

Chemistry 212 Syllabus
Analytical Chemistry Laboratory

Spring 2006

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Date (week of) Experiment (page numbers are from the C212 Lab Manual)

Jan. 16	Introduction; check-in; lecture on safety and use of equipment; set-up and begin Expts. 1 or 3
Jan. 23	Group 1. Expt. 1: Calibration of Glassware, p. 25 (in C212 Lab Manual) also see pp. 48-50, textbook. (Report is due week of Feb. 6 by 5 pm on your lab day) Group 2. Expt. 3: Gravimetric determination of soluble chloride, p. 31. Also see pp. 314-328, textbook. (Report is due week of Feb. 6 by 5 pm on your lab day)
Jan. 30	Group 1. Expt. 3: Gravimetric determination of soluble chloride, p. 31. Also see pp. 314-328, textbook. (Report is due week of Feb. 6 by 5 pm on your lab day) Group 2. Expt. 1: Calibration of Glassware, p. 25 (in C212 Lab Manual) also see pp. 48-50, textbook. (Report is due week of Feb. 6 by 5 pm on your lab day)
Feb. 6	9: Spectrometric determination of iron, p. 49.
Feb. 13	Finish Experiments 1, 3 & 9. (Boil water for Expt. # 4)
Feb. 20	4: Titrimetric determination of potassium acid phthalate. p. 33.
Feb. 27	5: Potentiometric titration and numerical analysis of titration curve, p. 36
Mar. 6	6: Complexometric titration of calcium in impure calcium carbonate (water hardness), p. 40
Mar. 13	(No new experiment; a day to catch up on all work so far.)
Mar. 20	10: Ion-selective electrodes: Determination of fluoride, p. 53.
Mar. 27	>>> SPRING BREAK <<<<
Apr. 3	First set of Rotation Experiments. Your experiment is whatever you are signed up for.**
Apr. 10	Second rotation
Apr. 17	Third rotation
Apr. 24	Fourth rotation
May 1	Complete all experiments; Check out

** For the rotation experiments only, students will work as partners because of equipment limitations. All other experiments will be performed individually. The rotation experiments will be chosen by the instructor from among several possibilities. These will be announced before spring break.

Course Expectations

Safety and Cleanliness: The first priority of the laboratory is safety, of the student and of the instrumentation. 15 points (1 point per lab session) will be assigned based on the instructor's perception of your attitude and behavior in regard to safety, cleanliness, and the proper use of instrumentation. "Demerits" are points that will be deducted from your 15 possible points, and may be given for a violation of any of the following rules. Usually, you will be told at the time the demerits are given, so it will not be a surprise at the end of the term.

1. Laboratory safety requires that you wear protective eyewear at all times. The only exception to this is during pre-lab lecture.
2. Closed-toe shoes must be worn; sandals or open-weave footwear are not appropriate. Do not wear high heels.
3. Hair that is shoulder length or longer must be tied back away from the face.
4. No food, drink or tobacco is allowed at any time in the laboratory.
5. Clean up your own spills promptly, safely and correctly. If you are unsure how to clean up a spilled material ask the instructor. Usually water is the best diluent, but there may be exceptions.
6. Use instruments and equipment in the instructed manner: always leave the balance area clean and sweep off the balance pan after use.
7. The complete torso, upper arm to the elbow and legs to below the knee must be covered. Tank tops or mid-riff-exposing apparel is strictly forbidden. Avoid flammable fabrics, such as nylon. Wear a lab coat or apron if desired.
8. Know the location and proper use of safety equipment in the laboratory: shower, eyewash, fire extinguisher, fire blanket.
9. Dispose of chemicals and reagents properly. Ask the instructor if you don't know how. Dispose of glass in the marked receptacle especially for broken glass. Use broom and dust pan to sweep up after broken glass.
10. **Label everything.** Identify the contents of all containers by name of compound, solvent (if any), concentration, and known additives or criterion pollutants (if any). Include your name and the date of preparation. Any time you need labels, JUST ASK!

