

## Quantitative Analysis—CHEM 201 Winter 2013

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**Instructors**

Lecture &amp; Laboratory:

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Office hours: T, Th - 10:00 – 11:30 a.m.  
F—1:00 – 2:00 p.m.

Laboratory:

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Dr. Greg Santillan, LKH 253  
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**Lecture** T,Th: 11:40 a.m. – 1:00 p.m., SH C240

**Laboratory** Section 02: T,Th—8:00 – 10:50 a.m., ASCB 150  
Section 03: T,Th—1:30 – 4:20 p.m., ASCB 150  
Section 04: T,Th—6:10 – 9:00 p.m., ASCB 150  
Section 05: M,W—1:30 – 4:20 p.m., ASCB 151

**Required Textbook**

Quantitative Chemical Analysis, 8<sup>th</sup> Edition (7<sup>th</sup> Edition is also fine), D.C. Harris, W.H. Freeman and Company, New York, 2007

**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

**Lab Handouts**

Detailed handouts of each of the seven laboratories and safety requirements will be available on the Chemistry Department's website (<http://www.calstatela.edu/dept/chem>) under the Class Notes tab.

**Lecture presentation style**

Most lectures will be presented using PowerPoint format. PowerPoint lectures will be posted on the Chemistry Department's class notes website only after a chapter has been completed. Lecture notes will not be posted prior to beginning a chapter or section.

**Tentative Lecture Schedule**

| Week | Dates                | Subject                                                                                          | 7 <sup>th</sup> Edition Chapters | 8 <sup>th</sup> Edition Chapters |
|------|----------------------|--------------------------------------------------------------------------------------------------|----------------------------------|----------------------------------|
| 1    | Jan 8, 10            | Analytical Processes/Basic Review of Concepts; Solutions and their Concentrations; Stoichiometry | 0, 1, 2                          | 0, 1, 2                          |
| 2    | Jan 15, 17           | Experimental Error and Calibration Methods                                                       | 3, 4, 5                          | 3, 4, 5                          |
| 3    | Jan 22, 24           | Equilibrium, Gravimetric Methods of Analysis                                                     | 6, 27                            | 6, 26                            |
| 4    | Jan 29<br>Jan 31     | Volumetric/Titrimetric Analysis<br><b>Mid-term Exam #1</b>                                       | 7, 8                             | 7                                |
| 5    | Feb 5, 7             | Spectrophotometry                                                                                | 18 – 21                          | 17 – 20                          |
| 6    | Feb 12, 14           | Acid-base Equilibria & Titrations                                                                | 9 – 11                           | 8 – 10                           |
| 7    | Feb 19, 21           | Complex Formation and Redox Titrations                                                           | 12, 16                           | 11, 15                           |
| 8    | Feb 26<br>Feb 28     | <b>Mid-term Exam #2</b><br>Electrochemistry & Potentiometry                                      | 14, 15                           | 13, 14                           |
| 9    | Mar 5, 7             | Analytical Separations; Gas Chromatography                                                       | 23, 24                           | 22, 23                           |
| 10   | Mar 12, 14<br>Mar 19 | Gas Chromatography; HPLC<br><b>Final Exam, 10:45 a.m.</b>                                        | 24, 25                           | 23, 24                           |

**Lecture Attendance**

Attendance all lecture classes is expected. Absent students are responsible for all discussion and lecture material 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 on the dates specified below. Homework should be neat and state the Homework Set # and the problems assigned at the top of the first page. Multiple pages must be stapled together so pages are not lost by the instructors. Students should work these problems independently with all calculations clearly visible in a step by step manner. Complete worked-out solutions to assigned problems will be posted next to the laboratory the following week except where indicated.

*Late homework:* Homework turned in late will be deducted 2 points for each lab period following the due date.

| HW Set | Due date     | 7 <sup>th</sup> Edition |                          | 8 <sup>th</sup> Edition |                          |
|--------|--------------|-------------------------|--------------------------|-------------------------|--------------------------|
|        |              | Chapter                 | Problems                 | Chapter                 | Problems                 |
| 1      | Jan<br>16/17 | 1                       | 17, 22, 31, 33, 34       | 1                       | 11, 22, 31, 33, 34       |
|        |              | 2                       | 18, 19                   | 2                       | 17, 19                   |
| 2      | Jan<br>23/24 | 3                       | 7(a,c,d,f,g), 11, 12, 13 | 3                       | 7(a,c,d,f,g), 11, 12, 13 |
|        |              | 4                       | 13, 14, 15               | 4                       | 13, 14, 15               |
|        |              | 5                       | 2, 23, 29                | 5                       | 2, 24, 30                |
| 3      | Jan<br>30/31 | 6                       | 6, 20, 40, 50            | 6                       | 6, 20, 39, 49            |
|        |              | 27                      | 16, 17, 18, 22           | 26                      | 16, 17, 18, 23           |
| 4      | Feb<br>6/7   | 7                       | 14, 20                   | 26                      | 45                       |
|        |              | 8                       | 3, 12, 17, 20            | 17                      | 21                       |
|        |              |                         |                          | 7                       | 3, 12, 18, 21            |
| 5      | Feb<br>13/14 | 18                      | 6, 10, 12, 16            | 17                      | 6, 11, 13, 16            |
|        |              | 19                      | 3, 16, 17                | 18                      | 3, 17, 19                |
|        |              | 21                      | 5, 14, 19, 20            | 20                      | 5, 16, 22, 23            |
| 6      | Feb<br>20/21 | 9                       | 4, 13, 33, 37            | 8                       | 4, 13, 33, 38            |
|        |              | 10                      | 1, 5, 13, 28             | 9                       | 1, 5, 15, 30             |
|        |              | 11                      | 17, 21, 47               | 10                      | 17, 21, 47               |
| 7      | Feb<br>27/28 | 12                      | 6, 14, 15, 30            | 11                      | 7, 15, 16, 31            |
|        |              | 16                      | 2, 5, 14, 18             | 15                      | 2, 5, 14, 18             |
| 8      | Mar<br>6/7   | 14                      | 16, 21, 25               | 13                      | 16, 21, 25               |
|        |              | 15                      | 3                        | 14                      | 3                        |
| 9      | Mar<br>13/14 | 23                      | 9, 11, 13, 18            | 22                      | 9, 11, 13, 19            |
|        |              | 24                      | 20                       | 23                      | 21                       |
|        |              | 25                      | 9                        | 24                      | 10                       |

