CHEM 6 – Physical and Biophysical Chemistry Laboratory
Winter Quarter 2016-2017
Schedule

Week 1	January 6	Organizational Meeting, F 14:50 - 15:55, 151 Crellin
Week 2/3	Jan 9/16	Lab 1 Due date: Jan 30, 5 pm
Week 4/5	Jan 23/30	Lab 2 Due date: Feb 13, 5 pm
Week 6/7	Feb 6/13	Lab 3 Due date: Feb 27, 5 pm
Week 8/9	Feb 20/27	Lab 4 Due date: Mar 13, 5 pm

Instructor:

Dan Weitekamp 228 Noyes
Ext. 6579 weitekamp@caltech.edu

Teaching Assistants:

None.

Course Web Site:

http://chemistry.caltech.edu/courses/ch6/index.html
Or navigate to Courses/Ch6 from chemistry.caltech.edu

The menu of experiments will be fixed, with all students working on the same experiment in each of the four 2-week slots indicated above. Further information will be available at the organizational meeting.

List of Experiments

In each quarter, students will perform four laboratories selected from the list below. Some experiments are installed in B133 Noyes (formerly 33 Noyes), which is specifically designated for this laboratory course, while others are done using research facilities within the Chemistry department.

1. Scanning Probe Microscopy
   Surface imaging with scanning tunneling microscopy (STM) and atomic force microscopy (AFM) (BI).
2. Diode Laser Spectroscopy: Fundamentals of Chemical Instrumentation and Data Acquisition
   Introduction to signal processing, methods for acquiring data, data analysis, and applying diode laser spectroscopy (B133 Noyes).
3. Raman Spectroscopy
   The vibrational spectra of polyatomic molecules; Resonance Raman spectroscopy of cytochrome-c (BI Laser Resource Center, basement).
4. Microwave Spectroscopy
   The pure rotational spectrum. Molecular structure, dipole moment and quadrupole hyperfine interactions of OCS (B133 Noyes).
5. Ion Chemistry by Ion Trap Mass Spectrometry
   Structures, thermodynamic properties, and reaction dynamics of organic and biological ions in the gas phase. Determination of proton affinity of betaine (BI – Beauchamp lab).
6. Electron Paramagnetic Resonance
   EPR spectra of Mn(II), V(IV) and 2,2'-diphenyl-1-picrylhydrazyl (Crellin Basement).
7. Surface Science
   Surface structure by low energy electron diffraction (LEED) (B133 Noyes).
8. Ultrafast Spectroscopy
   Multi-photon spectroscopy; pulse autocorrelation.
9. Nuclear Magnetic Resonance
   Methods of Fourier transform nuclear magnetic resonance; Two-dimensional chemical exchange (Mead or Crellin NMR).
10. Single Molecule and Super Resolution Microscopy
    Fluorescent labeling of cell components for imaging beyond the diffraction limit (Not offered W 2016-2017).

Independent Project (Student's second term only, pending approval of instructor). This option substitutes for two regular experiments (4 weeks). See below for additional information.

Introduction to the Physical Chemistry Laboratory

The principal objectives of the Physical Chemistry Laboratory are to acquaint the students with a variety of phenomena that are the contents of this subject, to familiarize them with a number of the techniques in use, to develop a facility in the making and interpretation of measurements, and to give them some basis for judging the reliability of data. Both lecture and laboratory courses in physical chemistry are in the process of constant change, with considerable emphasis on the material necessary to enter rapidly into those fields that are most alive and developing.

The course will familiarize the participants of the class with several techniques useful in research even for those whose interests are primarily in organic, inorganic or biological chemistry, and should help develop the skill in making and interpreting measurements. But particularly, it should help cultivate an appreciation for the meaning of measurements, a healthy skepticism as to their reliability, and an open mind regarding the possibility of hidden sources of error.

Chemists in all fields increasingly are using physical methods, often with commercial instruments of a high degree of complexity that they cannot expect to understand in full detail. It is necessary to develop a healthy attitude towards these "black boxes". In general, one should not be afraid to attempt the repair or even modification of commercial instrumentation in the interest of making progress toward the solution of research problems. A little common sense and well-developed power of observation can carry one quite far with even the most complicated instrument. In the physical chemistry laboratory students will be conducting experiments with both commercial and "home-made" equipment. They are encouraged to make simple repairs on equipment when they are called for.

