

CHEM 503 – INSTRUMENTAL ANALYSIS LAB
Sec. 001, Course#12239, 1.0 Credit Hr – FALL 2013

Course Goals:

The purpose of this course is to expose students to the various types of equipment that are likely to be encountered in an industrial or government chemical analysis laboratory. While the industrial or government lab **may** be better equipped than our academic one, the basic types of instrumentation will be the same. The instruments are located in various places within Sims, but most are located in Sims 310, along with sample and standard preparation workspace.

You will work in small groups (2 or 3 students) for data collection, but further **collaboration on processing data or reporting is prohibited**. Background references are required reading, and can assist in processing data, introductory, and discussion concepts.

Each group will have an unknown sample(s) for most projects. Directions for the operation of each instrument (student manuals) are located near each device. You are expected to familiarize yourselves with this manual before you begin the instrumental work. Usually, the instructor will demonstrate the operating procedures prior to your measurements, but this is not a substitute for reading the instructions. Feel free to ask questions when they arise. Each group member is expected to contribute to making solutions as well as getting hands-on experience with each device.

Each instrument you encounter contains delicate components, even those instruments that appear to be out-dated, so handle them with care. If you believe that a malfunction has occurred, inform the instructor before continuing. You should always consult the instructor before making changes in instrumental parameters other than those described in the student manual and **never** perform an operation you have not been “checked-out” on by the instructor. These instruments are typically quite expensive (\$5,000 - \$200,000).

Winthrop University's faculty adopted a set of four University Level Competencies (ULCs) that describe the qualities our students develop during their Winthrop career. It is easy to see that this course involves solving problems and developing written communication skills. However, you will also learn the responsibilities of chemists to the greater good of our planet and society, as well as the global nature of the chemistry enterprise. Within the discussions of chemical analysis instrument design and applications to the environment, health and materials we use every day, I think you will find this course fits well with all four competencies:

Competency 1: Winthrop graduates think critically and solve problems.

Winthrop University graduates reason logically, evaluate and use evidence, and solve problems. They seek out and assess relevant information from multiple viewpoints to form well-reasoned conclusions. Winthrop graduates consider the full context and consequences of their decisions and continually reexamine their own critical thinking process, including the strengths and weaknesses of their arguments.

Competency 2: Winthrop graduates are personally and socially responsible.

Winthrop University graduates value integrity, perceive moral dimensions, and achieve excellence. They take seriously the perspectives of others, practice ethical reasoning, and reflect on experiences. Winthrop graduates have a sense of responsibility to the broader community and contribute to the greater good.

Competency 3: Winthrop graduates understand the interconnected nature of the world and the time in which they live.

Winthrop University graduates comprehend the historical, social, and global contexts of their disciplines and their lives. They also recognize how their chosen area of study is inextricably linked to other fields. Winthrop graduates collaborate with members of diverse academic, professional, and cultural communities as informed and engaged citizens.

Competency 4: Winthrop graduates communicate effectively.

Winthrop University graduates communicate in a manner appropriate to the subject, occasion, and audience. They create texts - including but not limited to written, oral, and visual presentations - that convey content effectively. Mindful of their voice and the impact of their communication, Winthrop graduates successfully express and exchange ideas.

Meeting Location/Time: Room 310 Sims Science Building
Monday, 2:00 – 4:50 pm

Instructor: Dr. Cliff Calloway (callowayc@winthrop.edu)
312-B Sims Science Building (x4945 or from off campus: 323-4945)
Office Hours: 10:00-12:00 MTWF and by appointment

Text: *Winthrop University Instrumental Analysis Lab Manual* and
Skoog, D.A., Holler, F.J., Crouch, S.R. *Principles of Instrumental Analysis*, 6th edition, 2007.

Pre-requisites: Chem 314 (Quantitative Analysis Lab)

Co-requisites: Chem 502 (Instrumental Analysis)

Student Learning Outcomes:

- Gain hands-on experience with sophisticated laboratory instrumentation
 - separation, electrochemical and spectroscopic techniques
 - computer instrument control and data acquisition
 - handling techniques for solid and liquid samples/reagents
- Develop an understanding of the components of laboratory instrumentation and how different parameters can influence data output.
 - instrument modules and design
 - basic electronics for data collection and instrument control
 - signal optimization
- Perform rigorous data collection and data analysis with the aid of desktop computers.
 - spreadsheets for data domain conversion and processing

- graphical representations of instrument response and performance
- Improve technical writing skills through written reports.
- familiarity with instrument, analytical and scientific literature resources
- format and writing formal reports, and verbal interactions with colleagues

Required Materials (for lab each week):

- Experiment handouts and course textbook (i.e. – the “Skoog book”)
- Laboratory notebook, one per student pair is adequate...the type with duplicate pages is handy, since each student can have a copy of the raw data (black ballpoint pen preferred)
- Safety glasses or goggles
- A calculator that performs least squares analysis, mean and standard deviation
- Lab coats (provided)

Attendance and Laboratory Hours:

The laboratory will meet for the **full** scheduled time. You may **not** work in the laboratory at other times without permission and supervision. The laboratory work, as designed, is to be completed within the laboratory period. Make-up labs are not typically scheduled without validation. Validate illnesses through Student Health. Non-illnesses should be validated through Student Affairs. Sports commitments are validated by Athletics.

Course Requirements:

There will be a total of **twelve** lab assignments valued at **50 pt each** and a **comprehensive lab final exam** valued at **50 pt** (distributed the last week of lab, due December 9th, 3:00 pm). Each group will complete one assignment per week. An assignment generally consists of: 1) a lab **report** (30 pt), 2) a lab **notebook** (10 pt), and 3) a lab **quiz** (10 pt).