### 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.

### Laboratory 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.

**Laboratory Experiments**

- Hardness of water determined by EDTA
- Determination of manganese in steel
- Determination of iron in a ferrous ammonium sulfate sample
- Gravimetric determination of nickel
- Determination of copper in an aqueous sample by atomic absorption spectroscopy
- Determination of soda ash
- Ion-selective determination of fluoride
- Research project of student's choice

**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 two of the assigned experiments as well as your chosen 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<sup>th</sup> week). Topics deemed appropriate will be discussed in lecture and a handout will be provided during the 6<sup>th</sup> week.

### **Laboratory 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. 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 never transfer samples in the analytical balance (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.

### Point Distributions for Grades

Lecture:

|                                      |                   |
|--------------------------------------|-------------------|
| Mid-term exams (2 @ 100 points each) | 200 points        |
| Final exam                           | 150 points        |
| Homework                             | 90 points         |
| <b>Total</b>                         | <b>440 points</b> |

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 1 <sup>st</sup> grading, 30 pts 2 <sup>nd</sup> grading) | 55 points         |
| Lab quizzes                                                               | 70 points         |
| <b>Total</b>                                                              | <b>470 points</b> |

**Total points for course** **910 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.

**Chem 201 Laboratory list of reminders*****House Keeping***

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 never be removed from the hood
7. FAN in the hood should be turned on—if the fan is not on when you go to the fume hood, turn it on
8. POUR concentrated acids/bases into a beaker, and 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 minutes before the end of the lab—clean up takes time
13. IF you need to obtain additional unknown sample, it will cost you 5 points on your unknown report
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) into your notebook (there will be a 5 point penalty if you use pencil or transfer data from a paper into your lab book)
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)

**Chem 201—Quantitative Analysis**  
**Schedule of Laboratory Experiments**

|          | Week    |           |           |           |           |           |           |                  |
|----------|---------|-----------|-----------|-----------|-----------|-----------|-----------|------------------|
| Locker # | 1       | 2         | 3         | 4         | 5         | 6         | 7         | 8                |
| 1        | Calcium | Manganese | Iron      | Fluoride  | Nickel    | Copper    | pH        | Research Project |
| 2        | Calcium | Iron      | Fluoride  | Nickel    | Copper    | pH        | Manganese | Research Project |
| 3        | Calcium | Fluoride  | Nickel    | Copper    | pH        | Manganese | Iron      | Research Project |
| 4        | Calcium | Nickel    | Copper    | pH        | Manganese | Iron      | Fluoride  | Research Project |
| 5        | Calcium | Copper    | pH        | Manganese | Iron      | Fluoride  | Nickel    | Research Project |
| 6        | Calcium | pH        | Manganese | Iron      | Fluoride  | Nickel    | Copper    | Research Project |
| 7        | Calcium | Manganese | Iron      | Fluoride  | Nickel    | Copper    | pH        | Research Project |
| 8        | Calcium | Iron      | Fluoride  | Nickel    | Copper    | pH        | Manganese | Research Project |
| 9        | Calcium | Fluoride  | Nickel    | Copper    | pH        | Manganese | Iron      | Research Project |
| 10       | Calcium | Nickel    | Copper    | pH        | Manganese | Iron      | Fluoride  | Research Project |
| 11       | Calcium | Copper    | pH        | Manganese | Iron      | Fluoride  | Nickel    | Research Project |
| 12       | Calcium | pH        | Manganese | Iron      | Fluoride  | Nickel    | Copper    | Research Project |
| 13       | Calcium | Manganese | Iron      | Fluoride  | Nickel    | Copper    | pH        | Research Project |
| 14       | Calcium | Iron      | Fluoride  | Nickel    | Copper    | pH        | Manganese | Research Project |
| 15       | Calcium | Fluoride  | Nickel    | Copper    | pH        | Manganese | Iron      | Research Project |
| 16       | Calcium | Nickel    | Copper    | pH        | Manganese | Iron      | Fluoride  | Research Project |
| 17       | Calcium | Copper    | pH        | Manganese | Iron      | Fluoride  | Nickel    | Research Project |
| 18       | Calcium | pH        | Manganese | Iron      | Fluoride  | Nickel    | Copper    | Research Project |
| 19       | Calcium | Manganese | Iron      | Fluoride  | Nickel    | Copper    | pH        | Research Project |

**Important Note:** Laboratory check-out will occur the last scheduled lab period of the quarter. There will be no experimental procedures performed during the last scheduled lab period of the quarter.