Quantitative Analysis—CHEM 201
Fall 2009

Instructors

Lecture:

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T,Th—1:30 – 2:30 p.m.

Laboratory:

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Office hours: M,W,F—11:00 a.m. – 12:15 p.m.
Th—4:00 – 5:00 p.m.

Lecture T,Th: 11:40 a.m. – 1:00 p.m., SHC 162

Laboratory Section 02: M,W—1:10 – 4:00 p.m., P.S. 824
Section 03: T,Th—8:00 – 10:50 a.m., P.S. 824
Section 04: T,Th—1:10 – 4:00 p.m., P.S. 824
Section 05: T,Th—6:10 – 9:00 p.m., P.S. 824

Course Description

CHEM 201 is a sophomore level course which introduces students to principles and techniques in proper quantitative chemical analysis. The formal prerequisite is a full year of General Chemistry (CHEM 101-103 or equivalent from another university with a grade of C or better). It is assumed that students have a solid grasp of college level math including algebra.

Course Objectives

The main objective of CHEM 201 is to develop students' understanding of the principles of analytical chemistry. More detailed objectives include:

- To learn both classical and modern instrumental aspects of analytical chemistry
- To learn the analytical process as applied to basic laboratory research
- To become familiar and later master the elements of good laboratory practice
- To ultimately apply his/her knowledge of analytical chemistry in an independent manner

Required Textbook

Quantitative Chemical Analysis, 7th Edition, D.C. Harris, W.H. Freeman and Company, New York, 2007, http://bcs.whfreeman.com/qca/

Lab Handouts

Detailed handouts of each of seven laboratories and safety requirements will be provided by the instructor.

Learning Video

"Chemistry" has six sections—the Safety section and Pipetting section will be shown during the first laboratory meeting. Demonstration of the use of an analytical balance will also be given.

Lectures will be presented using PowerPoint format. A pdf version of each chapter's lectures will be posted following completion of the chapter on the **Class Notes** page of the Chemistry Department's website: www.calstatela.edu/dept/chem

Tentative Lecture Schedule

Week	Dates	Subject	Chapters
1	Sep 24, 29	Analytical Processes/Basic Review of Concepts; Solutions and their Concentrations; Stoichiometry	0, 1, 2
2	Oct 1, 6	Experimental Error and Calibration	3, 4
3	Oct 8, 13	Gravimetric Methods of Analysis	6, 27
4	Oct 15, 20	Volumetric/Titrimetric Analysis	7, 8
5	Oct 22 Oct 27	**Mid-term Exam #1** Spectrophotometry, Calibration Methods	 5, 18 – 21
6	Oct 29, Nov 3	Acid-base Equilibria & Titrations	9 – 11
7	Nov 5, 10	Complex Formation and Redox Titrations	12, 16
8	Nov 12 Nov 17	**Mid-term Exam #2** Electrochemistry & Potentiometry	 14, 15
9	Nov 19, 24	Analytical Separations; Gas Chromatography	23, 24
10	Dec 1, 3	Gas Chromatography; HPLC	25
	Dec 8	**Final Exam, 10:45 a.m.**	

Attendance all lecture classes is expected. Unannounced quizzes and group work are to be anticipated by all students. Absent students are responsible for all discussion and lectures they missed.

No makeup exams will be given for the Mid-term exams. At the discretion of the instructor in cases of documented family emergencies and illness (with doctors excuse) only, a student may be excused. Under no circumstances will a student be excused from taking the test due to lack of preparation

Homework

Homework problems will be assigned at the beginning of each week and will be handed in at the **first laboratory period the following week** unless otherwise stated. Homework should be neat and state the Homework # and the problems assigned at the top of the first page. Students should work these problems independently with all calculations clearly visible in a step by step manner on **stapled** paper. Complete worked-out solutions to assigned problems will be posted next to the laboratory the following week except where indicated. *Late homework:* 2 points will be deducted for homework sets handed in one lab period late, thereafter 1 point will be deducted for each additional lab period.

