

CALIFORNIA STATE UNIVERSITY, LOS ANGELES
Chemistry 201
Course Outline
Winter Quarter 2007

Instructor: Dr. Feimeng Zhou
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Lecture: T&Th 11:45-1:00 BIOS-244

Office Hours: M,W&F 10-11:30 am.

Lab-02 T&Th 8:00-10:50 in P. S. 824

Lab-03: T&Th 1:10-4:00 pm in P. S. 824

Lab Instructors: Dr. Feimeng Zhou (Lab-03)
Dr. Xin Wen (Lab-02): Office: PS 620; Ph: 323-343-2310,
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Office Hour: Th: 1-2 pm, Fri: 8-9 am and other slots to be announced in lecture.

General Course Description:

Chemistry 201, Quantitative Analysis, is a sophomore level course which introduces the student to the principles and techniques of quantitative analysis. This course has a formal prerequisite of a full year of freshman chemistry, Chem 101-3 and it is assumed that the student has completed Math 102, College Algebra or equivalent courses at another institution. There are two 75 minute lectures and two 3 hour laboratory sessions each week. The objectives of the course are as follows:

- (1) to introduce the student both to the classical methods of analysis and the use of instrumentation in analysis
- (2) to have the student learn the elements of good laboratory practice
- (3) to have the student apply his/her knowledge of stoichiometry, acid-base theory, equilibrium (pH and redox), electrochemistry, and spectroscopy (topics covered in freshman chemistry) to analytical chemistry
- (4) to have the student learn to work independently as well as with others in the laboratory
- (5) to have the student learn to plan their work prior to coming to the lab and to be able to work on more than one experiment at a time in order to optimize the time spent in the laboratory
- (6) to have the student use spread sheets to analyze, plot, and present data
- (7) to have the student be able to follow a standard laboratory protocol and to adapt a protocol from the literature to this laboratory

A student should expect to spend a minimum of 3 hours of study time outside of class for each 50 minutes of lecture and 3 hours of preparation outside the class for each laboratory session (this totals a minimum of 15 hours per week outside the lab and lecture). Formal reports must be independently prepared by the student using a word processing program. The course is graded on the basis of 900 points which include lecture exams, problems, laboratory quizzes, laboratory unknowns, and laboratory reports. 420 points are associated with the lecture portion of the course and 480 points with the laboratory. A detailed description of the point assignment and evaluation (grading) is given later in this syllabus. The final exam is **Tuesday, March 13, 10:45 a. m. -1:15 p.m.**

Required Text: Daniel C. Harris, Quantitative Chemical Analysis, 7th edition
(W.H. Freeman & Co., New York, 2007)

Lab Manual: Handouts developed by Dr. Feimeng Zhou and Modified by Drs. G. Santillan and G. Hanrahan.

Additional Reading:

QD75,9 B73	Brewer, S., Solving Problems in Analytical Chemistry (Wiley, 1980)
QD42 S47	Sienko, M. J., Chemistry Problems (Benjamin)
QD101 V6	Arthur I. Vogel, A Text Book of Quantitative Inorganic Analysis (Wiley, Third Edition, 1961)

QD101 F53 Robert B. Fischer & Dennis G. Peters, Quantitative
 Chemical Analysis (Saunders)
 QD101,2057 C. D. Christian, Analytical Chemistry (Xerox)
 QD75 S55 Douglas A. Skoog and Donald H. West, Fundamentals of
 Analytical Chemistry (Holt, Rinehart and Winston)

Learning Aids:

Two video tapes are available for overnight check-out in the 7th Floor Chemical Stock Room (P. S. 750).

The first tape, entitled "Chemistry", has six sections and the Safety section will be shown on the first meeting of the laboratory.

1. To be viewed prior to starting any laboratory work:
 Safety 20 min.

2. To be viewed during first two weeks of course:
 Use of a Pipet 13.5 min.
 Weighing 22 min.
 Use of a Buret 17.5 min.
 Solution Preparation 17.3 min.
 Spectrophotometric Analysis 15 min.

