

Chemistry 214, Quantitative Analysis Laboratory

Spring 2014 Syllabus

Chem 214-002, Quantitative Analysis Laboratory (1 credit hour), Tuesdays/Thursdays 2:45-5:30pm, FH-313
Prerequisite: Chem 106/102 and 112; Chem 222/224 and 226 as well as active attendance or completion of lecture Chem 212.

Instructor: Katrina Binaku

Office: Flanner Hall 104

Phone: (773) 508-8715

Email: kbinaku@luc.edu

Office Hours: Mondays 11am-12pm, Fridays 1-2pm,
and by appointment.

Graduate Teaching Assistant (TA): Jackie Arroyo

Office: Flanner Hall 407

Phone: (773) 508-3137

Email: jarroyo1@luc.edu

Office Hours: Mondays 12-1pm, Fridays 9-11am,
and by appointment.

Course Objectives:

- 1) To acquaint students with some of the classical and modern techniques in analytical chemistry
- 2) To teach wet chemical lab skills, efficiency and planning of experiments
- 3) To teach critical evaluation of experimental results
- 4) To become familiar with conventional data collection in commercial and academic laboratories.

Attendance Policy: It is a requirement to be present for every scheduled lab session. Additional time will not be provided to students who are absent from lab. Students are allowed to attend only the section in which they are enrolled. Students must have required materials and be properly dressed to perform experiments in the lab. Make-up exams and quizzes will not be given unless approved by the Teaching Assistant and/or Instructor.

Footwear/Clothing: Closed toed, closed heel shoes are required [no slippers, Crocs, perforated shoes, etc.] No skin on the foot can be exposed (ballet flats are not allowed unless socks are worn). Long pants are recommended. Shorts and skirts are not allowed, as bare skin on the lower extremities is a safety hazard: Be advised, concentrated acids/bases will be used in most of the experiments. Lab coats are required and must be worn at all times during lab experiments. Students will be sent home if proper clothing/footwear are not worn; this counts as an absence. A safety lecture will be given the 1st day of class; it is required to perform lab experiments. Students will sign a safety sheet acknowledging their understanding of lab safety rules/policies.

Required Materials:

- One bound (NO SPIRAL) *laboratory notebook* such as a national-brand Composition book.
- An inexpensive *calculator* having logarithm (base 10 and e), exponential, and trig functions.
- A pair of *lab goggles* [safety glasses NOT allowed] which must be worn at all times in the laboratory.
- *Lab coat*. Offers a layer of protection against hazards. It must be worn at all times during lab experiments. Any color is fine, must be long sleeve. Can be found on Amazon or the bookstore.
- Laboratory procedures and handouts (posted in Sakai)
- Non-erasable pen

Bring all materials to every lab session. For some experiments, it may be advantageous to bring a laptop computer for data entry, analysis, and calculations. If it is deemed to be a distraction or hazard, the TA or Instructor may request that it be put away.

Note: Cell phones are not a substitute for a calculator and will NOT be allowed for use during quizzes, the midterm, or final exam.

Laboratory Procedures:

Instructor and TA will explain the procedures and goals for each assignment prior to its execution. Experiment handouts will be available on Sakai. Students are responsible for/expected to print and bring experiment procedure handouts to lab. The TA will have several extra procedure copies but will not actively print a quantity for all students. A laboratory schedule for the semester, detailing the projected start dates for each experiment, pre-lab quizzes, lab reports, and other information will be provided on the first day of class. This schedule will be posted on Sakai and in the laboratory, FH-313.

Experiment Unknowns:

In most cases, you will be assigned a standard unknown sample whose composition is known to at least **FOUR** significant figures. **You will determine the concentration of your unknown sample and be graded on how accurately your determinations reflect the true composition.** Make sure to write down the unknown # in your notebook, in addition to signing for it on unknown sheets provided by the TA.

For each assignment, you will report the values of your individual determinations, the mean concentration (or percent composition) and the standard deviation associated with the overall determination. You will be permitted to repeat each lab only once as time permits in order to earn a better accuracy grade. However, you will need to analyze a new unknown sample and it must be undertaken in the period established on the laboratory schedule. In order to accomplish this, **you will need to report your results and calculations on SAKAI as soon as completed.** You will then be graded on the accuracy of the results and can thus decide if you want to repeat the experiment. Graded accuracy will determine about 60% of your overall grade.

Laboratory Notebook:

One bound notebook is required. Notebooks must be completed in PEN. Detailed notebook requirements are listed later in the syllabus. Briefly, the notebook must contain all data, observations assembled during each experiment. It should be organized but not necessarily perfect and thus can contain strikeouts.