HW Set	Due date	Chapter	Problems
1	Oct 5/6	1 (p. 18) 2 (p. 37)	17, 22, 31, 33, 34 18, 19
2	Oct 12/13	3 (p. 51) 4 (p. 75)	7(a,c,d,f,g), 11, 12, 13 13, 14, 15
3	Oct 19/20	6 (p. 117) 27 (p. 641)	6, 20, 40, 50 16, 17, 18
4	Oct 26/27	7 (p. 137) 8 (p. 155)	7, 14, 20 3, 12, 17, 20
5	Nov 2/3	18 (p. 399) 19 (p. 418) 5 (p. 92) 21 (p. 472)	6, 10, 12, 16 3, 16, 17 2, 23, 29 5, 14, 19, 20

6	Nov 9/10	9 (p. 178) 10 (p. 196) 11 (p. 223)	4, 13, 33, 37 1, 5, 13, 28 17, 21, 47
7	Nov 16/17	12 (p. 246) 16 (p. 344)	6, 14, 15, 30 2, 5, 14, 18
8	Nov 23/24	14 (p. 294) 15 (p. 323)	16, 21, 25 3
9	Nov 30/ Dec 1	23 (p. 525) 24 (p. 553) 25 (p. 585)	9, 11, 13, 18 20 9

Laboratory work
Items required for laboratory include: a scientific calculator, approved safety goggles (must have Z87 shatterproof lenses), and a bound notebook (quad-ruled composition book, bookstore). Students must inform their instructors in advance if a laboratory will be missed. Failure to do so may result in the student being dropped from the class. All laboratory work for this class must be performed in the quantitative analysis lab, not individual research labs. No switching laboratory sections unless approved by both instructors. If you must receive a new unknown sample, 5 points will be deducted from your lab score. All unknown laboratory work will be graded based on overall precision and accuracy. If you fail to bring safety goggles to lab, 5 points will be deducted for each incident.

Quizzes
A written quiz will be given in the laboratory on the day that you are scheduled to start each unknown experiment. Quizzes will cover laboratory work assigned for the particular experiment to be completed. Typically, the quizzes test your knowledge of the chemistry involved as well as your ability to do quantitative analysis based on data given.

Flow Charts
A work outline (flow chart) with estimated times for each step in each experiment is required at the beginning of each lab and must be written in the left side of notebook before each new experiment (see notebook format below). Before getting an unknown sample, students will be given an oral quiz on the information which should be in the outline.

Laboratory Notebook Format (no spiral notebooks will be accepted)
Record all experimental work in a stiff-covered, permanently bound notebook (National, # 53-110) and use the right hand pages of the notebook for the experimental records. Flow chart must be placed on left hand pages. The first two pages of the notebook are to be saved for Table of Contents. Pages must be numbered. All data must be written directly into the notebook with a date on every data page. No pencils, "white-out", or erasable ink may be used. If an error is made, simply place a single horizontal line through mistake and enter correction. Be sure to write down important procedures in lab book before each lab. No photocopies of manual or procedures in lab book. Always have your notebook in class for spot inspection or grading. **Note: Up to 5 points will be deducted for each violation of the above policies!**

Each experiment record and report must include the following:

1. Name, section #, date, quarter
2. Experiment title, and objective(s)
3. Principles: a concise description of the chemical principles (equations included).
4. Data and important observations (IN INK)
5. Calculation methods: at least sample calculations of all calculations must be included.
6. Results and discussion (graphs included)

Note: Points will be deducted for any missing information!

Note: Any balanced equations or mathematical expressions are to be put in the "Principles" section. The penalty for not having a lab book is **10** points. Outside of book must be labeled with name (printed), Chem 201, Lab section #, time of meeting, locker #, instructors name and quarter taken. Lab books will be graded twice during the quarter (unannounced).

Informal Reports

Results of every unknown are to be handed in on the tear-out sheet in back of your experimental handouts at the beginning of the first period following the scheduled completion of experiments. 2 points will be deducted for late reports on the first lab period after the due date and 1 point will be deducted for each additional lab period. Reports must include averages, standard deviations and relevant graphs (Excel or other graphing programs). Informal reports should be handed in before the formal report to access unknown grades. Be sure to include all data for each determination along with calculations and experimental set-up and label all graphs appropriately.

Formal Reports (a detailed description of sections of the report is given below)

Three (3) formal, typed-written reports will be handed in during the course (experiments included will be announced by lab instructor). These reports must follow the format below. These reports will be due the lab period following submission of the informal report for that same experiment. As with homework, 2 points will be deducted for late reports on the first lab period after the due date and 1 point for each additional lab period. Reports must be a concise description of the experiment with all appropriate data and graphs. **Formal reports will be required for the calcium (Ca) and acid-base (pH) titration experiments as well as the research project.**

The format includes (in order):

1. Cover page with title of experiment, name, date, locker # and lab section
2. Abstract
3. Introduction
4. Experimental procedures
5. Results, including data and relevant graphs
6. Conclusion
7. References
8. Acknowledgments

Research project

Students have a choice of research projects based on the instructor's overall choice of topics. However, projects must be within the constraints of the laboratory equipment and chemicals in the laboratory. Students should give the instructor a short procedure with list of equipment and chemicals for approval (by the end of the 6^{th} week). Topics deemed appropriate will be discussed in lecture and a handout will be provided during the 6^{th} week.