Laboratory Manual: The C212 Laboratory manual is available for you to download from BlackBoard. It is also available for purchase from the copy center, in the lobby of the Education Building. All assigned experiments are in this manual, with the possible exception of one or more rotation experiments. These will be provided as hand-outs.

Preparation for the Experiment: It is the student's responsibility to be ready to work during the scheduled lab period. Some of the experiments cannot be completed in time unless you are organized and diligent. This means you **MUST** read and understand the instructions before coming to the lab if you wish to complete the experiment in the time allowed. You must know in advance if there will be wait times during the procedure, and how you plan to use those times efficiently so time is not wasted. It will be essential to multitask. Students are advised to prepare a flowchart of the experiment prior to the scheduled lab period.

The lab will not necessarily be open at any time other than the scheduled time, to accommodate students who were not prepared, or who arrived late. Students may come to other scheduled C212 sections with instructor permission, but must give precedence to the students enrolled in that section for use of balance, instruments, or other equipment. Occasionally the lab may be open at other times, if several students need extra time. In the event of an instrument

malfunction, which is not due to student's mistreatment of the instrument, every effort will be made to allow the student to make up the experiment, at a time convenient to the student.

Lab Notebook: A grid-lined, bound, laboratory research notebook is required. Loose leaf, spiral bound or three-ring binders are not acceptable. 2 points per report will be based on your observance of the following guidelines:

1. The laboratory notebook is a real-time record of work done in the lab. All laboratory work will be recorded directly in the bound notebook in ink, at the time the measurement or observation is made. Stray scraps of paper should not be used, and the instructor will confiscate them. The lab book is to be carried to the balance room, to other instruments used in the experiment, or to the computer lab on the third floor of SN building. However, the laboratory notebook will remain in the student's locker at the end of the day, and will not be taken home for data "massage" after the fact. If the notebook is removed from the building during the semester, fraud will be assumed and an appropriate penalty (such as a failing grade on the experiment) will be applied. The student may remove the pages that will be turned in, for reference only, to assist in writing the summary report which is a separate page.

2. Errors are corrected by drawing a **single line** through the entry that is in error, and then writing the correct information beside it. Write **your initials** by the correction, at the time you make it.

3. The lab notebook must include a Table of Contents, as the first 1-2 pages in the book. Every page in the book must be numbered sequentially, starting with the table of contents.

4. **Every page** of the notebook must be **signed and dated** as the work is completed.

5. No pages are to be left blank. If large sections of a page must be left blank (e.g. to start a new day's work on a fresh page) the large blank page or section must be X'd out, and the statement written, "This section intentionally left blank", or "NFETP" ("no further entries this page") with signature and date. Leaving a blank page for later use is **forbidden**.

6. Use appropriate headings to identify each section. Section headings will usually include:

Purpose (e.g. "To determine fluoride in tap water, toothpaste and an unknown")

Procedure, data and observations. (Also see Procedure, below) The organization of these headings may vary with the type of data taken. Always include a brief statement of the steps taken, all the raw data and all observations appropriate to each procedure step; (e.g. "Replicate ~0.5 g samples of the unknown were weighed to ± 0.0001 g on the 4-place analytical balance. The unknown is a pale blue solid powder. Data: (here include the actual data for each sample as mass of weigh paper, total mass, and subtracted mass of sample.))

Calculations (At least one fully worked example of each **type** of computation. You may include all computations, if desired.

Conclusion (Must relate to the stated purpose, e.g. "fluoride content of tap water was found to be 0.4 ppm")

7. Procedure: It is not necessary to re-write the entire set of instructions from the manual, for each experiment. However, **enough detail must be included** that it is clear to a reader what was being done. The C212 manual can be referenced and any deviation from the instructions in the manual made clear. For example:

"The precipitate was filtered according to instructions in the C212 Manual, page 9, and the entire filter paper with precipitate and all filter paper wipings were placed in the crucible. A muffle furnace was used instead of a Bunsen burner, to ash the precipitate."