Students must come to lab prepared in order to optimize their lab efficiency. At the start of every NEW experiment each student must have written in their notebook:

- 1) The title of the experiment and the date
- 2) A paragraph introduction to summarize the lab experiment's purpose, background information, and an overview that may include a very brief procedure synopsis to aid in getting started. The first sentence of the introduction should include the purpose of the lab experiment.

*Instructor and/or TA will review and sign off on this portion of the lab notebook (while a pre-lab quiz is being taken) and will prevent a student from starting a lab if the notebook is not satisfactorily completed. The student will not be allowed to start the experiment until the notebook is filled out with these requirements.

Laboratory Reports:

Lab reports are to be computer generated and must follow the format defined later in the syllabus. All data sets must be included in the final report. The lab report will thus contain the data from the first attempt and second if repeated (redo). Final accuracy will be determined as the better of the two reported findings. Graded lab reports will determine 22.5% of your overall grade (see breakdown below).

Lab report due dates are located on the semester schedule. Lab reports **will not** be accepted via email unless otherwise specified. Reports must be printed and handed in at the beginning of lab on the due date. **Lab reports turned in late will receive a penalty of 10% each day the report is late and result in a grade of 0 if not received within one week of the established due date.** In order to help students better address deficiencies in content within the initial lab report; the first lab report (only) may be resubmitted after grading to receive at most ½ the lost points. Please be sure to discuss any questions, concerns about lab report format or grading with the TA or Instructor.

Over the course of the semester a total of 8 experiments will be performed. You are required to complete all 8 experiments and will turn in results for accuracy for each. Writing skills are important to express and explain test results and other important information in the “real world,” and we realize that completing lab reports is labor intensive. Therefore, you will only write lab reports for four (4) of the eight (8) labs in this course.

The following list* includes the experiments for which a completed lab report is required:

- 1) Acid-Base Titration: Determination of % Carbonate in an Unknown (Lab 2)
- 2) Spectrophotometric Determination of Iron (Lab 3)
- 3) Assay of SO_3 by Gravimetric Analysis of Sulfate (Lab 4)
- 4) Weak acid titrations: ID and Quantification of Polyprotic Acids (Lab 8)

*At the discretion of the Instructor or TA, this list can be modified at any time over the course of the semester.

Laboratory Exams:

Two written exams will be given which cover concepts pertaining to all of the laboratory experiments. The Midterm exam will include **Experiments 1-4** and the Final exam will include **Experiments 5-8**. Exams will cover the theory as well as related calculations.

Laboratory Quizzes (Pre-lab Quizzes):

Before the start of each new experiment a written pre-lab quiz will be given asking questions in regard to the procedure and calculations to determine preparedness for the lab. **Quizzes will be given during the first 15 minutes of lab. Thus, one MUST be punctual in getting to lab on time! If one arrives late to lab, no extra time will be given to complete the quiz.** Quiz answers must be written in pen or credit will not be given.

Journal Article Assignment

Referencing scientific journal articles is an important aspect of research. Each student will be responsible for selecting, reading, and writing a report on an article published in either *Analytical Chemistry* or the *Journal of Analytical Chemistry*. This assignment will aid students in recognizing principles of analytical chemistry and the importance of method development and application. Detailed information will be handed out at a later in the semester. The due date for this assignment is on the laboratory schedule.

Academic Honesty

Instructor and TA encourages students to consult one another during lab experiments and outside of class. Students can converse, brainstorm, and work through questions together but copying other students' work and presenting it as one's own is unacceptable. There is a difference between sharing knowledge and cheating.

If it is determined that lab reports or other materials in this course are plagiarized or have been shared between students (current or past), no credit will be given for the assignment. Cases of suspect academic dishonesty will be handled according to University policy and guidelines. Please review Loyola University Chicago's policy on Academic Integrity via this link: http://www.luc.edu/academics/catalog/undergrad/reg_academicintegrity.shtml

Grading Policy

The established grading policy is subject to change at Instructor and/or TA discretion. Please note the University uses a +/- grading scale system and it will be implemented in this course.

Grading Category	Pts	Percent
Analytical Findings (Accuracy)*	1400	60.9%
Detailed Laboratory Reports	400	17.4%
Lab Quizzes	90	4.0%
Lab Notebook	100	4.3%
Journal Article Report	110	4.8%
Midterm exam	100	4.3%
Final exam	100	4.3%
Total	2300	100.0%

Grade Assignment:

Points Range	
2070 - 2300	A
1839 - 2069	B
1608 - 1838	C
1377 - 1607	D
Below 1377	F

*6 labs @ 200pts/each, 2 labs @ 100pts each for accuracy

Lab Report and Notebook Grading Rubrics

The following is a generous guide provided by the Instructor and TA with a rough estimate of the grading breakdown of lab reports and notebooks. Points can be redistributed between all categories within lab reports or notebooks at the discretion of the Instructor and TA.

