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 considered 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. Although Chemistry 220.2, Methods in Chemical Research, is also a prerequisite for this course, other departments offer similar courses tailored to the needs of their disciplines which may address much of the same material. Here is a rundown of the non-General Chemistry stuff the student is expected to know as a result of such a course. If you don't know some of it, you'll find adequate treatments in the eighth edition of 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. 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 of the mean of a group of measurements (4-1)
    - The slope and intercept of the linear least squares fit to a set of (x, y) data (4-7)
2. Be able to find the standard deviation in
    - a sum (or difference) given the standard deviations of its terms (3-4)
    - a product (or quotient) given the standard deviations of its factors (3-4)
    - An interpolation into a linear least squares fit to a set of (x, y) data (4-7)
3. Be able to determine:
    - 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)
4. 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.

TEXT

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

Harris, Daniel C.*; Quantitative Chemical Analysis*, Eighth edition (Freeman, 2007) ISBN 978-1-4292-5436-6

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 even 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 will be devoted exclusively to group-centered activities and addressing specific student questions. The activities will provide a framework within which the student may build the ability to solve analytical 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 participation on a regular basis.

READING

Regular readings from the textbook will be assigned which the student is expected to do outside of class. Each will be accompanied by a few questions which the student may use to test

his or her grasp of some of the important points of the material included in the reading. These questions will be addressed in class only in response to specific student requests.

EVALUATION INSTRUMENTS

Some of the materials used in this course are currently in development by the ANA-POGIL project, an NSF-funded program to develop and test a student-centered approach to the Analytical Chemistry course to which your instructor is a major contributor. From time to time the student may therefore be asked to provide feedback in a formal way to some aspect of the course. In order to provide motivation for the student to submit these instruments a small contribution to the grade will be offered for turning them in completed. With the exception of the final exam, the instructor will not be concerned with the content of these instruments (they are for assessment of materials and outcomes on a national basis) and in any case the content is not suitable for scoring in any way. Note that the final course evaluation which is required by the college and will be carefully analyzed by the instructor as well as other constituencies of the college following the submission of final grades, does not contribute to this credit.

HOMEWORK PROBLEMS

Many of the class activities include so-called Applications, the ANA-POGIL buzzword describing sometimes divergent problems that require the student to apply the material of the activity to a potentially realistic situation. Assigned Applications are to be addressed by the student outside of class and turned in for a score. Students are permitted to discuss the Applications with one another and may work on them in informal groups of two or more if desired. In the case of a group submission, each member of the group will receive the score assigned the submission.

Homework submissions will be due at the beginning of the class period following completion of the activity of which they are a part. The benefit to the student in addressing these Applications diminishes rapidly as the activity in question fades into antiquity so promptness in submission is essential. Late work will not be scored.

EXAMINATIONS

Three 70-minute in-class examinations will be held. These exams will be administered open-book. The student may consult the textbook, his or her own notes, or any other printed reference brought to the exam but may not communicate with anybody else in any way or use materials brought by others.

Examinations are scheduled to be given on the following dates:

First exam	Thursday, 17 February
Second exam	Thursday, 24 March
Third exam	Thursday, 21 April

The ACS Standardized examination will be administered as the final exam on Thursday, 5 May at 8:30 AM. Note that this exam is timed (100 minutes) and is to be completed closed-book (without the aid of text, notes or other reference).

CLASS ATTENDANCE

While a formal record of class attendance will not be kept for that express purpose, the fact is that it's impossible for a student who's not in class to contribute to that day's class activity. Students who miss class should expect to see a corresponding reflection in their participation scores.

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.

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.

The analyses 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.	17	Mohr Determination of Chloride
	26	Determination of the Alkalinity of Soda Ash
Feb.	9	Spectrophotometric Determination of Manganese in Steel
	21	Gravimetric Determination of Sulfate
March	14	Identification of a Weak Acid
	23	Determination of Iron in an Ore
April	4	Bromine Equivalent Weight of an Organic Compound
	13	Determination of Copper

Careful planning will be necessary to make the best use of time in the laboratory. This is especially true as the analyses are not strictly coordinated with the class. It is not unusual to have more than one analysis 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 lab notebook

Unlike most other chemistry courses, in Quant it isn't customary to prepare formal lab reports. Rather, the student is expected to submit for periodic examination a working lab notebook. I am still a proponent of handwritten entries in a permanent record as close as possible to the process of obtaining data. 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. The fact is, though, that some people just aren't neat and organized by nature and the notebooks they end up with aren't a lot of help to them while they're doing an analysis or to me once they think they've finished (or they need help with a problem that requires me to understand what they did). For that reason I'm going to try an experiment this year. You may choose one of the following two options for reporting your lab work:

Notebook Option 1. Traditional research notebook

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). 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 traditional 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.

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. 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.