I. Tentative Lecture Schedule:

Week	READING Dates	SUBJECT	CHAPTER	ADVICE
1	Jan 2	Introduction and Fundamental Concepts. Steps in a Chemical Analysis. Solution Preparations.	0,1,2	
2	Jan 9	Gravimetric Methods of Analysis: Principles, Techniques and Procedures Treatment of Analytical Data	27 3,4	
3	Jan 16	Colorimetric and Spectrophotometric Methods of Analysis	18,19,20,21	
4	Jan 23	Chemical Equilibrium	6,8,9	
	Feb 1	EXAM ONE		
5	Feb 6	Chemical Equilibrium, Titrimetric Methods of Analysis Acid-Base Equilibria Acid-Base Titrations in Aqueous Precipitate-Formation Titrations	7, 10,11	
6	Feb 13	Complex-Formation Titrations	12	
7	Feb 20	Fundamentals of Electrochemistry	14	
	Feb 22	EXAM TWO (Covers material through week 6)		
8	Mar 1	Potentiometry, Redox titrations	15, 16	
9	Mar 6	Electroanalytical Techniques	17	
10	March 13	FINAL EXAM 10:45 am -1:15 p.m.		

*For certain topics, this text goes into more detail than students are required to learn in the course. It is recommended that the student omits some sections and skim others as is listed above. "Skim" means that the student should read the section rapidly in order to obtain a general knowledge of its content and to be able to relate it to the topics in the rest of the chapter. While a few problems are assigned which the use of

formulas developed in these sections, a detailed knowledge of the content of the section will not be required for exams.

II. HOMEWORK

Homework problems are assigned at the beginning of each week, and are usually to be handed in the first laboratory period of the following week. Late homework earns at most half credit. Students are strongly advised to solve the problems themselves. All calculations should be detailed and presented in a neat and orderly manner and the homework papers STAPLED or CLIPPED together. Complete worked-out solutions to assigned problems will be posted next to the laboratory the following week except where indicated.

WEEK	DATE DUE	CHAPTER	PROBLEMS
1	Jan 9	1 2	17, 22, 31, 33,34 18,19
2	Jan 16	3 4 27	7(a,c,,d,f,g), 11, 12, 13 13,14, 15 16, 17, 18
3	Jan 23	19 21	3, 16,17 2, 17
4	Jan 30	6 8 9	6, 20, 40, 50 3, 12, 17, 20 4, 13, 33, 37
5, 6	Feb 13	7 11	7, 14, 20 8,12, 18, 31
7	Feb 20	12	6, 14, 15, 30
8	Feb 27	14	8, 10
9	Mar 6	15 16 17	2 3, 25 9, 28,29

III. QUIZZES

There will be a written quiz in the laboratory on the day that you are scheduled to start each unknown experiment, on the Tuesday of the week that you are scheduled to do the experiment. Quizzes cover the laboratory work assigned for the particular experiment to be completed.

IV. LABORATORY WORK

The first laboratory period is for check-in, the use of the single pan analytical balance, and preparation for Experiment 1. A student who does not attend the second (or a subsequent) lab meeting and who does not inform his/her instructor in advance of the lab, may be dropped from the course and another student checked into the locker if other students are waiting to get into the course.

The last period is for check-out. All unknown sample vials must be thoroughly cleaned and returned at this time. Take black markings off with acetone.

All lab work must be conducted in the time period for the lab section in which you have enrolled. Any deviations from this rule require prior written permission by the Analytical Coordinator.

The student should read and understand the assigned experiment before the laboratory period commences. Planning done outside the laboratory and before starting the experiment is essential in order to complete the experiments on schedule. A work outline with estimated times for each step is required before starting

experiments. Before getting your unknown from the instructor, you will be given an oral quiz on the information which should be in the outline.

Note: An * designates recommended laboratory procedure on this and pages following.

1. Gravimetric Procedures: One Experiment (9 hours)

Gravimetric Determination of nickel (Ni)
Textbook: Ch. 2, 27

2. Titrimetric Procedures: One Experiment (6 hours)

Complexometric determination of the water hardness by EDTA
titration using Eriochrome Black T (Ca).
Text: Ch. 12

*Obtain unknown from instructor by providing a clean, dry, stoppered 250 mL Erlenmeyer, marked in pencil with your name, locker number, and element (Ca).