Lab Report	Points
Title Page	4
Purpose	6
Materials	10
Procedure	15
Results	35
Conclusion	20
Grammar/Formatting/Spelling	10
TOTAL	100

Notebook*	Points
Table of Contents	8
Title of experiment (1pt/experiment)	8
Introduction (signed**, 2pts/experiment)	16
Results/ Raw Data and Calculations (5pts/experiment)	50
Conclusion (2pts/experiment)	16
Organization (sections labeled, writing legible)	2
Total	100

*Point breakdown based on 8 experiments completed, this may change based on the progress of the course.

**Unsigned Introduction sections will receive no credit

SEE THE FOLLOWING PAGES FOR THE LAB REPORT FORMAT & LAB NOTEBOOK REQUIREMENTS

Lab Report Format and General Guidelines

CHEM 214: Quantitative Analysis Laboratory

Lab reports for Quantitative Analysis should be more detailed and complete than reports in the past for General or Organic Chemistry. This is an upper division lab class, and more thoroughness is expected of the student. The report write-up is a **VERY IMPORTANT** part of a laboratory based course, especially at the junior/senior undergraduate level and of course for graduate level work.

Lab reports consist of the following elements:

Title page – lab experiment name and number centered on the page; your name, lab partners name (for partner labs only), unknown #, and date the report is due should be placed in the lower right hand corner of the title page

Purpose – brief statement of the reason for performing this experiment

Materials – two separate lists: one for a listing of all equipment (including the balance used) and the second list for chemicals used (including any unknowns, but excluding any solutions the student makes)

- Concentrations of solutions used should be included. Include concentrations of stock solutions, not concentrations of solutions made. Concentrations of solutions made should be given under the results section.

Procedure – this is a list of all the steps that you did to perform the lab, including any changes that you may have made to the original printed procedure.

- This can be summarized from that listed in the lab handouts but must be rewritten in your own words! **Do not plagiarize**
- It must be so clear that anyone not familiar with the lab would know exactly what to do.
- It should not contain the actual masses, volumes, etc. used by the student.
- Be careful how you write your prep instructions for solutions. You will dissolve/dilute chemicals in a volume smaller than what the final volume will be and then dilute to the final volume mark. For example: Dissolve 12 g KOH in 300 mL DI water, dilute up to 500 mL mark, and shake to mix well.

Results – list data obtained, such as volumes measured, weights, temperatures, in a table format

- Multiple trials must always be done to verify data as having good precision. All data must be shown, including repeat lab data. Teaching assistants will grade lab reports based on precision.
- Data must be represented in table format with appropriate column and row headings and include the individually determined values, averages (for concentrations, percents, unknowns, etc.), standard deviations, and other necessary values. When applicable include units in column headings i.e. “NaOH volume (mL)” or “mL of NaOH.” Tables must be labeled with appropriate brief titles describing the contents within a table.
- Statistical analysis of your data should also be put in this section.

- If applicable, graphs should go in this section, and they must be clearly labeled with a title and proper x axis and y axis names as well as units. Graphs should be done in Excel.
- Include calculations in this section labeled appropriately with units, chemical identity and properly identifying what is being calculated and the trial # the calculation is being completed for.
- Include general equations corresponding to each calculation necessary i.e. general equation for **dilutions** (see example below), average, standard deviation, ppt, to name a few.
(ex.: Calculation for volume of HCl for 0.1M HCl). The calculations may be written, but please write them neatly so they can be read and understood.
 - Show an outline of equation being used and at least one example with your numbers
 - ex.: $m_1v_1=m_2v_2$ $12M \times (v_1)=0.1M \times (1000mL)$ $v_1=8.33 \text{ mL}$
 - Please utilize leading zeros before the decimal point (0.1mL and not .1mL).
- A paragraph statement of the results must also be present to interpret/summarize the data shown in tables and graphs.
- If graphs/figures are included, such as spectra or chromatograms, they should be accompanied with a proper label i.e. Figure 1, and brief description directly below it.

Conclusion – a restatement of your results, and what the results mean

- Include a **detailed** analysis of error (at least 3 errors). This should be done based on the students own data and results. An analysis of error can also be done on theoretical errors as well though the student may not have made these errors.
 - How does the error change the outcome (concentration higher/lower than it should be, etc.)? How does the error affect the subsequent steps in the experiment?
- How can the experiment be improved and/or made simpler?
- How can the student's technique be improved?

