

ANALYTICAL CHEMISTRY LABORATORY SYLLABUS FALL 2006

Laboratory: 3-4 hours, Mondays and Tuesdays, 1-5 pm, CP 236

Pre-Lab Recitation: 1 pm, CP-208

Teaching and Laboratory Assistants: Smita Joel, Erin Peters, and Daniel Scott

Lab Fees: \$90 – See 226 Website for more details at
http://www.chem.uky.edu/courses/che226/005-Lab_Charges.pdf

Lab Hours:

- Sections 1 & 3 - Mondays, 1-5 pm
- Sections 2 & 4 - Tuesdays, 1-5 pm

TEACHING ASSISTANT CONTACT INFORMATION

	Smita Joel	Erin Peters	Daniel Scott
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Office Hours	2-3 pm Tue 4-5 pm Thur	10-11 am Mon; 1-2 pm Wed; 1:30-2:30 Thur	9-10 am Wed 2-3 pm Thur

LABORATORY EXPERIMENTS

Expt	3 Credit Students	4 Credit Students	Grader
1	Laboratory Techniques	Laboratory Techniques	D. Scott
2	Gravimetric Chloride Analysis	Gravimetric Chloride Analysis	E. Peters
3	Titration of Zinc with EDTA	Titration of Zinc with EDTA	S. Joel
4	Sodium Atomic Emission	Sodium Atomic Emission	S. Joel
5	Iron Absorption Spectroscopy	Iron Absorption Spectroscopy	D. Scott
6	Molecular Fluorescence	Molecular Fluorescence	E. Peters
7	-----	Acid-Base Titration	D. Scott
8	-----	Electrogravimetric Copper	E. Peters
9	-----	Kinetic Determination of Glucose	S. Joel

TENTATIVE LABORATORY SCHEDULE

The schedule below is very rough and very tentative. For example, it may take greater or lesser amounts of time to provide you an overview of the experiments. The *instrumental* experiments will be scheduled for you on a specific date later in the semester, and will take only one lab period to complete. However, owing to a lack of equipment, students will be rotated through the experiments. You may be scheduled early in the semester, late in the semester, or both. You are largely “on your own” for Experiments 1-3 (and 7 for the 4-credit students). You must plan

ahead and prepare well ahead of time to get them completed in a timely manner. It may take you greater or lesser amounts of time to get them done than the tentative schedule indicates.

The days on which the Pre-Lab Lectures will be given may likely vary. Just keep coming to CP-208 at 1 pm unless informed otherwise.

The logistics are further complicated because of the loss of Monday, September 4 to the Labor Day holiday (shown as *No Lab* in the table below), so the Monday sections will generally be one week behind the Tuesday sections.

While the overall situation in the 226 Laboratory may sound complicated, this is a good experience for you in your future career. Life, even life in a lab, is a lot like this. Professionals have to plan well ahead and then invariably adapt, often to last-minute or emergency situations.

Monday	Tuesday	Pre-Lab Lecture Topics CP-208	In Lab CP-236
8/28	8/29	Lab Syllabus Check-in procedure Lab techniques Tour of the Lab	Check in Clean out locker thoroughly Submit Cl^- unknown container Begin Techniques Lab
<i>No Lab</i>	9/5	No Pre-lab lecture	Techniques Lab Crush/dry chloride unknown Clean crucibles, dry in oven Submit Zn unknown container
9/11	9/12	Gravimetric Chloride Zn EDTA	Weigh out/precipitate/dry Cl^- samples Finish Techniques Lab
9/18	9/19	Acid-Base Titration	Finish Cl^- Lab Prepare EDTA solution
9/25	9/26	Iron Absorption Quinine Fluorescence	Finish Cl^- Lab Prepare Ca standard solution Standardize EDTA solution
10/2	10/3	4-Credit: Glucose Kinetics	Titrate Zn Unknown Submit Acid unknown container
10/9	10/10	4-Credit: Na Emission Cu Electrogravimetry	Prepare KHP standard solution Prepare/standardize NaOH solution
10/16	10/17	No Pre-lab lecture	Titrate unknown acid <i>Instrumental Experiments</i>
10/23	10/24	No Pre-lab lecture	Complete first 4 experiments <i>Instrumental Experiments</i>
10/30	10/31	No Pre-lab lecture	<i>Instrumental Experiments</i>
11/6	11/7	No Pre-lab lecture	<i>Instrumental Experiments</i>
11/13	11/14	No Pre-lab lecture	<i>Instrumental Experiments</i>
11/20	11/21	No Pre-lab lecture	<i>Instrumental Experiments</i>
11/27	11/28	No Pre-lab lecture	<i>Instrumental Experiments</i>
12/4	12/5	No Pre-lab lecture	CHECKOUT ONLY

