

Chem 15: Chemical Equilibrium and Analysis Laboratory Fall 2008

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Course Description

Laboratory experiments are used to illustrate modern instrumental techniques that are currently employed in industrial and academic research. Emphasis is on determination of chemical composition, measurement of equilibrium constants, evaluation of rates of chemical reactions, and trace metal analysis.

Objectives

Understand through practical application concepts and techniques of analytical chemistry including

- Accuracy and Precision
- Limitation of gravimetric and volumetric measurements
- Solution preparation and handling
- Calibration
- Use of blanks, spikes and internal standards to validate method performance.

Lab Hours

Lab Times: We will try to have the lab open 4 afternoons per week, depending on student and TA availability.

Locations: Noyes 33, Keck 136 and 204, and possibly others.

Experiments

1. Water Hardness by EDTA Titration
2. ICPMS - Trace metal analysis
3. Competitive Sorption of Phosphate and Oxalate on soils by Ion Chromatography
4. Enzyme-Substrate Interactions Employing Michaelis-Menten Methodology/UV-VIS
5. LC-MS-MS Analysis of Melamine and cyanuric acid.

Grading

You will be graded on your performance in the laboratory, your laboratory notebook and the analysis and interpretation of your data. There will be no separate papers or examinations in this course.

Each experiment will be worth 100 points, i.e. they will be weighted equally in your final grade. Number grades will correspond to letter grades such that A \geq 90%, B \geq 80%, C \geq 70%, D \geq 60%, F < 60%.

You will have two lab notebooks for this course. While one is in the hands of a TA being graded you will be recording the next experiment into the other. After you complete each experiment you will turn in your notebook for grading. The grade for each experiment will be divided as follows:

10% TA's evaluation of preparedness and effectiveness in the laboratory. It is essential that you have read the material and thought through the work you will do before you walk into the lab. Be prepared to ask and answer questions regarding the experiment. This includes answering any pre-lab questions in the laboratory notes.

40% Laboratory Notebook. Your record of your work in the lab will be graded on thoroughness, organization, and orderliness. While neatness will not be graded for specifically, we must be convinced you can read your own writing.

50% Experimental write-up, analysis, and interpretation. Your analysis may include figures, tables, and error analysis; conclusions supported by evidence and statistics; and answers to questions posed in the laboratory notes.

Your lab notebook will contain all written work - answers to prelab questions, observations and data recorded during your experiment, follow-up analysis, discussion, and conclusions. You should make entries into your notebook as you work. Include copies or originals all essential spectra, chromatograms and graphs taped in your notebook. No separate report is required.

All reports must be turned in. Failure to do so will result in a course grade of “fail.”

Due Dates and Times

Lab notebooks are due at 5:00 pm on the dates specified. Turn your notebook in to the appropriate TA or TA's mailbox.

Late Work: One reason we establish due dates is so that all reports can be graded at the same time, thus ensuring some degree of consistency. We understand, however, that unexpected situations may arise. Thus, you will get an allowance of 5 late days for turning in reports. (For this purpose, a day is the fundamental quantum - any fraction of a day late uses one day of your allowance). To use these late days you must state in your notebook how much of your late allowance you are using. Once you have exhausted this allowance, late reports will be penalized 10% of graded value. For example, a grade of 90% will be 81% after the penalty is applied. No exceptions to this policy will be made without an appropriate note from the Dean's Office.

Class Policies

- Each student must maintain their own laboratory notebook. Refer to the more detailed suggestions below on keeping a detailed notebook.
- You must complete all laboratory work during normal lab hours.
- Students may only work in the lab when at least one TA or Instructor is present.
- Dress code:
 - You must wear closed-toed shoes.
 - You should wear a standard, long-sleeved lab coat. We will provide this.
 - You must wear long pants that reach your ankles.
 - As always, you should wear safety glasses with side shields.
 - If you have long hair, you should keep it pulled back.
 - You will be asked to leave the laboratory until you comply with these requirements.
- A few other notes:
 - On the first day of lab (check-in), you will be assigned four drawers and supplies. You will verify you have all items and sign a laboratory inventory sheet. Please keep track of your supplies and keep them clean.
 - When you are done working in lab for the day, please make sure that you clean your work area and that any waste is properly tagged, capped and placed for pickup.
 - Label all chemicals clearly, including waste. You will be instructed in the use of the Caltech waste tag system and

