

**CHEM 502H - Instrumental Analysis - Honors**  
**Section 001, Course# 12411, 3.0 Credit Hrs., Fall Semester, 2015**

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**Time/Location:** 12:30 - 1:45 TR / Sims 112

**Professor:** Dr. Cliff Calloway, [callowayc@winthrop.edu](mailto:callowayc@winthrop.edu)

**Office/Phone:** 312-B Sims Science Building / 323-4945

**Office Hours:** MTWF 10:00 - 12:00 {And other times by appointment. Please don't hesitate to contact me.}

**Textbook:** Skoog, D.A., Holler, F.J., Crouch, S.R. *Principles of Instrumental Analysis*, 6<sup>th</sup> edition, 2007.

**Pre-requisites:** Grade of C or better in CHEM 313, 314 and CHEM 301

**Co-requisite:** CHEM 503, Instrumental Analysis Lab

**Registration Calendar** ([link to important dates for registration, S/U, graduation, etc.](#))

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**Introduction:**

Chemical analysis methods are used in biotechnology, pharmaceutical, environmental, geological, materials development, forensic, medical, nutritional, energy and industrial labs daily that have a profound impact on decisions made locally, regionally, nationally and globally. Scientists and engineers conducting research and development for these industries often seek answers to chemical identity, structure, or amounts questions. As such, chemical analysis plays a critical role in scientific development and the quality of our lives. Qualitative and quantitative methods of chemical analysis for organic, biochemical, and inorganic compounds fall into two categories, **classical (or wet)** methods and **instrumental** methods. In Chemistry 313/314, we surveyed some classical methods of analysis, including titration, gravimetric and volumetric analysis, as well as some instrumental methods of analysis (gas & liquid chromatography, atomic & molecular spectrometry, and electrochemistry). Although there is not always a clear barrier between the two, the primary difference arises from the type of physical property used to provide information. Classical methods often rely on reactivity or physical properties such as solubility, color, melting and/or boiling points, odors, or refractive indices for qualitative information, while gravimetric, volumetric, and titrimetric measurements provide quantitative information. Classical methods for the separation of mixtures are mainly solvent extractions, precipitation, and distillation. Instrumental methods of analysis typically utilize other physical properties such as absorption or emission of light, mass-to-charge ratio, electrode potential, current, or charge measured with modern sophisticated electronic devices. Separations are carried out by more efficient chromatography and electrophoresis methods.

Instrumental methods of analysis certainly extend well beyond the chemistry lab. Unfortunately, some scientists view and utilize these instruments as "black boxes". The term implies a device in which the scientist places a sample and somehow a number is generated that influences the scientist's decision-making process. It should be apparent that this approach could be dangerous, as the old saying "Garbage In/Garbage Out" is often true. As such any scientist using sophisticated instrumental equipment needs at least a basic understanding of how these devices are designed to work.

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.

**Course Goals:**

Instrumental Analysis is a broad and continually expanding subject as new technologies emerge, but these methods can generally be categorized as spectroscopic, electrochemical, or chromatographic. In this course, we will essentially take the cover off these "black boxes" to see how these instruments are constructed and how measurements are made from the underlying chemical and physical properties of the substance. In fact, you are likely to see instrumentation represented from other courses you've taken, demonstrating the broad impact instrumentation has in science. Quantitative problem solving will be utilized as a means to demonstrate the chemical and physical principles applied to the design and performance of instruments.

The goal of this course is NOT to make you an "expert" on every type of instrumentation to be encountered in a science lab, but rather to introduce and educate you to the common principles as well as the variety of instrumentation available for chemical analysis and the type(s) of information these instruments provide. It is my hope that you will then expand your knowledge of the instruments you come into contact with during your scientific career, thereby avoiding the "black box" problem.

**Student Learning Outcomes:**

By the end of this course, you should be able to demonstrate:

- an understanding of how chemical and physical properties of substances are used in the design and construction of modern sophisticated instrumentation used for chemical analysis
- a broad knowledge of the types of instrumentation generally available and the information provided by each, applications
- a knowledge of appropriate instrumental methods for addressing a chemical analysis problem
- the advantages, disadvantages, and limitations of different instruments used for similar types of analyses
- rigorous mathematical methods to evaluate instrument performance

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**Course Requirements for Grading/Evaluation:**

**Student Conduct Code:** "Responsibility for good conduct rests with students as adult individuals." Since all graded work (including homework to be collected, quizzes, papers, mid-term examinations, final examination, 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. Refer to the "Academic Misconduct Policy" in the online *Student Handbook*:

<http://www.winthrop.edu/uploadedFiles/studentconduct/StudentHandbook.pdf>

Grades in this course will be determined from three requirements, as follows:

- Homework (20%): Periodically, homework problems will be assigned from the textbook or as handouts, and collected. Due dates for each assignment are the final date each assignment will be accepted. Your lowest homework assignment will be dropped before averaging.
- Mid-term Exams (60%): There will be 4 exams given during the term covering the topics listed below. Make sure to bring pencil and scientific calculator to the exam. Make-up exams will not be given except under extreme circumstances. If you plan to miss an exam with valid excuse, let me know as soon as possible. The exams are scheduled as follows:
  - Exam 1: September 10
  - Exam 2: October 6
  - Exam 3: November 3
  - Exam 4: December 1
- Final Examination (20%): Thursday, December 10, 11:30 p.m. - 2:00 p.m. This will be a cumulative final examination given during exam week. If you score higher on the final exam than your lowest mid-term exam, the final exam grade will replace the lowest exam grade, before averaging.

