

CHEMISTRY 222

## QUANTITATIVE CHEMICAL ANALYSIS

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### RATIONALE

Analytical chemistry is that branch of chemistry which deals with the identification and assay of materials. Quantitative analysis, in particular, refers to the latter of these processes in that it responds primarily to the question "How much?" The ubiquitous nature of the analytical problem is apparent not only in the chemical industry and research, but also in such peripheral areas (well, that's how they look to analytical chemists anyway) as the health services, agriculture and food production, environmental protection, criminalistics, etc.

This course approaches quantitative analysis from a classical point of view. That is, techniques are discussed which have survived decades of critical use. While it is true that the bulk of today's analyses are performed with the help of sophisticated instrumentation, much of it automated, it is typically necessary to calibrate instruments, and essential to verify the correctness of the results obtained. It is these classical methods that are accepted as standard in such applications. For the situation where a non-routine analysis is needed the classical approach, which is typically straightforward and requires relatively simple equipment, is often a cost-effective solution. It may also come as a surprise (pleasant, we hope) to the student that, after only a few semesters of chemistry, he or she has the knowledge and much of the skill needed to perform a significant variety of interesting analyses on everyday materials, only a minute taste of which may be savored as a formal part of this course.

### OBJECTIVES

The goals of this course are several:

- To encourage the student to develop system and precision in laboratory technic.
- To review and expand upon the student's understanding of some of the fundamental properties of matter which are useful in quantitative analysis.
- To provide the student with an appreciation of chemistry as an exact science.
- To further refine the student's ability to intelligently apply a body of information to the solution of real or hypothetical problems.

## PREREQUISITES

The Chemistry 113–114 sequence is a formal prerequisite to Chem 222 and we will build on much of what you learned there without review. In addition, most chemistry majors or wannabes have taken Chemistry 220.2, Methods in Chemical Research, because it is recommended for those wishing to take this course. Other departments offer similar courses tailored to the needs of their disciplines which may address most of the same material. Here is a rundown of the non-General Chemistry stuff I expect you to know as a result of such courses. If you don't know some of it, you'll find adequate treatments in Harris, parenthesized below, that will get you up to speed. Just be aware that you'll have to take responsibility for acquiring this skill/knowledge yourself because we won't address it formally in this course. We'll just use it.

1. Know the difference between accuracy and precision, as well as determinate (systematic) and indeterminate (random) error (3-4).
2. Be able to express measurements and the results of calculations to the correct number of significant digits (3-1 – 3-3).
3. Be able to calculate:
  - The sample mean of a group of measurements (4-1)
  - The sample standard deviation of a group of measurements (4-1)
  - The sample standard deviation in the mean of a group of measurements (29-4)
  - The confidence interval within which a population mean lies given the desired confidence,  $\alpha$ , and the sample mean and sample standard deviation of a group of  $n$  measurements (4-2)
  - The slope and intercept of the linear least squares fit to a set of  $(x, y)$  data (5-1)
4. Be able to find the standard deviation in
  - a sum (or difference) given the standard deviations of its terms (3-5)
  - a product (or quotient) given the standard deviations of its factors (3-5)
  - an exponential given the standard deviation in its exponent (3-5)
  - The slope and intercept obtained from a linear least squares fit to a set of  $(x, y)$  data (5-2)
  - An interpolation into a linear least squares fit to a set of  $(x, y)$  data (5-2)
5. Be able to decide:
  - If the difference between a sample mean and a population mean (true value) is significant with 95% confidence (4-3)
  - If the difference between two sample means is significant with 95% confidence (4-3)
  - If a datum can be rejected (is not a part of a population) with 90% confidence (4-6)
6. Be able to use a spreadsheet program such as Microsoft Excel to calculate results and statistics for a set of replicate trials (2-10)

## COURSE TOPICS

1. **Solution Conventions and Equilibrium.** Formal, molar, and relative concentration, normal conventions, activity, computational techniques for competitive solution equilibria.
2. **Precipitation Equilibria.** the gravimetric method and precipitation titration.
3. **Acid-base Equilibria.** Estimation of titration curves, buffers, titration of mono- and polyfunctional acids and bases, acid-base indicators.
4. **Complexation Equilibria.** Computations involving polydentate and multiply-coordinate species, aminopolycarboxylic acids, conditional formation constant, complexometric titration.
5. **Redox Equilibria.** Electrochemical cells, the Nernst equation, mixed equilibria, formal potential, redox titration.
6. **Potentiometry** Direct potentiometry, reference electrodes, indicating electrodes, calibration curve, standard addition.
7. **Liquid-liquid Extraction.** Distribution between two liquid phases, extraction of metal complexes, designing a separation, multiple extractions.