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.

Notebook Option 2. Electronic submission

The student will record all data, perform all calculations and submit results in an electronic document which is e-mailed to the instructor on or before the date when the work is due for grading. Microsoft Excel is suggested as the application to be used for preparing this document. Students who prefer to use something other than Excel should talk with the instructor beforehand to be sure that the instructor will be able to read the resulting document since no hard copies will be exchanged (if what you really want is to turn in a hard copy lab notebook buy a bound notebook and do it right as per Option 1).

There are a couple of serious risks associated with the electronic approach. First of all, unless you plan to lug your laptop into the balance room with you or wherever else you might generate some data you're going to end up writing the data on scraps of paper towel and such or trying to remember it while you dash between rooms. Data gets lost or corrupted this way. This

is a major reason for the rules surrounding the use of the bound notebook in Option 1. Second, electronically recorded data is routinely lost to fate. If your laptop contains a mechanical hard disk, that disk will fail someday. It's not a matter of "if" but "when". When the disk drive fails your data goes with it. The failure of magnetic media is so certain that there's a technological solution for the problems that result. It's called "backing up your data". You need to have a flash drive or two, or another hard drive, or an optical media burner and you have to back up your data any time you have enough that you wouldn't want to have to go back and repeat the work. If you wouldn't mind repeating a week's worth of work, you only have to back up once a week.

Most students who have kept hard copy notebooks in the past haven't taken any special precautions to avoid losing them or to keep them secure from dogs. Yet everybody's painfully aware that "I lost it" or "the dog ate it" won't earn a lick of credit when it's not submitted for grading. Likewise, expect that electronic lab documents that aren't turned in on time due to computer problems will be regarded score-wise as not having been turned in.

It's convenient to edit electronic documents. This is one of the major benefits of using them. Consequently it is expected that entries in the document, whether raw data, calculated results, sample calculations, statements of conclusion or whatever will be presented neatly and in a logical order based on the order in which an experienced analyst (which you are once you finish an analysis) would do the work. This might not reflect the actual order in which you did things. In a hard-copy notebook it's occasionally necessary to point to out-of-sequence pages with additional trials or other entries that pertain to something earlier. Not so in an electronic document. Fix it so it looks as though you knew what you were doing the entire time, even if that's a bit of a stretch.

Additional lab considerations

Analyses are due for grading on the following dates:

Friday, January 28 Mohr Determination of Chloride
Friday, February 25 Alkalinity of Soda Ash, Manganese in Steel
Friday, April 29 All the rest

Hard-copy notebooks are to be turned in to my office or individual documents for each analysis e-mailed to my office e-mail address (Langhus@chem.moravian.edu) on or before the appropriate date. No hybrid submissions (hard-copy electronic documents, scans of handwritten entries) are permitted. Choose one format or the other on a per-analysis basis.

The point of turning in the Mohr Chloride by itself 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 analyses will not be scored at this time.

The second due date, a week prior to midterm, is a pacing point to encourage the student to remain current in the laboratory and also to provide a basis for midterm warnings, should any be in order. No work on subsequent analyses will be scored at this time.

The remaining analyses beginning with the fourth (Gravimetric Determination of Sulfate) will be due after the laboratory has closed for the term. The laboratory average will be computed at that time.

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 1:15 pm to 4:15 PM on Monday and Wednesday when classes are in session. The scheduled laboratory periods are the only times during which the student should count on the lab being actively serviced (that is, stock chemicals replenished, unknowns or standards dispensed, and direct assistance rendered with lab-related difficulties).

Historically students have been permitted to work in the lab outside of regular lab hours provided that a staff person is present and aware of the work. Note that the lab is shared with Biochemistry II Lab this term and it will be necessary at the very least to seek the permission of the instructor in charge if it’s desired to work on Friday afternoons. Note also that it’s customary in some labs for the instructor to share information by means of a lecture format and it’s very awkward to do this while people are wandering around the lab or otherwise going about their business. It would be best to simply avoid Friday afternoon altogether.

We’ve all heard the horror stories about the long nights in the Quant lab come the last week in April. Here are some conditions that 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 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 1:30 PM (“it’s the only time I have to eat lunch”) and leave at 3:00 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 without a backlog come the end of term.
- Repeating analyses. Students who find themselves frequently starting each analysis 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. The fact is that it’s rarely necessary to 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.

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, remember, 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 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.
- 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	5%
Evaluation Instruments	5%
Homework problems	10%
Laboratory	40%
Class exams	30%
Final exam	10%

The overall score will be computed by normalizing the average obtained 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 27. 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 29, 2011.

Students who wish to request accommodations in this class for a disability should contact the Assistant Director of Learning Services for Disability Support, 1307 Main Street (extension 1510). Accommodations cannot be provided until authorization is received from the office of Learning Services.