8. Preliminary plots of data may be made by hand directly in the notebook. If data or plots are generated by an instrument or computer, they can be included by doing the following:

- Neatly staple, glue or tape the hardcopy into the book.
- Initial and date the hardcopy in your own handwriting, even if that is part of the printed information on the hardcopy.
- Refer to the hardcopy in the text of your notebook **on the same page**, and indicate where it is on the page, using an arrow or other graphical device.
- Also attach a photocopy of the instrument-generated paper to the report you turn in.

9. Do not crowd the entries in your notebook. There are many more pages in the notebook than you will use in the semester.

10. Entries must be neat, legible, and clearly identified as to what they are.

These rules are **part** of the Federal guidelines for "Good Laboratory Practices" (GLPs), from the Code of Federal Regulations, 40 CFR 160. Not all aspects of regulatory GLP's are practical, necessary, or desirable for instructional laboratories, but you will be expected to know and follow them in any scientific or engineering career, anywhere else. Therefore we teach some of them here, and require those that pertain to your notebook (2 points per report).

Laboratory Reports: The written report for each experiment consists of two parts: (1) the original copy of your grid-lined notebook for that experiment and (2) a report in which you summarize your findings and answer theoretical questions. Written reports are usually due **one week after completion** of each experiment. If you are unable to complete the work in the time scheduled for that experiment, you may, at the instructor's discretion, negotiate a new due date. Obtain the instructor's signature and the new due date in your notebook. Otherwise the penalty for lateness will apply. Typically half of the grade for each experiment will be based on the accuracy and precision of your analysis. Reports **must be legible**. The instructor will decide what is legible.

Due Dates: All reports are due one week from the date the experiment is performed. They are due on the same day of the week as your scheduled laboratory class, by 5 pm. of that day. Late penalties will accrue as follows; only "business days" Monday-Friday are counted:

Report turned in by:	5 pm. one day late	-10%
	5 pm. two days late	-20%
	5 pm. three days late	-30%
	5 pm. four days late	-40%
	5 pm. five days late	-50%
	more than five days late	-100% (report will not be graded)

Analyst Certification: In the "real world", scientific laboratories certify or standardize their analysts, instruments, methods and reagents, to ensure the quality of their results. In this course, the student has the opportunity to earn BSU Analyst Certification in calibration, statistical data handling, titrimetry, gravimetry and spectroscopy. To earn this certificate you must obtain, on each of the designated experiments, results of $\geq 70\%$ score for your accuracy and precision. You will be eligible for certification any time in the semester that this skill level is demonstrated on these designated experiments. These analyses may be repeated up to two times for analyst certification (3 trials, total) however the original grade for the experiment will not be changed. The designated experiments are : # 1, 2, 4, and 9, from the C212 Laboratory Manual.

Learning Objectives:

Students will gain hands-on experience with classical chemical analysis techniques, including use of volumetric glassware, the 5-place electronic balance, principles of equilibria, and statistical handling of data. In addition, students will be introduced to modern practices in analytical chemistry including spectroscopy, chromatography and electrochemistry, and scientific recordkeeping as regulated by the Federal Government (namely, the Good Laboratory Practices specified in the Code of Federal Regulations). Students will have some opportunity to learn to work as one member of a team who cooperate in their tasks and data sharing, to explore a larger problem. Students may also earn Analyst Certification.

Outcomes Evaluation:

Students will demonstrate how well they have learned the objectives through the reports turned in for a grade. The reports have three components: the analysis itself in terms of precision and accuracy, student responses to questions about theory and practice, and scientific record keeping. A grade of $\geq 70\%$ is required to demonstrate satisfactory proficiency.

Course Grade: is assigned as follows:

Reports:	11 x 20 =	220	90-100 %	A
Lab notebook	11 x 2 =	22	80-89 %	B
Safety, cleanliness, attitude	<u>15</u>		70-79 %	C
	total	257	60-69 %	D
			$\leq 59\%$	F