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.

l. 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 or 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.

Laboratory etiquette

It is essential that you consider others in the lab. Be careful with acids and hot reagents and clean up any spills that may occur. Use of the analytical balances: do not use weighing paper and <u>never transfer samples in the analytical balance</u> (points will be deducted). Keep your work areas clean and neat. Cleaning assignments will be given by instructor each lab period. You are expected to stay focused on your experiment. Cell phone activity should be kept at a minimum and outside the lab.

Collaborative Experiment:

For both of the copper experiments (AA and ASV) you will be paired with another student. During the first experiment, one student will be the manager and one the assistant. The manager will oversee the assistant's work and will report on the experimental success of this student in his/her own report and laboratory notebook. On the next experiment, the assistant will become the manger and do the same. This collaborative exercise will help students in gaining valuable laboratory management practices. For once, you will be the instructor!

Point Distributions for Grades

Lecture:	
Mid-term exams (2 @ 100 points each)	200 points
Final exam	150 points
Total	**350 points**
Laboratory:	
Unknown samples (7 @ 35 points each)	245 points
Formal report (2 @ 25 points each)	50 points
Research project	50 points
Lab book (25 pts 1st grading, 30 pts 2nd grading)	55 points
Quizzes	80 points
Homework	70 points
Total	**550 points**
Total points for course	**900 points**

Grades will be assigned as traditional (A, B, C, D, & F) and based on points accumulated. However, class attendance, distribution of points and individual performance will be taken into consideration on final grades. To pass this course, students must complete all laboratory work. Students will receive an (F) if laboratory work is not completed (unless a valid, documented excuse is provided).

Dropping the course/incompletes

Hopefully, a student will not drop 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/she drops the course and during the normal lab period. Lockers not properly checked out will result in the student being charged a $10 fee in addition to broken or missing equipment.

An incomplete grade (I) will only be given to a student earning a "C" in the course with a valid, documented reason why the course cannot be completed during the term (an illness or serious accident). An INCOMPLETE GRADE REPORT form must be completed and given to the instructor by the end of the final examination week.

Final Comments

This is a challenging course and requires a good deal of time for laboratory preparation and lecture problem solving. Keep up with all lectures and homework and follow all laboratory experiments closely (flow chart will help you). If you have questions, ask. Finally, relax and enjoy the course.

Formal Report Sections – Detailed Description

Abstract

A summary of the technique and contains key results. No background material should be included here.

Introduction

Here one states the aim, the historical and theoretical context of the work. Include any relevant chemical equations or reactions. Should be concise, but have enough information for a complete introduction. It is important to describe the chemistry (chemical equations) as well as the mathematical equations and formulas that will be used. Showing molecular structures is recommended whenever appropriate.

Experimental Procedures

This section is used for describing experimental procedures, conditions, apparatus and reagents used in the experiment. Do not use outline form. Paraphrase the instructions in the manual in your own words.

Data

The raw data obtained in the experimental section is presented here.

Analysis and Results

The data listed in the data section is analyzed by calculations or graphs in this section. Final results are tabulated for easy perusal. Show all calculations carried out including mean and standard deviation.

Conclusion

This section contains a critical evaluation of the data gathered and the errors inherent in them. Be specific and thorough in your narrative.

References

Throughout the body of the manuscript, statements used which derive from external sources require defense based on previous work. These statements must be denoted numerically either with a superscript or in parenthesis in the text, and then the source listed in the Reference section by that number.

Example:

1. J. Peabody and H. Originales, "The Behavior of Copper Ions in Aqueous Solution." Journal of Analytical Results, **21**, 345-350.

Acknowledgments

All assistance, funds and gifts should be acknowledged as a matter of courtesy. For example, financial support and scholarships, laboratory assistance, etc…should be stated.

Laboratory and Unknown Procedure Guidelines

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.

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.

l. Gravimetric Procedures: One Experiment (9 hours)
   Gravimetric Determination of nickel (Ni)—*Harris* Ch. 2, 27
2. Titrimetric Procedures: One Experiment (6 hours)
   Complexometric determination of the water hardness by EDTA titration using Eriochrome Black T (Ca)—*Harris* 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—*Harris* 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.)—*Harris* Ch. 21

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

(a) Analysis of soda ash by pH titration. (pH)—*Harris* 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) $\Delta pH/\Delta v$ 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—*Harris* Ch. 14, 17

(c) Redox titration—*Harris* 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.

Chem 201 Laboratory list of reminders

House Keeping—Always do the following:

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, and 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 HNO_3—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 "NH_3" solution is same as bottle of "NH_4OH"
24 CLEAN crucibles with about 8-10 mL of NH_3 (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, no experiments 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.