The present wealth of research equipment available to scientists offers tremendous possibilities for the rapid accumulation of knowledge. It may have some tendency to discourage the independence, resourcefulness, and manipulative skills necessary in a really outstanding experimentalist. It would be unfortunate indeed if the progress of science were dictated by the limitations of equipment with which instrument makers see fit to provide us. The existence of a commercial instrument in general indicates that a corresponding technique is already well established. A new field may well require a revolutionary approach (scanning probe microscopy is a good example). Some students may elect to participate in the development of new apparatus and experiments to be incorporated into the physical chemistry laboratory course in the future. Students should feel free to offer suggestions for new experiments.

A final but important objective of this course is to develop the habit of keeping the kind of laboratory record one will later find important in any research activity.

<u>Requirements</u>

Completion of four experiments and submission of Lab Notebook every two weeks. Students will in general work in pairs.

In general, each group will do one lab each two week period. Plan to run experiments in the first week, and write-up/analyze results in the second week. Your laboratory notebook for a given lab will be due at the date specified in the schedule for each laboratory period.

<u>Come to lab prepared. We expect that you will have read the laboratory outline and answered all pre-lab questions (in your notebook!) before coming to class.</u> You should be sufficiently familiar with the work to be done that you can organize your efforts with some efficiency. For many of the experiments, supplementary reading is recommended. References are given for many of the experiments and a number of useful texts are on reserve in the library. Remember, 10% of your grade will be based on the TA evaluation of your preparedness.

Most labs are designed to take approximately one afternoon to complete. Some labs will require multiple sessions (these in general have reduced write-up/analysis requirements).

Your lab notebook will contain all written work – answers to prelab questions, observations and data recorded during your experiment, and follow-up analysis, discussion, and conclusions. You should make entries into your notebook as you work. No separate report is required.

Grading

You will be graded on your performance in the laboratory, and your analysis and interpretation of experimental data present in the laboratory notebook. There will be no examinations administered in this course.

50% Experimental write-ups and analysis (in notebooks): including analysis of results (figures, tables, error analysis, and comparison with literature values or theoretical models where appropriate), conclusions (with discussions where appropriate), and any questions to be answered in the laboratory notes.

30% Laboratory Notebook: The notebook will be graded on thoroughness, organization, and orderliness (not necessarily related to neatness).

10% TA evaluation of preparedness and effectiveness in the laboratory. It is essential that you read the material handed out for each experiment before coming to the lab and be prepared to ask and answer questions regarding the experiment.

10% Prelab questions.

LATE PENALTIES :

-5% for reports turned in less than 24 hours late.

-10% for reports turned in more than 24 hours late.

-20% for reports turned in after Finals Period ends, in the absence of an extension by way of an E or I grade.

Request extensions from the instructor only, not the TAs. In general, only illness or unanticipated and sudden need to travel will be valid excuses and documentation will be requested.

Instructions Regarding Notebooks

You will require two quadrille lined laboratory notebooks in which all data are to be directly recorded, and in which experiments are to be written up. At most times one notebook will be in your hands and the other in the custody of the teaching assistants. Turn your reports in on time! Unless prior arrangements have been made with the instructors, a penalty of 10% will be deducted from the grade received on all overdue reports.

Make your notebook entries **contemporaneously**, as you do your work. Do NOT take notes anywhere else and enter into the notebook later. All pre-lab notes and answers to pre-lab questions should be entered in your notebook as well.

1. Name of Experiment, date performed, and name of laboratory partner.

2. Prelab notes and answers to prelab questions.

3. Original data (indicate date and time recorded to preserve order in which data were recorded -this can be important when interpreting results at a later date). If you have used reasonable care, it should not be necessary to recopy the data, but we insist that the record be understandable and easy to follow. A tabular recapitulation of the data may sometimes be useful. When data or their interpretations are presented in the form of graphs (which are to be firmly secured to the notebook), label them with regard to content and indicate units. All points on graphs should be indicated by circles, crosses, or other clearly visible symbols. It may be useful to indicate probable errors for data points on graphs.