Additional Considerations

- Order is also important for excellent scientific work – the lab report write-up must follow the order listed on these directions.
- Lab reports should have page numbers specifically located in the bottom center of each page. Please staple your lab reports before handing them in.
- Feel free to print double-sided; we are a sustainable university after all!
- All parts of this report must be typed (calculations are an exception). Please use 12 point font, 1.5 lines spacing for paragraphs, and 1 inch margins.
- Please keep entire tables on a single page. If you must split up a table, remember to include column and row headings again on the next page.
- Reports should have good spelling, sentence structure, etc. Do not use run-on sentences, fragments, or personal pronouns (I, we, me, etc.).
- Take the time to check over your work and re-read your report to make sure that what you wrote is clear and makes sense.

The following has been said:

“A student could do mediocre work and write up an excellent lab report, and the work will be thought of as wonderful. A student could do wonderful work and write it up poorly, and the work will be thought of as mediocre.”

Lab Notebook Guidelines and Grading Rubric

NOTEBOOK MUST BE COMPLETED IN PEN

Leave the first 2 pages of the notebook blank. At the top of these two pages please write TABLE OF CONTENTS. Over the course of the lab, label the pages in the lab notebook. Then in the table of contents simply write the name of each experiment. Next to the name, write the page # that the experiment starts on. It doesn't need to be more detailed than that.

On every day of lab work, the date should be written in the notebook at the beginning of class. This will allow you to keep track of what was completed on a particular date, including solutions prepared, experimental work and calculations.

Each of the sections of the notebook should be labeled as such using roman numerals and the section headings as displayed below.

At the start of each new experiment the following is required in the notebook at the beginning of lab (i.e. completed before coming to lab):

- I. Title of experiment, date
- II. Introduction: A paragraph synopsis/overview of what the point of the experiment is, methods (titration, precipitation, etc.) or instrumentation (if applicable) utilized in the experiment. From this short paragraph, someone reading your notebook will have a basic idea of what the experiment entails. The FIRST SENTENCE of the introduction should state the purpose/what will be discovered in the particular experiment. This paragraph can be roughly $\frac{1}{2}$ a page but no more than 1 page long.

Note: The instructor or TA will initial these sections of each experiment. It is the student's responsibility to get their notebook signed as these required initials will count towards the point value of the notebook. If this section is not initialed, 1pt deduction per missing signature.

- III. Procedure (optional)
If students find it helpful to write out the entire experiment's procedure in their own words in detail, they can do so in their notebook in this optional section. It is not a requirement as students will have the printed experimental procedure to reference while completing each experiment.
- IV. Results
First, the unknown number should be clearly written at the beginning of this section. This section, as described earlier in the syllabus, should contain only calculations for solutions you physically prepare in class, and all observations and pertinent data that is generated during the experiment. This includes but is not limited to color changes (initial solution color and endpoint color in a titration for example), initial/final buret readings for all experimental trials, balance weights for solid samples, balance #,

instrument settings, etc. Values written down should have units and chemical identity accompanying them i.e. 15.05mL of NaOH. All data should be written in pen. Strikeouts are acceptable as no notebook is perfect. If alterations or changes in an experimental procedure occur, this is the section to include that information as well.

V. Conclusion

Brief. Restate the purpose of the experiment and what was accomplished (one or two sentences that state your unknown number and what you found). If any major errors occurred in the experiment i.e. you accidentally dumped out one of your samples, etc. state that here as well.

Example: The purpose of this experiment was to determine the percent sodium carbonate in an unknown sample. In unknown #12, it was determined that the unknown sample contained 39.57% sodium carbonate.

*Format Check: You can request a format check after lab #1 by your TA/Instructor.

