

CHEMISTRY 222 L

QUANTITATIVE ANALYSIS LAB

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Introduction

It would probably be fair to say that the laboratory experience is the essence of Quantitative Analysis. A great deal of time and energy is devoted thereto, and the significant contribution of the lab average to the final grade reflects this emphasis.

The lab experiments/analyses listed in the schedule below will be performed this semester. The lab work will be carried out **only during the scheduled lab periods** (i.e. Monday and Wednesday, 1:15-4:15pm...the lab will close at 5pm). There are 3 lab periods assigned to each lab experiment (except for the acid-base titration lab during week 1) – this means that you will have at least 9 hours of lab time to complete each experiment. Although the lab experiment sequence and schedule presented below are seemingly rigid, the lab instructor reserves the right to be “flexible” with regard to the scheduling of lab experiments:

Lab Experiment	Dates	Due Date
Acid-Base Titration	Wed. 1/16	---
Alkalinity of Soda Ash	Wed. 1/23, Mon. 1/28, Wed. 1/30	Mon. 2/4
Gravimetric Determination of Sulfate	Mon. 2/4, Wed. 2/6, Mon. 2/11	Fri. 2/15
Identification of a Weak Acid	Wed. 2/13, Mon. 2/18, Wed. 2/20	Mon. 2/25
Mohr Determination of Chloride	Mon. 2/25, Wed. 2/27, Mon. 3/11	Fri. 3/15
Spectrophotometric Determination of Zn	Wed. 3/13, Mon. 3/18, Wed. 3/20	Mon. 3/25
Complexometric Determination of Cu	Mon. 3/25, Wed. 3/27, Wed. 4/3	Mon. 4/8
Determination of Iron in an Ore	Mon. 4/8, Wed. 4/10, Mon. 4/15	Fri. 4/19

Careful planning will be necessary to make the best use of time in the laboratory. You may occasionally have more than one analysis and/or procedure in

progress simultaneously. Remember that you are scheduled to spend a minimum of six hours weekly in the lab doing manual operations. This **should not include** reading the lab handout (other than to “refresh your memory” with regard to what you’ve already read prior to starting the lab), preparing write-ups in your lab notebook, waiting for things to dry in the oven and/or cool off, or keeping your neighbors company. **You must be VERY familiar with the lab procedure, set up your notebook accordingly and have a plan of action for completing the work BEFORE walking into the lab on the first scheduled day of the experiment!**

Lab Reports

Unlike most other chemistry courses, in Quant it isn’t customary to prepare formal lab reports. Rather, the student is expected to submit photocopies of the appropriate pages from your lab notebook for each experiment on or before the due dates listed in the schedule on page 1. Your laboratory notebook represents a permanent record of the work that you complete toward some intended goal, whether it be for a lab experiment in a chemistry course or for a potentially patentable idea that you want to test and refine as necessary (testing an idea to see if it works is called “reduction to practice” in patent terminology). For such a record to be of any subsequent use, it’s necessary that the entries be legible and carefully organized in some sensible sequence. If you do not do this, then the notebook entries that you end up with will not be a lot of help to you while you’re doing an analysis or to me once you’ve finished (or if you need help with a problem that requires me to understand what you did). There should be sufficient detail and organization to your lab notebook such that someone “skilled in the art” (more patent terminology...in this case, this would be someone having knowledge and skills in chemistry) could read and understand what you did, and could carry out the same experiment based solely on the information recorded in your notebook.

In terms of a laboratory/research notebook, you should have an 8-1/2 by 11 inch or larger permanently bound (not spiral) notebook with pages numbered in the upper outside corners (you may have to number the pages yourself). You should sign and date each page of the lab notebook once you have completed recording information and/or data on that page (sign and date on the bottom of the page). The point of using a large-format book is so that computer printouts can be neatly affixed therein. If for whatever reason you elect to use the more common 7 by 9-1/2 inch format, expect that printer output won’t fit and will have to be trimmed. Each insert to the notebook must be fastened down to a blank area of the page at all four corners, minimum. It is unacceptable for pages to be loose, to fold out, or to protrude from the edges of the notebook when closed.