4. Interpretation of the data. It is unnecessary to outline either the theory involved or the experimental procedure unless some departure is made from the directions provided. Indicate clearly the method of your calculations, the actual data used in each step, and the results of each step. It should be possible for the instructor to reproduce all calculations without undue effort. Where several parallel sets of data are to be treated, the results of the various steps of the calculations should be presented in tabular form. Indicate clearly your final results, in tabular form where possible. Designate units of all quantities. Use ink and cross out errors with a single line. You may need that data later. Do not tear pages from your notebook.

5. Discussion of errors. This should be brief but definitive. Avoid trivialities. You will find that the accuracy of data and derived quantities varies considerably in the different experiments. In some cases you may be able to measure quantities with errors of a few tenths of a percent, but in others large errors may be unavoidable. Do not be misled by the apparent precision and reproducibility of measurements. Watch for sources of *systematic error* that may sometimes be very large. Remember that a result may be in question not only because of errors in measurement but also because of the interpretation of measurements by use of inadequate or approximate theory. When the discussion of errors is not explicitly outlined in the experiment, we desire your considered opinion as to the major source(s) of error with a brief but quantitative elaboration.

6. Answers to any questions in the laboratory notes.

Hints

The notebook should be a log or diary of your activities that should be readable by others. Using titles, entering information in outline form, and annotating with comments and description all help to keep the notebook well organized. It will be helpful if you number pages and provide a table of contents for your notebook.

You should record your data and observations in systematic and compact form. Where possible, enter observations in tables. Label all quantities and indicate units. Properly identify all computer output and spectra and attach them in the laboratory notebook (one attachment per page).

Be especially careful in reading dials and meters where orders of magnitude or other scale factors are involved! If you exercise a little care at this point, you can avoid much subsequent work. Anyone familiar with the experiment should be able to follow your record without undue effort.

You may find it helpful to keep your notebook on one side of the page only (e.g. the right hand page) and use the back for scratch calculations.

Perform your analysis and write up your results as soon as possible after completing the lab work. The experiment will be fresh in your mind.

Many of the experiments involve the determination of quantities (e.g. a dipole moment) that may already be well known. You should compare your results to known values and comment on any differences. Agreement will generally be quite good. If this is not the case you should check your calculations for possible errors.

Collaboration Policy

Ch6 is designed to introduce you to "realistic" laboratory situations while also teaching you about various techniques in experimental physical chemistry. For this reason the collaboration policy is very loose; in fact, collaboration is encouraged. Lab partners can work together on all aspects of the lab including data plotting and analysis. Groups may also work together figuring out how to do analysis, comparing answers to prelab/report questions, and discussing other issues that they may have. However, some restrictions to the above policy are necessary:

1. Each student should submit a ***distinct*** report.
This means that *everything* in the report should be in the student's own words; while students in groups may have similar answers/explanations, it should be apparent that each student understands what is going on and can explain it.
2. Each group should use its own data unless they have been given explicit permission by the TA or a Professor.
The equipment and experiments in Ch6 are often very sophisticated, but there is a price to complexity. Experiments don't always work as expected; this is the nature of science. In these cases, students may be given raw data (but not analysis) from past years, or even from another group in the same year. However, the notebook should explicitly identify such data *and* state what is wrong with their data and why their data may be bad.
3. Each group must perform their own full data analysis.
4. While members of a group may use the same plots, each student should be able to recreate all plots and explain any part of their report if asked to do so by the TA.

Ch6 INDEPENDENT PROJECT

If you have taken one term of Ch6 already, then with instructor's permission, you can choose to do an optional independent project, which will SUBSTITUTE for TWO labs. It should take approximately four weeks worth of time to complete. Since we cannot possibly design projects to cover every physical chemistry technique, the independent project will allow you to work on any (feasible) physical chemistry project of interest without having a pre-designed lab. This will give you wide flexibility in the systems studied and the methods used.

Students will do two regularly scheduled experiments in the first five weeks. The project will be done in the last two periods, but you will be required to propose and design the experiment in the first half of the class. Only students who have had one term of Ch6 will be permitted to do this project.

EXAMPLES

1. Measure electron transfer rates between a donor and acceptor by varying the acceptor concentration and comparing to predictions based on calculation using Marcus theory
2. Characterize isotope shifts, effect of substitution, etc. by spectroscopic characterization (using at least two methods, e.g. IR and Microwave) of two species and compare with quantum chemistry calculation
3. Unimolecular decomposition of an ion in a trap; compare to statistical rate theory
4. Measure rates of a gas phase reaction using the diode laser apparatus to follow the loss of reactant or formation of a product.
5. Size-dependent spectroscopic properties of quantum dots
6. ESR and UV/Vis or IR to study properties of environment of a metal in a metal-containing protein.