Hint: Carry out trial titration on a test-tube scale in a qualitative manner to provide RAPID checks on reagents, dilutions, color changes to be expected, and procedure. Use double the recommended amount of buffer.

3. Radiant Energy Methods: Two Experiments (6 hours each)

(a) Spectrophotometric determination of manganese in steel and testing Beer's Law using four diluted concentrations from one of the known solutions (Mn). Do in duplicate, i.e., two standards and two unknowns. Note that if you use automatic electro-balances (e.g. Sartorius A 200S) you should place your sample vial on top of an inverted 50 mL beaker since the sample is ferromagnetic.

Text: Ch. 19,20

DO NOT DRY (Mn) SAMPLES IN THE OVEN.

Dilute your two knowns quantitatively to 500 mL as well as unknowns (unless the unknown is a very light pink color). Measure the absorbance at 4 wavelengths as suggested in the lab manual for only one of your knowns. For your values of absorbance used in the calculation of your two known and two unknown samples, take readings directly from the meter. If readings are not within the absorbance range of .35 to .70, quantitatively dilute the samples so that they are within the range and re measure. By serial, quantitative dilution of this sample 4 times (each time by a factor of 2), determine if Beer's Law is obeyed. Include a plot of your discrete scan and Beer's Law Absorbance with your informal report.

(b) Determination of the concentration (ppm) of sodium and potassium in a sample of water by flame photometry.

*Provide stoppered, labeled (NaK) flask to instructor for your unknown solution. (e.g., Same procedure as under 2a above.)

Text: Ch. 21

4. Acid/base titration and electroanalytical Methods: Two Experiments (6 hours each)

(a) Analysis of soda ash by pH titration. (pH)

Text: Ch. 9, 10, 11

Do two indicator titrations on each of the standardization and unknown, followed by one potentiometric (pH-glass electrodes) titration on the known standardization and one potentiometric titration on the unknown.

The color change of methyl orange may be from straw to a pink color rather than to pale orange - check this with a trial titration done qualitatively on a test-tube scale.

If the volume of your titrant does not equal about 25 mL, adjust the increments of addition proportionally as in the format on p. 99. Plot curves (i) pH vs. volume (v) of titrant, and (ii) pH/ v vs. average v. Determine the end point from the latter curve.

In reporting your results, the total HCl titer required to reach the second end-point should be used to calculate the soda ash content as % Na_2CO_3 .

NOTE: Vials containing solid unknowns will be available. Empty, clean and dry vials should be handed in at check out. For liquid unknowns, (2, 3(b)) you should provide the instructor with a clean, dry, stoppered 250 mL Erlenmeyer flask with your locker number and element to be determined on the label. (Use flask with volume marks; mark white label on flask with a pencil, not a pen). Unknown vials should be cleaned after the completion of each experiment. They are to be handed in clean and dry at check-out during the final lab period.

(b) Stripping analysis of lead in brass using a dropping mercury electrode

Text: Ch. 14, 17

(c) Redox titration

Text: Ch. 16

5. Special Project: (9 hours)

A project of the student's choice, based on his/her library research and within the constraints of the available laboratory equipment and chemicals should be carried out by each student. A short half-page of the procedure to be used in the project together with a list of equipment and chemicals required should be given to the laboratory instructor for approval by the end of the 6th week of the quarter. A more detailed description of topics appropriate for this project will be given after the first midterm.

V. Report of Experimental Results

Results of every unknown determination are to be handed in on the tear-out sheets as provided in the back of the Lab Manual, at the start of the first period following the scheduled completion of the experiments. The report should also include, as applicable, standard deviations and 90% confidence limits, graphs, and any other information requested by the instructor. All graphs should be done on millimeter graph paper with points and the curve done with a pencil and the title and axes in ink (graphs produced on PC's are fine if scaled properly).