The college bookstore sells a lab notebook that, in my opinion, is an excellent option for Quant lab – it's "National Brand Laboratory Notebook", #43-591, 60 sheets, 5x5 quad, 8.5"x11", price = about \$9. The binding is flexible enough to allow for decent photocopying of lab work pages/submissions for grading purposes.

All data are to be recorded directly in the notebook in permanent ink, not on scraps of paper, etc. There are to be no erasures, use of correction fluid (e.g. "Wite-Out"), or other attempts to obliterate entries. If an erroneous entry is made it is to be voided neatly with a single line. An X may be used for large areas. No entries are to be made underneath computer printouts or other inserts.

The first two or three pages of the notebook are to be reserved for a table of contents. This must be kept up to date, as work lacking an entry therein will be regarded as absent.

Photocopies of the appropriate notebook pages are to be turned in to me on or before the appropriate date. Although my preference would be to grade and comment on your work directly in your notebook, I want you to have your notebook available at all times so that you set up the lab notebook pages with detailed procedures, data tables, etc. for future experiments.

Here are some conditions that contribute to difficulty in finishing the lab work satisfactorily within the allocated time:

- Lack of discipline. It is essential that you plan to spend the entire 6 hours in the lab each and every week, and that the work to be done be planned carefully beforehand so that you're working the whole time. If you habitually arrive at lab around 1:30pm ("it's the only time I have to eat lunch") and leave at 3:00pm or before ("I forgot to put so-and-so in the oven to dry so there's nothing to do") you're only spending three hours a week in the lab. Even if you're madly working the whole time you can't expect to finish and the experiment on time.
- Repeating analyses. Students who find themselves frequently starting each analysis over due to irrecoverable difficulties must either face the possibility of not finishing the experiment on time or else prepare for and execute analyses more carefully the first time. The fact is that it's rarely necessary to "cheerfully start over" ("CSO"). There's usually some way to make use of prior work. Be sure that your decision to make a clean start is justified.
- Excessive fastidiousness. Care and cleanliness are essential to acceptable results in this course. However, not every weighing need be performed to ± 0.1 mg, and not every volume need be known within 0.01 mL. When good precision is required, the student will do well to use care in obtaining it.

When it is not required, doing so anyway generally constitutes a waste of time.

LABORATORY SAFETY

Each student is expected to conduct him- or herself in an intelligent and orderly manner at all times in the laboratory. Disregard for sensible safety measures constitutes grounds for dismissal from lab. In particular, the following points are to be observed:

- Students will perform only those experiments assigned or otherwise bearing the prior approval of the lab instructor.
- Eye protection which provides protection from the front and sides is to be worn in the laboratory at all times.
- Footwear which covers the feet is to be worn in the lab at all times. No bare or stocking feet, flip-flops or sandals.
- Keep long hair tied back and out of the way of chemicals and equipment.
- Beware of hot glassware. Do not touch it until it has had time to cool. Use tongs and/or high temperature gloves when removing glassware/samples from the drying oven.
- Eating, drinking, smoking and other operations involving contact with the face are prohibited in the lab at all times.
- The rubber bulb or other mechanical device provided is to be used at all times for drawing solution into pipets. No pipetting by mouth.
- No gummed labels of the sort requiring moistening are to be used in the lab. If you wish to mark glassware, a permanent marker after the fashion of Sanford's Impact or Sharpie will do nicely, provided that the surface is initially dry. The mark may be removed with acetone or other organic solvent.
- Each student is responsible for the cleanliness of his or her area, including the sink adjacent thereto. No solids are to be discarded into the sink. Use the trash container at the door for paper and soft plastic, and the special box provided for broken glass, hard plastic and other sharps.
- Hazardous waste must be disposed of in the appropriate hazardous waste container located in the fume hood in the back of the lab.
- Labcoats and nitrile gloves will be provided and are recommended for additional protection.