Other Important Information - See the CHE 226 website (when it's updated):

<http://www.chem.uky.edu/courses/che226/>

- Guide for *Complete* Lab Reports, including samples
- *Safety in the Analytical Laboratory*
- Laboratory Fees
- List of Containers for Unknown Samples
- Using Material Safety Data Sheets (MSDSs)
- Procedures for Individual Laboratory Experiments

SOME IMPORTANT DATES FOR THE CHE 226 LABORATORY

- **Monday, September 4** – Labor Day. **No Lab (Monday)**
- **Wednesday, September 13** – Last day to DROP a course without it appearing on your transcript. Last day to change grading option: P/F, Credit/Audit.
- **Friday, October 20** – Last day to WITHDRAW from the course. To drop CHE 226, **you must officially check out of lab**. See the laboratory charges section of the website for details.
- **Monday, December 4** – Last day the laboratory is open for Sections 1 and 3. **All students in these sections *must* formally check out by this date. We encourage you to check out earlier if you are completely done with all your lab work.**
- **Tuesday, December 5** – Last day the laboratory is open for Sections 2 and 4. **All students in these sections *must* formally check out by this date. We encourage you to check out earlier if you are completely done with all your lab work.**
- **Friday, December 8** – Last day of classes. All CHE 226 laboratory reports must be submitted by 5:00 pm. **Absolutely no lab reports will be accepted after this deadline.**

UNKNOWNNS

The Teaching Assistants will dispense unknowns and have them ready for you **one lab period following receipt of the appropriate container**. That is, unknowns will *not* be dispensed on the same day as requested. The Teaching Assistants simply do not have the time to prepare unknowns during the busy laboratory periods. Therefore, it is very important that you plan ahead. For example, Experiment #3 requires that you prepare and filter an EDTA stock solution at least one day before you use it; this is critical. Most of the other experiments require you to turn in a labeled volumetric flask to the Teaching Assistants. The unknown is dispensed into the flask and you dilute it to volume with distilled or deionized water and mix thoroughly before taking an aliquot for analysis.

You may not have more than one unknown out until you have passed the Laboratory Techniques experiment (#1) with your final score (0 or 100%). The whole point of this experiment is to master basic lab skills one at a time. It makes no sense, for example, to try to do a titration experiment if you can't read a burette properly. **You may not have more than three unknowns out at any one time.**

LABORATORY NOTEBOOK

Each student is required to have his or her own Laboratory Notebook with carbon copies and *with numbered pages that tear out cleanly*. It does not have to be brand new; you may start the semester using a notebook from a previous course that has some room left in it. Leave at least the first three pages of your notebook empty in order to put in a Table of Contents. The notebook pages must be used sequentially in *historical time order*. For example, while preparing for a lab, don't move ahead several pages and "reserve" five or six pages, for example, for that particular experiment. In the laboratory, **all** information, data, calculations, notes, etc. should be recorded directly into this notebook and **not** on scrap paper. *Information written on anything other than the lab notebook pages will be confiscated.*

Writing should be clear and concise and in ink. Use a ballpoint pen so that the writing comes through on the yellow carbon copy. Errors should be crossed out with a single line and the correction written next to it. Don't scribble completely over the error. Your notebook must reflect the absolute truth of your laboratory experience. Each page of your notebook must be dated.

Before you leave the laboratory each time you have done lab work, one of the instructors must review your work and initial and date your notebook just below where your entries end.

The Lab Report itself can be simply the yellow carbon copy from the lab notebook. You should keep the (original) white copy for your records. Before entering the laboratory, the student should become thoroughly familiar with the experiment and prepare the notebook to make record keeping and report writing more convenient. Title, procedure, theory, and tables for data should be prepared prior to the laboratory period, *vide infra*.

Formal lab reports are required for all experiments except the Laboratory Techniques experiment (Experiment #1). Lab reports should be written according to the handout *Guide for Complete Lab Reports*.

GRADING LABORATORY PERFORMANCE

Except for Experiment #1, which is worth 100 points, each of the other labs will be worth 130 points. The points are distributed as follows:

- **Pre-Lab Write-Up** (15 Points)
- **Subjective Performance Evaluation** (15 Points)
- **Accuracy** (60 points)
- **Laboratory Report** (40 Points)

Therefore, the 6 experiments for those taking the class for 3 credits is 750 maximum possible points, and the 9 lab experiments for those taking the class for 4 credits total 1140 maximum possible points. The total points you accrue during the semester will be converted to a percentage score, % Lab Score = Points earned x 100% divided by 750 or 1140, as appropriate. This % score will then be used for that fraction of the final course grade appropriate for 3- or 4-credit students.

Penalty points can be assessed on lab reports. Lab reports are due *one week* after each experiment is completed. Reports will be penalized 5 points for every week or fraction thereof that the report is late.