Letter grades will be assigned 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- |         |    |         |    |         |    |         |    |

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#### **Attendance:**

You are expected to attend each class meeting for the full scheduled time. Instrumental Analysis is a difficult upper-level course and this will help you to concentrate on the appropriate material and reinforce the assigned readings and problems. You are required to bring pencil, paper, textbook, and a scientific calculator to each class. Cell phone calculators are not acceptable.

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#### **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.

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#### **Additional Requirement for Honors Credit:**

To receive Honors credit, you will construct and optimize a modern atomic flame emission spectrophotometer. This will include assembly of the gas burner/drain system and gases (ventilation hood will be provided), the optical system including quartz focusing lens and CCD spectrophotometer, as well as computer data acquisition system. Opto-mechanics will be used to align the system to maximize signal-to-noise ratios.

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#### **Additional Requirement 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 can be found by reviewing either the "Fundamentals Review" or "Applications Review" issues of the journal, *Analytical Chemistry* (June 15<sup>th</sup> issue of even & odd years, respectively). The paper must cite a minimum of 10 primary literature sources and must be submitted by December 5, 2013. 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.

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#### **Syllabus Changes:**

While unlikely, the Professor reserves the right to change the course syllabus if circumstances dictate. You will be notified of any change via class meeting time and/or email.

**Course Calendar:**

**\*Revised August 24, 2015**

| <b>Tentative Schedule*:</b> |                                   |                                      |
|-----------------------------|-----------------------------------|--------------------------------------|
| <b>Date</b>                 | <b>Lecture Sections</b>           | <b>Topic</b>                         |
| T, 25-August                | Introduction, Ch.1                | Figures of Merit                     |
| R, 27-August                | Ch. 2A                            | Electronics Basics I                 |
| T, 1-September              | Ch. 2B-C                          | Electronics Basics II                |
| R, 3-September              | Ch. 3A-C                          | Operational Amplifiers               |
| T, 8-September              | Ch. 5A-B, Review                  | Signals and Noise, Review            |
| R, 10-September             | <b>Exam 1 (Ch. 1-3C, 5)</b>       |                                      |
| T, 15-September             | Ch. 6A-B                          | Spectroscopy I                       |
| R, 17-September             | Ch. 6C-D                          | Spectroscopy II                      |
| T, 22-September             | Ch. 7A-B                          | Optical Components I                 |
| R, 24-September             | Ch. 7C-D                          | Optical Components II                |
| T, 29-September             | Ch. 7E-7F                         | Optical Components III               |
| R, 1-October                | Ch. 7G-7H, Review                 | Electronic Spectroscopy I, Review    |
| T, 6-October                | <b>Exam 2 (Ch. 6-7H)</b>          |                                      |
| R, 8-October                | Ch. 13A                           | Electronic Spectroscopy II           |
| T, 13-October               | Ch. 13B-D                         | Luminescence I                       |
| R, 15-October               | Ch. 15A-C                         | Luminescence II                      |
| T, 20-October               | <b>Fall Break</b>                 |                                      |
| R, 22-October               | Ch. 16A                           | Infrared Spectroscopy I              |
| T, 27-October               | Ch. 16B-C                         | Infrared Spectroscopy II             |
| R, 29-October               | Ch. 18A-C, Review                 | Raman Spectroscopy, Review           |
| T, 3-November               | <b>Exam 3 (Ch. 13, 15, 16,18)</b> |                                      |
| R, 5-November               | Ch. 19A-C                         | Nuclear Magnetic Resonance I         |
| T, 10-November              | Ch. 19D-H                         | Nuclear Magnetic Resonance II        |
| R, 12-November              | Ch. 20A-B                         | Mass Spectrometry I                  |
| T, 17-November              | Ch. 20C-E                         | Mass Spectrometry II                 |
| R, 19-November              | Ch. 30A-B                         | Capillary Electrophoresis I          |
| T, 24-November              | Ch. 30C-D; Review                 | Capillary Electrophoresis II, Review |
| R, 26-November              | <b>Thanksgiving Break</b>         |                                      |
| T, 1-December               | <b>Exam 4 (Ch. 19, 20, 30)</b>    |                                      |
| R, 3-December               | Review & Course Evaluation        |                                      |
| T, 8-December               | <b>Study Day</b>                  |                                      |
| R, 10-December              | <b>Final Exam - 11:30 am</b>      |                                      |

\*Subject to change, if weather or events make it necessary.