SCHEDULE

Week 2. Project proposal (1-2 pages) due Friday (latest).

Weeks 3-5. Develop details of project design. Update of plan should be turned in weekly. Detailed design of experimental plan due at middle of week 5. Preparation (reagents, glassware, machining etc) should also be anticipated and done/ordered.

Weeks 6-9. Execute experiment. Perform analysis.

Week 10. Complete analysis. Turn in laboratory notebook.

GUIDELINES:

Content. This is a physical chemistry laboratory experiment. You are to design an experiment to test a physical chemistry hypothesis using physical experimental methods. We strongly discourage (a) instrument building/new techniques (due to time problems), (b) try-it-and-see experiments with no hypothesis/prediction, (c) projects which involve substantial time spent on nonphysical or nonexperimental aspects, e.g. synthesis, computation.

Experimental emphasis. Since this is a physical chemistry lab, the project should have a significant experimental component. We expect that you will spend a minimum of 12 hours in the lab, preferably more. There is a limited budget for new glassware, machining, chemicals, etc. You are encouraged to apply more than one method that you have learned/is available.

Due to the severe time constraints (and Murphy's Law), you must limit your experiment to existing, functioning apparatus, except for minor changes, e.g. straightforward modifications, adaption of apparatus. Past attempts at similar projects which involve newly fabricated equipment have too often ended in students spending the entire time trouble-shooting with no results and no time spent doing science per se.

You may use methods available in other labs, e.g. the dye laser system in the BI Laser Resource Center, pending permission of the relevant authority figures.

Analysis. The analysis should involve concepts and methods developed during Ch 6 or Ch 21/24. You are expected to apply error analysis as needed. It can involve computational work, e.g. quantum chemistry, but these should not be the major emphasis of the project.

Write-up. To be consistent with the rest of the course, we ask that you turn in a detailed notebook following the guidelines set forth in the course for other labs.

HOW DO WE DECIDE WHAT PROJECT TO DO?

Ideally, we would like you to come up the idea for the project on your own. However, since this may be very difficult, some ideas for projects are listed above. If you have any other ideas, even if they are very general, feel free to talk to the TA and work together to come up with a more specific idea. Otherwise, you can use one of the general ideas below and develop a more specific plan (or modify one of the plans). If you think a project listed below sounds interesting but you don't know how to proceed, then feel free to talk with the TA.

Week 1-2. Proposal

Once you have decided on a project and worked with the TA to come up with an initial plan, you will need to write up and submit a proposal. This should be submitted as soon as possible into the term, but no later than the end of Week 2. This will let you start working on preparing for your project.

Your proposal should include

- OBJECTIVE. A concise statement of the goals (what you want to measure/do) including a ***statement of your hypothesis.***
- INTRODUCTION. A brief summary of the background (why is this interesting).
- PLAN OUTLINE. What needs to be done.
- TIMELINE (a schedule).

The proposal should be about 1-2 pages long. This proposal will factor into your grade for the project and will be graded on clarity, completeness, and understanding of the project.

Week 3-4. Develop Detailed Experimental Plan

When your proposal is approved you should start writing down a more detailed plan for your experiment. This should be

Itemize what needs to be done in detail (what reagents, how much, where will they be ordered; what instruments, what modifications need to be done, if any;

Signal-to-noise considerations (will it work; what sensitivity is required, how much sample do you need).

Analysis and error analysis plan

Anticipate possible problems.

You have a small budget to purchase supplies. You will need to purchase them 1-4 weeks in advance, to allow for typical order processing and delivery times, so do not delay!

If you use departmental instrumentation, you may need to line up permission and scheduling in advance. For some instruments, e.g. NMR, you or the TA may need training.

Week 5-9. Experiment Time

You should primarily work independently on your project; however the TA will be available to show you how to work the instruments, help troubleshoot, and answer other questions.

Week 9-10. Analysis and Write-up

Grading

Grading will be based in equal parts on

1) Proposal
2) Planning and Preparation
3) Experimental approach; problem solving
4) Analysis (including quantitative use of statistics).