Pre-Lab Write-Up (15 Points)

Prior to doing a laboratory experiment, you must understand the lab thoroughly and be prepared to work efficiently. Read through the handout for that laboratory thoroughly and plan your work ahead of time. Except for Experiment #1, you must have essentially written up the “Procedure” and “Theory” sections in your Laboratory Notebook **prior** to beginning the experiment. This should be a logical and structured set of notes that summarize the steps in the experiment. Ideally, this should enable you to do the experiment without referring to the handout, except perhaps for the detailed step-by-step instructions for operating an instrument. The Pre-Lab needs to be summarized in your own words; do not just copy the procedures as written in the handout.

If you wish, you can always rewrite the Procedure and Theory sections more elegantly in the Laboratory Report itself.

You *must* show the Pre-Lab Write-Up to one of the Teaching Assistants and have it reviewed, initialed, and dated, *before* you may begin any experiment. If your Pre-Lab is seriously deficient, you may well be asked to add to or re-do it before you are permitted to begin an experiment.

You *must* also have the work you do each day in the Laboratory and have written in your Notebook reviewed, initialed, and dated by one of the Teaching Assistants *before* you leave the laboratory.

Subjective Performance Evaluation

The Instructor and the Teaching Assistants will be circulating through the laboratory to answer questions, assist you, and also to observe your overall laboratory technique and performance while doing an experiment. Assessing this factor can only be subjective, and the final number you receive on a lab will be discussed among all the instructors in the course. We will try to provide some indications as to the reason(s) for loss of points.

Examples of the things that will lead to loss of points include, but are not limited to –

- Arriving late for the laboratory session, especially if this persists or involves a scheduled instrumental experiment.
- Poor safety practices, especially not having your safety glasses on at all times while in the lab.
- Dirty or disorganized work areas.
- Using dirty glassware – e.g., reagent spots on the inside of burettes or pipettes
- Misuse or abuse of the instruments, especially the balances.
- Indications that you have prepared poorly and do not understand the lab sufficiently. This does not mean “Don’t ask questions.” If you have any questions or need confirmation of some point, *ask*. What is meant here is that “deer in the

headlights” look indicating you’re clueless, or that you are constantly reading the laboratory handout. Major steps of parts of the experiment should have been outlined in your Prelab.

- Not cleaning up your lab bench area and any common work areas that you used before you leave for the day.
- Not turning off (and covering) your balance at the end of the lab.
- Insubordination

In addition to penalty points for not cleaning up after yourself before you leave the lab, egregious sloppiness or major abuse of the instrumentation can result in your being assigned to do general laboratory cleaning tasks at the end of one of your lab periods.

Accuracy (60 points)

Sixty points of the lab report depends on the *accuracy* with which you analyze the unknowns. The value you report for your unknown will be within a certain **range around the true or known value**. Your Accuracy grade will be higher the closer that the value you report for your unknown comes to the true value.

Accuracy is graded in 10-point increments: 60, 50, 40, etc. Each experiment has its own *tolerance* or *window* within which your result must fall for a particular grade. For example, if the true value for your unknown is 1000 (in whatever units), and the tolerance for that experiment is ± 3 parts-per-thousand (ppt) or $\pm 0.3\%$ *relative error*, your reported result must fall in the range 1000 ± 3 , (from 997 to 1003) to earn the full grade of 60. If your result lies outside this range, but within the next set of 3 ppt windows, 1000 ± 6 , you earn a grade of 50, and so forth. If the tolerance for an experiment is $\pm 2\%$ relative error, your result your reported result must fall between 1000 ± 20 , (from 980 to 1020) to earn the full grade of 60 points. Outside this window, but inside 1000 ± 40 , (from 960 to 1040) for 50 points, etc.

If your reported result is wrong because of some calculation error, even something as simple as a factor of 2 or a power of 10, your result is wrong. A wise precaution is to check your calculated final value with the document “Ranges of Values for Unknowns” on the 226 Web site.

The Teaching Assistants will not search through your report for calculation errors and correct them, so be very sure about your calculations before you turn in your report. Just about the only thing an analytical chemist has to show for all her laboratory work and care is a report – some words and numbers on a sheet of paper. A result is just as inaccurate if it suffers from an arithmetic error as from poor technique. If you have taken data carefully, however, a calculation error can be discovered.

If you discover that you have made a calculation error in a report you have turned in, you may re-submit the report with the new calculations and results attached for re-grading. There will be a 5-point penalty for this. So, if you get a report back with a very low accuracy grade, say 0 points, it would be a good idea to recheck all your calculations carefully.

Laboratory Report (40 Points)

The final 40 points of the total lab report grade depends on how well and completely your report is written. You must follow one of the four preparation guidelines listed below for each report. There are sample lab reports on the 226 Web site to help guide you.