## TEXT

The following text, ordered for this course, is available for sale in the College bookstore:

Harris, Daniel C.; *Quantitative Chemical Analysis*, Sixth edition (Freeman, 2003) ISBN 0-7167-4464-3

This book is very expensive. If you do not already own it, but have access to earlier editions they ought to do as well for most purposes. We will not begin to consider most of the material offered in the last half of this volume. Texts offering a more corresponding treatment cost only a few dollars less and exhibit serious deficiencies in areas of significance to us. Therefore, for those who must have exactly the book recommended, the indicated text has been chosen as the best value currently available considering its utility as a reference and its contemporary treatment, even though it seems ridiculously overpriced.

## CLASS WORK

Class time will be divided between group-centered activities and assigned readings from the textbook. Reading assignments will be accompanied by a few questions which the student will be expected to do outside of class. During a subsequent class period there will be an opportunity to address any items with which students experienced difficulty. The class activities will encourage the student to build the ability to solve chemistry problems with the help of small-group interaction.

Active participation on the part of each student is essential to the success of this approach to learning. The contribution of each student has value in the learning process, even though it may not necessarily express "the right answer". Students who withhold their participation are not only refusing to learn themselves, but are also hindering the learning of others. There is therefore a contribution to the overall course average for class participation which will be reduced if in the instructor's judgment a student clearly avoids participating on a regular basis.

## QUIZZES

From time to time a short impromptu quiz will be administered at the beginning of the class period. The purpose of these quizzes is primarily to encourage those students needing it to actually do the work assigned prior to class. They also potentially serve as an indication to the instructor regarding the degree of student understanding without invoking the major contribution to the course average incurred by an examination.

## HOMEWORK PROBLEMS

On a regular basis mathematically-oriented problems will be assigned which are to be completed outside of class. These are offered in this way in lieu of including them in examinations in order to give the student the benefit of having an opportunity to consider the solution over the course of several days rather than being expected to come up with it instantly during the exam hour. Each of these problems will be due one week following its distribution. Since these problems function in some ways as examination it is expected that they will represent the student's own best work. The following points are to be strictly observed:

- Students may use any textbook, notes, or other reference material they wish in the preparation of solutions to these problems.
- Students may talk about these problems if they wish with anybody else associated with this course.
- Students may not turn in a solution to any of these problems prepared in whole or in part by somebody else.
- Students may not turn in a solution to any of these problems that has been dictated in whole or in part by anybody else.

Students must offer in their solutions an organized, legible and sufficiently complete record of the progress from initial data to the final result that partial credit may be unambiguously awarded where appropriate ("show your work!").

## EXAMINATIONS

Because the major component of exams, the homework problems, is prepared outside of class, hour examinations will be limited to a few short-answer style questions mainly to test the student's understanding of the theory of the course. The student may consult the textbook, his or her own notes, or other references brought to the exam but may not communicate with other students or use materials brought by anybody else.

Examinations are scheduled to be given on the following dates:

First exam	Tuesday, February 7
Second exam	Thursday, March 2
Third exam	Thursday, April 6

## CLASS ATTENDANCE

A formal record of class attendance will not be kept, although the student might bear in mind that in a class of this size it's pretty clear if somebody's missing. Neither is there any direct contribution of attendance to the overall course average. It would be well to note, however, that it is impossible for a student who misses class to contribute to class activity.

Attendance at examinations is mandatory. If the student finds him- or herself, for reasons of illness or other significant inconvenience, unable to appear for an exam, he or she should notify the Dean of Student's office which will circulate a memo to the instructors involved attesting to these circumstances. Only upon receipt of this memo will a makeup exam be administered. Note that, since it is clearly unfair to the bulk of the class if a makeup is easier than its regular counterpart, and since it is impossible to prepare different examinations of exactly equal difficulty, makeup exams will appear slightly more rigorous than corresponding scheduled examinations. If the student knows in advance that he or she will be unable to appear for an exam as scheduled, it may be advantageous to arrange with the instructor to take it ahead of time.

There will be no makeups of missed quizzes. If a student's absence from class is approved in the manner indicated above, the missed quiz will simply not be counted in the average. Otherwise the score of zero will be assigned that quiz.

## LABORATORY

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. While some students find the lab work hectic and excessively demanding in terms of time, careful attention to detail and self discipline from the start will entirely eliminate these phenomena, as has been amply demonstrated by many students in the past.

The experiments listed in the schedule below will be performed. The timetable is not rigid, as some students will work more efficiently than others, or may have unusual difficulty with a particular procedure. It does, however, serve as a guideline by which the student may assess progress in order to finish by the end of the semester. Analyses which, according to this timetable, have been completed are also fair game for examinations.

Jan.	16	Determination of the Alkalinity of Soda Ash
	25	Determination of Chloride
Feb.	6	Spectrophotometric Determination of Manganese in Steel
	15	Identification of a Sodium Salt
	27	Identification of a Weak Acid
March	13	Determination of Calcium
	22	Determination of Iron in an Ore
April	3	Bromine Equivalent Weight of an Organic Compound
	12	Determination of Copper

Careful planning will be necessary to make the best use of time in the laboratory. This is especially true as the experiments are not strictly coordinated with the lecture. It will be normal to have more than one experiment in progress simultaneously. Remember that you are scheduled to spend a minimum of six full hours weekly in the lab doing manual operations. This does not include reading the lab handout, preparing writeups, waiting for things to cool off or dry, or keeping your neighbors company.