FORMAL REPORTS are required for the following experiments:

1. Determination of Nickel in an ore.
2. Determination of Percent Iron in a Ferrous Ammonium Sulfate Sample.
3. Research project. (This report will not be returned to the student)

For a satisfactory format of a formal report, see P. K. Dea & H. Keyzer, "Practical Introductory Quantitative Analysis". Include an abstract at the start of the report; answer any question given in the lab text or this handout. Also, the report should indicate the manufacturer and model number of any instruments used. The first cover page of the report should include the dates the lab work was performed and the date that the report was submitted. Formal reports must be prepared with a word processor. Pertinent data must be tabulated and a sample calculation given. Graphs must be done on a computer. Students may use the instructional computing labs on campus by showing their student identification card. Students who have not used a computer before are advised to use Microsoft WORD on the Macintosh. An introductory document on WORD for the Mac is available from the laboratory instructor. EXCEL is useful for making graphs of titration data. Include spread sheets of your data as well as the plots. The model of the PC used and the word-processing software used should be indicated at the end of the report. For the research report, include a Xerox of the procedure where it is taken from the literature.

Formal reports are due one week from the scheduled completion of the experiment.

VI. SAFETY:

Safety must be a primary consideration for all persons entering a chemical laboratory. Experiments have been selected for their didactic as well as expected safety aspects in context of students learning new techniques. Students have the responsibility for learning and understanding appropriate safety features for each experiment. Further, each student has an obligation to consult the instructor when safety procedures are not clear. The following general procedures must be observed. See also the recommended Practical Introductory Quantitative Analysis text, pp. 1 and following and pp. 23 and following.

1. Eye protection must be worn at all times while working, or while others are working in the laboratory. Appropriate eye protection includes approved safety spectacles (Z87) or goggles. Normal prescription glasses are not acceptable. Contact lenses do not constitute proper safety spectacles.
2. Smoking and eating are not permitted at any time in the laboratory. Open-toed shoes are not permitted in the laboratory. A lab coat or plastic apron is recommended as are rubber or plastic gloves such as surgical gloves. Even the detergents used in the lab sometimes irritate sensitive skin.
3. Work is not permitted in the laboratory except during regular periods when an instructor is present. Performance of unauthorized experiments is not allowed.
4. Before beginning the first experiment, determine in the laboratory the location of the fire extinguishers, safety shower, eye wash, safety solutions, and the emergency telephone. Consult your instructor about the proper use of these items.
5. Waste chemicals must not be poured down the drain without proper treatment or neutralization. Consult your instructor before excess chemicals are disposed of.
6. Read the information on laboratory safety in your texts. Always record any safety precautions in your laboratory notebook.

7. Please note that microwave ovens are used in this course for drying some samples. If you wear a pacemaker, please inform your instructor before checking into the laboratory, use the convection ovens for drying, and arrange to work away from microwave ovens. Under no circumstance should you dry either organics (they char, burn, or explode) or metal samples (they destroy glassware and the support plate of the microwave oven). This means that you should not dry your unknown or standard for manganese in steel nor the platinum electrodes used in the determination of copper. Do not heat water in these ovens since it could ruin another student's sample being dried to constant weight.

8. Report any accident, even the most minor, to your laboratory instructor. In case of a chemical splash, flush the area thoroughly with water. Use eye wash for at least 15 minutes for chemicals in the eyes. Fires can usually be extinguished by smothering. Use fire extinguisher with care.

VII. LABORATORY NOTEBOOK AND REPORTS

Laboratory Notebook:

1. The notebook should be stiff-covered and permanently bound with consecutively numbered pages. (National, #53-110 with Quadrule paper is available in the bookstore and is satisfactory if you number the pages yourself). Spiral notebooks are not acceptable.

2. The first two pages of the notebook should be reserved for a Table of Contents which must be kept up-to-date.

3. The title of the experiment should be clearly written and underlined and the date of the experiment recorded. The objective of the experiment (in one or two sentences at most) should follow along with balanced chemical equations and mathematical expressions appropriate to the experiment and a reference to the instructions and procedure.

4. It is required that you prepare a brief outline of the operations involved in the laboratory and it is advisable that you include the time you estimate that they will take. This outline of operations should directly follow the objective and equations and before the entry of data. It is advisable to prepare part of the notebook for recording data prior to coming to the laboratory. These preparations will help you make the most efficient use of your limited laboratory time.

