0.01 \text{ moles}} = (1.04 \pm 0.09) \times 10^{-3}$$

- If a repetitive calculation is done many times on similar data, it is sufficient to give the calculation *once* in detail, and just report the results of the subsequent calculations.
- If a computer program or a spreadsheet were used for calculations indicate the origin; a typical output should be included in your report, but make sure that the printed output is properly presented (careful with significant digits and column heading alignment).

An essential part of Calculations is a calculation of the errors (or uncertainties) expected in the final result. This must be done from your estimates of the error in original experimental quantities and a calculation of how these errors combine to produce errors in the final calculated result. If you do not know how to do error propagation calculations, read the corresponding section in S&G, or the ERROR ANALYSIS section in the Appendix of this manual, or other specialized reference. The reader must be told how accurate the final result is expected to be, based on the actual experimental data collected and the technique which has been used.

Discussion

This section should give any necessary concluding discussion, including comparison of results with literature or theoretical values, if available. Answer or comment on points of discussion raised in the text book and in the lab manual.

Conclusion

This should be a short statement summing up the experiment and the results, usually in the context of the objective set out at the start of the experiment.

References

Should give a numbered list of all sources quoted, in order of appearance in the report. The corresponding number should be inserted as a superscript¹ (or in between square brackets[2]) in the body of the report, at the point where the quotation is made. Common reference formats are:

- 1 Author1name, Initials.; Author2name, Initials. *Journal*. **Year**, *Volume*, firstpage - lastpage.
- 2 Author3name, Initials.; Author4name, Initials. *Monograph or Book Title*; Edition, Publisher: City, Year.

Other format may be used, but be consistent.

GRADING GUIDELINES

The reports will be graded according to the following guidelines:

- *A*, very good, *ie*, clear writing and correct presentation, sensible data, correct calculations and results, correct interpretation of the data at hand, discussion complete and correct.

- *B*, good but some points amongst those listed above are incorrect or missing.
- *C*, acceptable but with some serious flaws in presentation, mistakes in the calculations, significant misconceptions or confusing writing.
- *D*, very serious flaws and not acceptable; if such a grade is assigned, the student may be given the chance to correct the mistakes and resubmit the report. In this situation, the new grade, after correction cannot be higher than *C*; note that if the corrections are judged insufficient, the original *D* grade may stand. Note also that *reports submitted the last week of classes will not have a chance to be resubmitted* for potential upgrade if a *D* grade is assigned.
- *F*, substandard report not acceptable, *report* handed in too late or not handed in.
- Breach of academic honesty will result in an automatic *F* grade for the particular report, but may have more serious consequences (see below, section “ACADEMIC HONESTY AND STUDENT CONDUCT POLICIES”).

IMPORTANT NOTES

Certain experiments require advance preparation from the instructor. Therefore, be sure to book an experiment at least a week in advance if you want this experiment to have a chance to perform smoothly.

Remarks on the Conduct of Experiments

For most of these experiments, the preparatory work and/or clean up is the lengthy part of the experiment. Once the samples have been prepared, and the experimental set up assembled, the actual data taking is usually simple and routine.

Therefore, make sure that you understand

- the principle of the experiment,
- the principle of the measurements,
- the techniques used,

and that

- you have checked in advance the present lab manual for suggested specific prelab preparations,
- your samples are carefully prepared,
- you leave the laboratory with all the necessary information,
- you clean up and put away the glassware used and switch off all electronics,
- you sign up for another experiment.

Some experiments may require more than one laboratory session. It is your responsibility to organize your work accordingly.

Remarks on Data Analysis

Data analysis can be greatly facilitated by the use of a spread sheet program in combination with some least-squares fitting program (see appendix in this manual)*. In particular, make sure you have a software which allows you to perform weighted least squares fit; if not, do get a copy of the two EXCEL templates provided for this purpose available on some of the Pchem Lab computers or on the course web site* (WLLSQFIT.XLT and WPOLYFIT.XLS).

Safety in the Lab

- Smoking, eating or drinking are not permitted in the laboratory.
- Proper eye protection is mandatory while in the laboratory.
- Lab coats are highly recommended.
- Be sure you know where the fire extinguisher, the shower and the eye-wash fountain are located.
- Safety data sheets on the chemical compounds used during the lab are available in the room. You are encouraged to read them before handling the chemical. When in doubt, ask the instructing staff.
- In the present manual, safety instructions or hints specific to each experiment are included; please read these suggestions before coming to the lab and apply them during the lab.
- Except for common alcohols (MeOH or EtOH) and acetone, *organic solvents must be discarded into a special waste solvent container; never directly to the sink.*
- *Never* perform experimental work alone in the laboratory.

ACADEMIC HONESTY AND STUDENT CONDUCT POLICIES

The following guidelines are excerpts from the Simon Fraser University policies regarding Student Conduct and Discipline (<http://www.sfu.ca/policies/gazette/student/s10-01.html>)

Statement of Principle

All members of the University community share the responsibility for the academic standards and reputation of the University. Academic honesty is a cornerstone of the development and acquisition of knowledge. Academic honesty is a condition of continued membership in the university community.

Academic dishonesty, like other forms of dishonesty, includes misrepresentation with intent to deceive or without regard to the source or the accuracy of statements or

*For this matter, custom spreadsheet files are available for some experiments; this is mentioned in the lab manual. Check also on the PCHEM computers (or the course web pages) as new ones may be added during the term. Note that these templates are provided for your convenience but without warranty as to their accuracy and their functionality; use them at your own risk. Actually you are encouraged to develop your own spreadsheets.

findings. Academic dishonesty, in whatever form, is ultimately destructive of the values of the University; it is furthermore unfair and discouraging to the majority of students who pursue their studies honestly. Scholarly integrity is required of all members of the University.

Forms of Academic Dishonesty

The illustrations presented below are considered to be representative but not definitive nor exhaustive of activities which could be considered to constitute academic dishonesty. Plagiarism is a form of academic dishonesty in which an individual submits or presents the work of another person as his or her own. Scholarship quite properly rests upon examining and referring to the thoughts and writings of others. However, when excerpts are used in paragraphs or essays, the author must be acknowledged using an accepted format for the underlying discipline. Footnotes, endnotes, references and bibliographies must be complete. Plagiarism exists when all or part of an essay is copied from an author, or composed by another person, and presented as original work. Plagiarism also exists when there is inadequate recognition given to the author for phrases, sentences, or ideas of the author incorporated into an essay.

- Submitting the same essay, presentation, or assignment more than once whether the earlier submission was at this or another institution, unless prior approval has been obtained.
- Submitting as one's original work, essays, presentations or assignments which were purchased or otherwise acquired from another source (e.g, internet download).
- Using or attempting to use other students' answers; providing answers to other students; failing to take reasonable measures to protect answers from use by other students in assignments, projects or examinations; or submitting identical or virtually identical assignments by students who studied together.
- Submitting false records or information, in writing or orally. This includes the falsification or submission of false laboratory results, documents, transcripts or other academic credentials.
- Stealing or destroying the work of another student.

Notification of Standards of Academic Honesty

All members of the university community have a responsibility to ensure that they themselves, and others, are familiar with generally accepted standards and requirements of academic honesty. Summaries of these shall be published in the University Calendar and in the Registration Handbook. Ignorance of these standards will not preclude the imposition of penalties for academic dishonesty.

Course instructors shall inform students at the beginning of the semester of any special criteria of academic honesty pertinent to the class or course. Encouragement of group work varies greatly between disciplines, and instructors shall convey to their students the acceptable level of group work.