The student will provide an 8-1/2 by 11 inch or larger bound (not spiral) notebook with pages numbered in the upper outside corners (avoid the cheap ones after the fashion of "Neat-books". They start falling apart in a week or two). 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 white-out, or other attempts to obscure 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. Computer printouts, and other loose documents are to be taped securely onto blank pages in the notebook at all four corners. No entries are to be made underneath them.

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.

It is essential that the data, results, and conclusions corresponding to each exercise be organized in such a way that somebody unfamiliar with the work is able to follow it. The lab handout for the first analysis includes rather specific details regarding ways in which to accomplish this. Confusion on the part of the instructor nearly always results in a lower than deserved grade.

Notebooks will be collected for grading on the following dates:

Friday, January 27

Friday, February 24

Friday, April 28

The first of these collections will be for the express purpose of scoring the first lab exercise (Determination of the Alkalinity of Soda Ash). The final score will be assigned at that time and no further work on that exercise will receive any credit. The point of this early collection is to provide an opportunity for the instructor to respond intelligently to the quality of the student's work before it becomes too late to make appropriate modifications. Work on subsequent exercises will not be scored at this collection.

The second collection is a pacing point to encourage the student to remain current in the laboratory. The second, third and fourth exercises (through the Identification of a Sodium Salt) will be scored at that time. Work on subsequent exercises will not be scored at this collection.

The third collection takes place at the end of the term after the lab has closed. All remaining exercises beginning with the fifth (Identification of a Weak Acid) will be scored and the laboratory average computed.

It is so that no “right answers” on laboratory exercises will be shared until after it’s too late to do anything about them. There is little reason, however, for unforeseen disasters as the collection schedule permits timely identification of gross deficiencies. The careful student will also find that these exercises have been structured in such a way that checks on the reliability of the results obtained are available, just as in the real world laboratory, provided that the student chooses to use them. There need be little doubt when a reasonable result has actually been obtained.

The lab is scheduled to be available from 12:45 pm to 4:00 pm on Monday and Wednesday when classes are in session. In addition, if a staff person is available to oversee activity, the lab may be available at other times when it is not in use by other classes. The scheduled laboratory periods are the only times during which the lab will be actively serviced (that is, stock chemicals replenished, unknowns or standards dispensed, and direct assistance rendered with lab-related difficulties).

Two decades of experience have pointed up three conditions which contribute to difficulty in finishing the lab work satisfactorily by the end of the semester:

- Lack of discipline. It is essential that the student plan to spend at least six (6) hours in the lab each week, and that the work to be done be planned carefully beforehand.
- Repeating analyses. Students who find themselves frequently starting each exercise over due to irrecoverable difficulties must either plan to spend more than six hours weekly in the lab or else prepare for and execute analyses more carefully the first time.
- Excessive fastidiousness. Care and cleanliness are essential to acceptable results in this course. However, not every weighing must be performed to plus or minus 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.

As Spring approaches, the urge to be elsewhere than inside a windowless laboratory becomes overwhelming. Putting off work until later in the semester is a potentially fatal exercise in terms of performance in this course.

## 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. If you want to try something wild, ask the instructor. He's a chemist too and has had his share of fun over the years. The only concern is that what you do not represent an unreasonable hazard to yourself or others.
- Eye protection which provides protection from all directions is to be worn in the laboratory at all times.
- Footwear must be affixed sufficiently securely that the entire bottom of the foot is protected from the floor at all times. No bare or stocking feet or flip-flops.
- No one may work in the laboratory alone. When working outside the regularly scheduled laboratory hours, make certain that somebody within earshot is aware of your work.
- Eating, drinking, smoking and other operations involving contact with the face are prohibited in the lab at all times. If necessary, these activities must be pursued outside the lab. No lab apparatus is to be used in connection therewith.
- 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.

## GRADING

The following component weights will apply:

Class participation	10%
Quizzes	5%
Laboratory	40%
Homework problems	20%
Class exams	10%
Final exam	15%



The overall score will be computed by normalizing the average computed in each of the categories above to its respective weight and summing the weighted averages to give a maximum score of 100. Grades will be assigned according to the following:

100–92	A	81–80	B–	69–67	D+
91–90	A–	79–77	C+	66–62	D
89–87	B+	76–72	C	61–60	D–
86–82	B	71–70	C–	59–0	F

The laboratory will close at the end of the lab period on Wednesday, April 26. All lab work must be completed by that time and each student must be checked out. Failure to check out will result in a substantial penalty on the student's last non-zero lab grade. All submissions to be considered in computation of the final grade, with the single exception of the final examination, are to be turned in on or before Friday, April 28, 2006.