- expected to use it at all times. This will be included in the preparedness and effectiveness evaluation of the TAs.
- Bring a permanent marker (Sharpie) for labeling solutions.
 - Honor Code. You must do your own work in the lab, keep your own lab book, and write reports that reflect this work and your notes. You may discuss the labs and reports with each other, but your report must reflect your own understanding.
 - Collaboration
 - We are building this course to provide realistic as well as timely analytical problems for you to solve. In the real world analysts frequently work together as teams, and regularly collaborate in person and through electronic means. Thus, we encourage you to collaborate with each other. In the event we have students work in small groups these groups may work together on all aspects of the lab including analysis and interpretation. Groups of students in the course may also work together to figure out analyses, compare answers to pre-lab or post-lab questions, etc. The following restrictions will, however, apply:
 - Each student shall submit a *distinct* report. Everything must be in your own words. It should be apparent that each student understands what is going on and can explain it if asked to do so by an Instructor or TA.
 - You must use your own data, obtained individually or in your own group. Any exceptions require explicit permission from one of the Instructors. This may happen in the event that one or more groups are unable to obtain their own data due to difficulties with equipment. Experiments often fail, and even modern instrumentation has ample opportunity for breakdowns. Unfortunately, this is the risk associated with including more sophisticated experiments in the course.
 - Each group must perform their own full data analysis.
 - Members of a group may use the same graphs and charts in their report. However, each student should be able to recreate all plots and explain any part of their report if asked to do so by a TA or Instructor.

Instructions for Laboratory Notebooks

You must have two quadrille lined, hardbound lab notebooks with numbered pages. One acceptable notebook the Caltech Bookstore sells is National Brand Chemistry Notebook Item No 43-571. At most times one notebook will be in your hands and the other in the custody of the teaching assistants.

Data is to be recorded directly into one of these notebooks with a blue or black ball point pen. 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 and post-lab questions should be entered

in your notebook as well. Do not tear pages from your notebook. Cross out errors gently with a single line. You may need that data later!

Original data should have some indication of 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 is often 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.

Interpretation of your data. It is unnecessary to outline either the theory involved or the experimental procedure unless some departure is made from the directions provided, or you have been asked to develop your own procedure. 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. Output of a spreadsheet is acceptable. Indicate clearly your final results, in tabular form where possible. Designate units of all quantities even and especially at intermediate steps.

Discussion of Errors. This should be brief but definitive. Avoid trivialities. You will find that the accuracy of data obtainable 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. 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.

A few suggestions for maintaining a good notebook follow:

1. Allow space at the front (5 pages) for a table of contents. Update this after every day in the lab.
2. Title and possibly subtitle pages. Use these titles in your table of contents.
3. Write the notebook as a log or diary of your work so that it could be followed by others.
4. Record your data and observations in systematic and compact form. Where possible enter data and observations in tables.
5. Label all quantities and indicate units.
6. Thoroughly identify any computer output.

7. Carefully note orders of magnitude, scale factors, and units that you read off of dials, labels, or computer screens. This will save confusion and subsequent detective work later on!
8. Once you complete the experimental work write it up as soon as possible. Recollecting all the details later is time consuming and essentially a duplication of effort.

Tentative Schedule

Reports are to be turned in to the appropriate TA or TA's mailbox by 5:00 PM on the date listed as due.

Week #	Week of	Lab in Progress		Writeup Due	
		Track A	Track B	Item	Date
1	29 September	OM; Check-in.			
2	6 October	Titration	Titration		
3	13 October	ICPMS	IC		
4	20 October	ICPMS Start IC	IC Start ICPMS	Titration	10/24
5	27 October	IC	ICPMS	Second Writeup	10/31
6	3 November	UV-VIS	LC-MS-MS	Third Writeup	11/3
7	10 November	UV-VIS	LC-MS-MS		
8	17 November	LC-MS-MS	UV-VIS	Fourth Writeup	11/21
9	24 November (Thanksgiving)	LC-MS-MS	UV-VIS		
10	1 December	Cleanup and	Checkout	Fifth (Last!) Writeup	12/5
Finals	12/10 - 12/14				