5. Entries on each page should be legible, consecutive, well-spaced from one another, dated and preferably in a tabulated form. Note that if two entries on one page are entered on different dates, each entry should carry its date. If an entry extends beyond one page, each page that its entered on should be dated.

6. All data should be entered in ink or ball-point pen directly into the notebook, and not on loose pieces of paper. No pencil marks or erasable ink may be used in the notebook. It is required that the laboratory book be brought to the lab every period. The penalty for not having the lab book in the laboratory is 10 points.

7. Erroneous entries should not be erased or obliterated. No whitener should be used to cover up any part of the notebook. If an error is made in entering anything in the notebook, it should be crossed out with a single horizontal line. Enter the correct form, located as neatly as possible. Numbers should never be written over. For example, suppose we have entered a wrong datum 23.3256 g instead of 25.2356 g. Cross out 25.3256 as shown below and enter

$$\text{mass of container} = \quad 25.3256 \text{ g} \quad 25.2356 \text{ g}$$

At no time may you tear pages out of your notebook. If you wish to use a notebook in a previous course, re-label the cover and tape or staple shut the earlier work.

8. Do all your calculations clearly and neatly in your notebook.

9. The label on the outside of the book must have your name (printed), Chem 201, Lab Section #, time of meeting, Locker #, your Instructor's name and the Quarter taken.

Result Report Forms (Informal Reports)

Results of every unknown determination are to be handed in on the tear-out sheets as provided in the back of the Lab Manual, at the start of the first period following the scheduled completion of the experiments. The report should also include, as applicable, standard deviations and 90% confidence limits, and any other information and graphs as requested by the instructor. All graphs should be done using EXCEL or a standard graphing package. Be sure to title the graphs appropriately.

1. Your name, locker number, experiment number, date reported and designation, Ni, etc.
2. Class schedule number of the lab section.
3. Calculated value for each determination.
4. Average of determinations. Explain any exclusion of results.
5. Standard deviation and 90% confidence limits if appropriate.
6. Other information or graphs as requested by the instructor.

On the rest of the sheet include your data for each determination and a sample calculation with both symbol and numerical set up for each different step. These informal reports should be handed in before the formal report is completed so that you can learn your unknown grades as soon as possible.

VIII. GRADES AND EXAMINATIONS

Traditional grades (A, B, C, D, & F) will be used in this course with plus or minus grades given. Grades will be assigned primarily on the basis of point accumulated as follows:

First lecture examination	100 points
Second lecture examination	100 points
Final examination	150 points
Homework problems	70 points
Experiment quizzes	80 points
Laboratory work (Unknown 7 x 35) (Formal report 2 x 25) (Research project 50) (Laboratory notebook 55)	400 points
Total	<u>900 points</u>

In grading laboratory work, the write-up, precision, accuracy and analysis of the results and errors will be taken into consideration, together with the techniques in performing the experiment. Points are deducted for late reports (1 point during the first week late and 2 points for each additional week late). In the event that an error in calculations is made on the informal report, it may be handed in again for re-grading. Include the original informal report with the new values next to the old values which have been crossed out with a single line. The new score minus two points is the resulting grade (note, it could be lower than the first grade although usually there is an improvement). Also include on a separate sheet an explanation as to the error in calculation. A repeat calculation may only be done once unless the instructor suggests a second try. The final deadline for all reports to be handed in is the last meeting of the laboratory (Check-out). At that time the lab book should be handed in for a second grading and must include all informal reports and all graphs.

The number of points accumulated by a student during the quarter determines his /her relative standing in the class. Final assignment of grades is, of course, a qualitative judgment and will be made on the basis of the instructor's judgment of the performance of individuals and of the class as a whole, as well as on the basis of the class distribution of points. A passing grade (D) for the course requires that all laboratory work be completed, and that the student earns a passing grade in both the lecture and the laboratory.

In general, make-up lecture examinations are not given. Students who miss an exam, due to illness for example, will be given a weighted average of their remaining exams, provided that a written substantiation of the illness from a doctor is presented. The weighting will include both the number of points and the class average of the exams.