- You may write your entire lab report *neatly and legibly* on the white pages of your Laboratory Notebook and turn in the carbons for your lab report.
- You may write most of it this way and insert pages of figures that you prepared on a computer at appropriate places in your report.
- You may write some of it in the notebook and some of the text using a word processor.
- You may prepare the entire report on a word processor.

This is your choice. If you write it out, your writing must be neat and legible. If you use a computer, make sure you back up your work in at least 2 separate places. In addition, if you use a computer, you may be asked to provide us a copy of the files and you must provide them within 48 hours.

The distribution of possible points for each section of the Laboratory Report is shown below:

- Theory – 10 points
- Procedure – 4 points
- Data – 4 points
- Calculations – 8 points
- Results/Error – 4 points
- Discussion – 10 points
- Total – 40 points**

Penalty Points.

- ***Inadequate Reports.*** A 5-point penalty will be assessed for reports considered grossly insufficient or inadequate – for example, one of the required Sections is simply missing. The report will simply be returned to you ungraded. The report should be revised, deficiencies and errors corrected, and the report returned within one week or an additional penalty will be applied. A 1-point penalty will be assessed for each day the report is late thereafter, or until the report is considered adequate in exposition.
- ***Late Reports.*** Laboratory Reports are due **one week** after the lab has been completed. Some students have a tendency to allow reports to pile up until the last week or two of the semester. This is almost invariably disastrous and should be avoided at all costs because this does not give you a chance to repeat an experiment if poor results are obtained. Reports prepared hurriedly are extremely prone to simple calculation errors which negate even the most painstaking lab work. Lab reports that are not turned in on time will have 5 points deducted *for each week or fraction thereof* that the report is late.
- ***Repeating an Experiment.*** If you wish to redo an experiment on which you received an Accuracy grade lower than you think you can get by repeating the experiment, you may ask the Teaching Assistant for a new unknown and reschedule a time to do the experiment if necessary. There is a 10-point penalty in the Accuracy grade for getting a

second unknown when the new report is graded, 20 points for the third unknown on a particular experiment, etc. **You must allow your TA at least one full lab period to prepare a new sample.**

LABORATORY REPORT FORMAT

Lab reports should have the following sections:

I. Procedure - Correct reference to the handout (experiment number and title)

1. A detailed list of the experimental procedure.

II. Theory - The chemical reactions that were performed.

1. The reagents and their roles in making the various reactions occur.
2. Chemical structures of important reagents.
3. Briefly describe any important instrumentation and how the method works.
4. Important definitions

III. Data - All *raw data* tabulated, that is the actual data taken down and written in the lab notebook while doing the experiment. Tables or places for raw data should be set up before you come to lab. **Tables must have *descriptive titles and column headings***, not just "Data for Experiment 2". Units must **always** be clearly indicated. If more than one, number each table sequentially.

1. Any calibration plots (figures). Figures must have a **descriptive caption** and units on the axes. If more than one, number each figure sequentially.

IV. Calculations - *ONE* sample calculation to show how the results and error (standard deviations) were obtained. It is not necessary (nor desirable) to show a calculation for each trial or sample.

V. Results and Error - Tabulated results from each trial and the mean (average value).

1. Analysis - Precision of results (standard deviation)

VI. Discussion - Restatement of results

1. The precision and what that implies about the overall validity of the results.
2. Systematic errors (physical and chemical) that can affect the experiment.
3. How various errors were introduced.
4. What can be done to minimize the error?

- Laboratory Reports are due one week after the lab has been completed. A typical report is about 3-5 pages long, plus any figures.
- The teaching assistants are not responsible for finding or correcting arithmetic errors that will affect the grade for the report.
- A lab report that is considered insufficient or incomplete, such as one that is missing one or more of the items listed above, will be returned to the student ungraded. The report must be brought up to minimum standards and returned **within one week**. A 5-point penalty will be applied.
- Lab reports that are not turned in on time will have 5 points deducted *for each week or fraction thereof* that the report is late.

LABORATORY ATTENDANCE

Attendance in Laboratory is mandatory for a passing lab grade. Barring an excused absence, you must attend your scheduled lab section throughout the semester until all your labs are completed.

Because of a limited number of instruments, the instrumental experiments (#5-9) will be scheduled for you to perform on *specific dates*. Often, these will be scheduled for students to work in pairs. If you miss one of these labs, you may reschedule it only if there is an opening. You must get permission from a Teaching Assistant, who will need to find an open slot to reschedule the lab for you.

If you fall behind in the lab, you may work on the afternoon that you are not normally scheduled, but only with permission of the TA and only if there is a spot available for you to work. As the lab is fairly crowded, you will probably not be able to work at your “normal” lab bench.

Revised August 23, 2006

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