- **Report:** The lab report will consist of: Title Page, Introduction, Procedure, Sample Calculations, Results, Conclusion, Discussion, and Reference sections. (See Laboratory Report below for further details.)
- **Notebook:** At the beginning of each lab period, each group must submit, for approval, a written experimental plan. The plan should be in the group’s notebook and include a detailed scheme for the preparation of solutions and for carrying out the task of the lab assignment. (See Laboratory Notebook below for further details.)
- **Quiz:** A written 10-15 minute quiz will be given at the beginning of the laboratory period covering the previous week’s lab. (See Laboratory Quiz below for further details.)

Letter Grades:

As you have probably calculated, 650 points are possible in CHEM 503. Your lowest project will be dropped. Letter grades will be assigned based on the percentage of 600 points as follows:

94-100%: A	90-93%: A-	86-89%: B+	82-85%: B
78-81%: B-	74-77%: C+	70-73%: C	66-69%: C-
62-65%: D+	58-61%: D	55-57%: D-	

Important Notes:

1. Again, lab reports are to be your own work, *no group effort*, and are covered by the Student Conduct Code: "Responsibility for good conduct rests with students as adult individuals." Refer to the "Academic Misconduct Policy" in the online Student Handbook: <http://www2.winthrop.edu/studentaffairs/handbook/StudentHandbook.pdf>

Since all graded work (including homework to be collected, quizzes, papers, mid-term examinations, final examinations, research proposals, laboratory results and reports, etc) are used in the determination of academic progress, no collaboration on such work is permitted unless the instructor explicitly indicates that some specific degree of collaboration is allowed. This statement is not intended to discourage students from studying together, seeking help from the instructor, or working together on assignments that are not to be collected. Copies of old lab reports from previous years are off limits.

2. All laboratory work (including reports) must be completed to receive a passing grade. The absolute deadline for submission of written work is Study Day, 5:00 pm.
3. Handwritten (rather than typed) reports will not be accepted. These will be returned and late points assessed. The late report policy is strictly enforced.
4. Remember that the lab experiment is a learning experience. Do not get overly upset if your results do not seem to come out as planned. Try to determine the source of error and comment in your **Conclusion** section. If your results are way off from an expected value, with **no** valid explanation, penalty point(s) will be assessed to the conclusion section.
5. Some of the procedures are lengthy, so you may begin to prepare solutions or clean glassware early, if you wish. In any event, all group members must be present when the lab period officially begins, when reports and notebooks are collected, and quizzes given.
6. **Students with Disabilities:** Winthrop University is dedicated to providing access to education. If you have a disability and require specific accommodations to complete this course, contact the Office of Disability Services at 323-3290 (Crawford Building, 110A), <http://www.winthrop.edu/hcs/default.aspx?id=23186>. Once you have your official notice of accommodations, please let me know as early as possible in the semester.
7. **Syllabus Change:** While unlikely, the Professor reserves the right to change the course syllabus if circumstances (weather or other events) dictate. You will be notified of any change through lab meetings and/or email.
8. **Additional Requirements for Graduate Level Credit:** Students wishing to receive graduate level credit for this course are required to complete a 5-7 page review paper on a cutting edge analytical technique. Resources for choosing a topic can be found by reviewing either the "Fundamentals Review" or "Applications Review" issues of the journal, *Analytical Chemistry* (June 15th issue of even & odd years, respectively). The topic requires instructor approval. The paper must cite a minimum of 10 primary literature sources and must be submitted by Study Day. Graduate students should be aware that Winthrop's +/- grading system is not applicable to courses taken for graduate credit. Letter grades will be assigned as follows: 92%-100% A; 83%-91% B; 74%-82% C, 55%-73% D.

CHEM 502 Lab Rotation Schedule - Fall 2013

NMR	Time/Frequency Domains and Digital Filtering
AAS	Limit of Detection in the Analysis of Coins by Flame Atomic Absorption Spectrometry
IR	Determination of Benzoyl Peroxide in Acne Solutions, Quantitative IR
EL1	Basic Analog Electronics and Circuits (No quiz.)
DP	Data Processing – Moving Average/Savitzky-Golay/Ensemble/Fourier Analysis with Excel (No quiz.)
UV1	Construction of a UV spectrophotometer; Basic Optics
MLS	Linearity and Heavy Atom Fluorescence Quenching
COUL	Coulometric Determination of the Iodine Number for Cooking Oils
EL2	Basic Digital Electronics and Instrument Control (No quiz.)
SA	Surface Analysis (No quiz.)
LCMS	Separation and Molecular Weight Determination of an Organic Mixture with Hyphenated Liquid Chromatography/Mass Spectrometry
CE	Plates and Resolution in Capillary Electrophoresis

Date\ Group	A	B	C
8/26	Introduction	Introduction	Introduction
9/2	NMR	AAS	IR
9/9	AAS	IR	NMR
9/16	IR	NMR	AAS
9/23	EL1	DP	UV1
9/30	DP	UV1	EL1
10/7	UV1	EL1	DP
10/14	Fall Break	Fall Break	Fall Break
10/21	MLS	COUL	EL2
10/28	COUL	EL2	MLS
11/4	EL2	MLS	COUL
11/11	SA	LCMS	CE
11/18	LCMS	CE	SA
11/25	CE	SA	LCMS
12/2	Final Exam	Final Exam	Final Exam