IX. DROPPING THE COURSE AND INCOMPLETES

Hopefully, a student will not be dropping this course; however, in the event that this is necessary the student must meet all university rules and deadlines. In addition, the student must check out of the laboratory at the time he drops the course and during the normal lab period. Lockers not properly checked out result in the student being charged a \$10 fee in addition to broken or missing equipment. In the case of an incomplete, the student must be making a (C) in the course, have completed all assigned laboratory experiments and have written substantiation from a doctor if for example, the reason for the incomplete is illness or an accident. An INCOMPLETE GRADE REPORT form must be completed and given to the instructor by the end of the examination week. If these conditions cannot be met, please make contact with the lecture instructor.

Chem 201 Lab list of reminders:

HOUSE KEEPING: Always. . .

- 1 LABEL your glassware (write your "locker #"): Use pencil, avoid tape.

- 2 PLACE stirring rods in beakers in hot plate
 - 3 ACID- and base-containing containers must be properly labeled
 - 4 CLEAN up after yourself-in the balance room and hoods.
 - 5 DON'T transfer chemicals inside balance chamber. (chemical spillage=-5 points!)
- SAFETY PROCEDURES. . .
- 6 FUME-evolving containers should not be removed from the hood
 - 7 FAN in the hood should be turned on. If not turn it on.
 - 8 POUR concentrated acids/bases into a beaker, then transfer into a graduated cylinder
 - 9 EXTRA care is needed when acid and water are mixed - much heat is evolved!
- LAB PERFORMANCE
- 10 Read the syllabus & lab book very carefully before class.
 - 11 DON'T waste acids, bases or other chemicals- use only what you need. Ask if anyone else needs your excess.
 - 12 END all lab work 10 min. before the end of the lab. Clean up takes time.
 - 13 UNKNOWNs lost? need additional unknown? automatic penalty = -5!
 - 14 SPILL someone else's unknown? penalty =-5 pts (either one of you or both get penalized)
 - 15 NO CROSSING over to the afternoon lab session is permitted
 - 16 QUESTIONS about the experiment?? Think before asking. Formulate questions clearly for clear answers.
- LAB NOTEBOOKS, LAB REPORTS & FORMAL REPORTS
- 17 DATA- all raw lab data must be written directly with pen (not pencil) onto your notebook (or -5 penalty)
 - 18 RESULTS are due the session after the lab experiment is scheduled to be finished.(-2 pts if late)
 - 19 NOTEBOOKS may be inspected at anytime (follow syllabus strictly). (no notebook? -10 points)
- EXPERIMENTS -- ADDITIONAL POINTERS. .
- NICKEL EXPERIMENT
- 20 HNO₃ --> use only about 75 mL, for washing crucibles
 - 21 DRYING samples and crucibles may take as long as 2 hours!!
 - 22 pH METERS (portable ones!) are located at the sides of the hoods (for adjusting to pH 8-9). TURN THESE OFF AFTER USE with switch on top of meter.
 - 23 "NH₃" solution is same as bottle of "NH₄OH"
 - 24 CLEAN crucibles with about 8-10 mL of NH₃ (crucibles half full)
 - 25 CLEAN crucibles with 8-10 mL of bulk ethanol. Let stand 5 min, then suction 5 min. before microwave.
- OTHER EXPERIMENTS
- 26 Fe & Mn NO DRYING - we repeat! - No drying of Fe or Mn samples!
 - 27 Ca & Na/K: supply instructor with a dried and stoppered 250 mL Erlenmeyer flask. (Label flask with: locker#, "Ca" or "Na/K" as may be appropriate)
 - 28 pH: make 500 mL of 0.1 M HCL
 - 29 Special Project: Rule of thumb: cut down all chemical reagent preparations 100-fold. No Ag, no organic flammables, expts requiring unavailable equipment, or body fluids and tissue that may transmit serious diseases (check if reagents are available before submitting a proposal).
 30. The abstract is a brief description of the experiment and should include the average or best value and the standard deviation or confidence limit of the analyte.