Chem 214-002, Quantitative Analysis Lab, Spring 2014 Tentative Semester Schedule

Chem 214 Quantitative Analysis Schedule* (Spring 2014)					
Assignment Dates	Week #	Class #	Date	Proposed Experiment	Proposed Tasks**
	1	1	Tuesday, January 14, 2014	Syllabus, Safety Lecture, Equipment Check-in	Review syllabus, course guidelines. Safety lecture. Lab equipment.
Lab 1 Quiz		2	Thursday, January 16, 2014	1) Determination of % KHP in an Unknown	Prepare and Standardize NaOH solution
	2	3	Tuesday, January 21, 2014		Titrate unknowns, complete calculations, submit to Sakai
Lab 2 Quiz		4	Thursday, January 23, 2014	2) Determination of % Carbonate in an Unknown	Check NaOH molarity from experiment 1; can use for lab 2. OR Redo lab 1
	3	5	Tuesday, January 28, 2014		Standardize HCl and titrate unknowns
		6	Thursday, January 30, 2014		Finish titrations; Begin redos for labs #1 & 2; clean flasks for lab 3
Lab 3 Quiz, Lab 2 Report Due	4	7	Tuesday, February 04, 2014	3) Spectrophotometric Determination of Iron	Must complete in 1 session; Continue redos for labs 1 & 2
		8	Thursday, February 06, 2014		Redos for lab 3; begin to heat/weigh crucibles for lab 4
Lab 4 Quiz	5	9	Tuesday, February 11, 2014	4) Assay of SO ₃ by Gravimetric Analysis of Sulfate	Prep unknown samples & digest, possibly filter. Heat/weigh crucibles
		10	Thursday, February 13, 2014		Filter digested samples, heat/weigh crucibles with product.
Lab 3 Report Due	6	11	Tuesday, February 18, 2014		Heat/weigh crucibles with product.
		12	Thursday, February 20, 2014		Clean out crucibles. Begin lab 3 & 4 Redos
	7	13	Tuesday, February 25, 2014	Possibly start 5) GC-MS Quantification of Halomethanes	Submit assay results; study for midterm exam. Clean glassware.
LAB MID-TERM		14	Thursday, February 27, 2014	Lab Midterm Exam / Notebook Check #1	Bring Calculator and Notebook! Today is the deadline to have assay results for labs 1-4 submitted via SAKAI
	8	15	Tuesday, March 04, 2014	Spring Break; NO CLASSES	No Class
		16	Thursday, March 06, 2014	Spring Break; NO CLASSES	No Class
Lab 5 Quiz, Lab 4 Report Due	9	17	Tuesday, March 11, 2014	5) GC-MS Quantification of Halomethanes	Water extractions; process GC-MS data
Lab 6 Quiz		18	Thursday, March 13, 2014	6) Vitamin C Redox Titration	Complete in 1 session. Prepare, standardize iodine soln; titrate unknown
	10	19	Tuesday, March 18, 2014		Process GC-MS data; Redo lab 6
Lab 7 Quiz		20	Thursday, March 20, 2014	7) EDTA Lab; Titration and IC Quantification of Ca and Mg	Prepare EDTA and Calcium Carbonate solutions; begin standardization
	11	21	Tuesday, March 25, 2014		Complete solution standardizations; titrate unknowns
		22	Thursday, March 27, 2014	Metrohm 883 IC analysis of unknowns	Titrate unknowns; prepare IC dilution of unknown (10x)
Journal Article Assignment Due	12	23	Tuesday, April 01, 2014	Metrohm 883 IC analysis of unknowns	Analyze unknowns on IC; record results in notebook
		24	Thursday, April 03, 2014		Redos for lab 7; remake unknown IC dilution
Lab 8 Quiz	13	25	Tuesday, April 08, 2014	8) Polyprotic Acids	Prepare NaOH and standardize OR Finish lab 7 Redos
		26	Thursday, April 10, 2014		Prepare NaOH and standardize
	14	27	Tuesday, April 15, 2014		pH titration of unknown polyprotic acid. Bring laptops if possible!
		28	Thursday, April 17, 2014	Easter Break begins Thurs Evening; No Class today	No class
	15	29	Tuesday, April 22, 2014		Redos for lab 8; study for final exam
LAB FINAL and Check-out		30	Thursday, April 24, 2014	Lab Final Exam / Notebook Check #2	Bring Calculator! Final Notebook Check & Equipment Checkout. ALL ASSAYS for labs 5-8 should be in SAKAI BY TODAY!
Lab 8 Report Due via EMAIL			Friday, April 25, 2014		Lab 8 Report Due via EMAIL by 9pm to Jackie. Will not be accepted late under any circumstances.

* This schedule is subject to change at the discretion of the Instructors or TA at any point during the semester

UNLESS OTHERWISE NOTED, LAB REPORTS ARE TO BE PRINTED OUT AND HANDED IN AT THE BEGINNING OF LAB ON THE DUE DATE. They will be considered late if not printed.

** Please be advised that these proposed tasks should be used as a guide and are under no circumstances the only tasks that can be performed. This is the bare minimum.

*This schedule is subject to alterations/changes at the discretion of the Instructor or TA at any point during the semester.

Lab reports are to be printed out, double-sided (let's save some trees), stapled, and turned in at the beginning of lab on the due dates. They will be considered late otherwise. Lab reports cannot be emailed unless otherwise noted.

**Please be advised that the proposed tasks column should be used as a guide and are under no circumstances the only tasks that can be completed. These are suggestions and a bare minimum of work.