

CHEM 461/661 Advanced Instrumental Methods of Analysis Spring Semester 2015

Instructor: Dr. Ryan J. White
Office: MEYR 462B
Phone: (410) 455-3053
Email: rjwhite@umbc.edu
Office hours: T,Th 11:30-12:30 or by appointment

Lecture: T, Th 10:00 – 11:15, MEYR 272
Lab Section 2: Monday 1:00 – 5:00 pm
Lab Section 3: Wednesday 1:00 – 5:00 pm

Laboratory Instructor: Dr. Brian Cullum; Office: MEYR 243B; Phone: (410) 455-2833; E-mail: cullum@umbc.edu; Office Hours: M, W 9:00 am – 10:00 am or by appointment

Important February 6 – Last day to drop without a grade of "W"

Dates: March 15 – 22 – Spring Break, No Class

May 19 – Final Exam 10:30 AM – 12:30 PM

Course Description: This course is a combined lecture-laboratory class, in which students will be exposed to the theory, instrumentation, and applications of modern instrumental techniques employed for chemical analyses. Students will obtain hands-on experience in operating and understanding the components of these instruments in the laboratory portion of class..

Course Objective: By the end of this course you will be able to: 1) *understand* the principles and theory behind operation of common modern instrumentation for chemical analyses, 2) *identify* the most useful instrumentation involved in chemical analyses, and 3) *interpret and critically assess* results and understand how to apply this knowledge to different samples.

Tentative Lecture Schedule (topics and dates are subject to change):

<u>Week</u>	<u>Topic</u>	<u>Chapter(s)</u>	<u>Exam</u>
01/26	Introduction/Statistics/Error	1,5	
02/02	Analytical Figures of Merit	5	
02/09	Electronics	2-4	
02/16	Electronics	2-4	
02/23	Electrochemical Methods	22-25	Exam 1
03/02	Electrochemical Methods	22-25	
03/09	Surface-Based Techniques	21	
03/16	Spring Break – No Class	-	
03/23	Electromagnetic Radiation/Spectroscopic Phenomena	6,7,13	
03/30	Atomic Absorption/Emission	9,10	
04/06	Molecular Absorption/Luminescence	14,15	Exam 2
04/13	IR/Raman	16-18	
04/20	Chromatography	26-28	
04/27	Capillary Electrophoresis/Mass Spectrometry	30, 20	
05/04	Biosensors/New Frontiers	-	
05/12	Last Day of Class	-	Exam 3

Course Requirements:

Textbook: Skoog, Holler and Crouch "*Principles of Instrumental Analysis*," 6th ed., Brooks/Cole Thomson Learning.

Lab Text: You will need to purchase a carbonless copy lab notebook entitled “The Official Laboratory Research Notebook” for the lab portion of the class.

Literature Reports (661 students only): A brief, typed summary (no more than one page) of a *current* journal article (published within the last year) is **due every Tuesday**, unless otherwise noted, before lecture begins. The reports can be written on an article taken from any chemistry or physics journal as long as the article is related to an interesting and novel aspect of modern bioanalytical chemistry. The report should concisely summarize, *in your own words*, the technique developed, the key discoveries reported and the importance of the discovery to bioanalytical chemistry. *Do not paraphrase the article* and do not describe experimental details unless they are truly necessary in understanding the conclusions presented. In your report please include the title of the article and cite the article in ACS format. **No reports are due on exam weeks.** Reports submitted late or that exceed one, single-spaced typed page will not be accepted.

Grading: The lecture and the laboratory portions of the course will be graded separately. **A grade of "D" or better is required in each portion to pass the course in order to pass.** Final grades for the course will be 60% lecture and 40% laboratory. Lecture grades will be comprised of three one-hour exams, one final exam, and classroom participation (e.g., group projects). Laboratory grades for Chem 461 students will be based upon ten laboratory reports, a laboratory final exam, and laboratory notebook condition. Laboratory grades for Chem 661 student’s will be based upon development and validation of their instrument and their laboratory notebooks. The weighting of these various components are described below.

CHEM 461	% of Grade	CHEM 661	% of Grade
Three one Hour Exams	45	Three one Hour Exams	40
Class Participation	5	Class Participation	5
Final Exam (ACS)	10	Weekly Reports	5
Laboratory Reports	30	Final Exam (ACS)	10
Laboratory Final	5	Instrument Development	30
Laboratory Notebook	5	Instrument Validation	5
		Laboratory Notebook	5
TOTAL	100	Total	100

Letter Grades for the class will be assigned based upon the following distribution.

Class Percentage	Letter Grade
90-100	A
80-89	B
69-79	C
56-68	D
0-55	F

Academic Conduct: Students are required to comply with the University rules of conduct, as described at <http://www.umbc.edu/NewsEvents/Student/oldstconduct.html> and related links. UMBC takes a strong stand against academic dishonesty in the classroom. To read the full Statement of Academic Integrity, go to <http://www.umbc.edu/provost/integrity/Honorcode.htm>.

Cheating, fabrication of data, plagiarism, and other violations will not be tolerated in this course. Remember, UMBC is an Honors University.

Turning in work that is reproduced in whole or in part from a classmate's paper, any internet source, or any textbook or journal article without proper citation will be treated as an academic integrity violation and reported to the Office of the Provost. UMBC states that the penalty for such a violation is to be at the discretion of the instructor; the penalty in this class will be at least a zero on the assignment for a first offense. Subsequent offenses will be rewarded with a failing grade in the class.

Makeups: Make up labs and exams will be given in accordance with University policy: a signed and readable note on letterhead paper from a physician, a police report, a certificate from a funeral home, etc. are required to be on file.

Exam Re-evaluation: In the event you would like your exam re-evaluated, please submit within one week your exam and 2-3 sentences on why you would like the particular question re-evaluated. If there was an obvious mistake in adding points/grading please see me first.

Laboratory reports:

10 lab reports (for experiments 1 to 10) must be turned in for grading **two weeks** after the respective lab experiment has been completed. Lab reports will be graded from 0 to 100. **A mandatory 10 point deduction will be assessed on the lab report grade for each day it is late (the end of a day will be considered as being 1:05 pm; the beginning of lab),** calculated starting from the due date. **A minimum 5 point deduction will be assessed for significant figures and rounding errors.**

Missing lab reports, or reports that are turned in after the week of May 5th, 2014 will be graded as 0's and be included in the calculation of the final grade for the laboratory portion of the course.

- Laboratory experiments will be performed on a rotating basis. Each laboratory section will be divided into groups (e.g., A, B, C and D) during the first laboratory class.
- Makeup Labs will be performed **within a week of the missed experiment**. It is your responsibility to schedule a make-up lab and to provide all necessary documentation.
- All laboratory reports will be **due at the beginning of lab** two weeks after the lab was completed. Late penalties (10 points/day) will accrue starting at 1:05 PM the day of lab and each day thereafter.

Week of	Monday				Wednesday			
	A	B	C	D	E	F	G	H
02/02	1	1	1	1	1	1	1	1
02/09	2	3	4	5	2	3	4	5
02/16	5	2	3	4	5	2	3	4
02/23	4	5	2	3	4	5	2	3
03/02	3	4	5	2	3	4	5	2
03/09	SNOW DAY MAKE-UP WEEK							
03/16	SPRING BREAK - NO LABS							
03/23	10	6	7	8	10	6	7	8
03/30	9	10	6	7	9	10	6	7
04/06	8	9	10	6	8	9	10	6
04/13	7	8	9	10	7	8	9	10
04/20	6	7	8	9	6	7	8	9
04/27	Laboratory Checkout/Laboratory Final							

Letters indicate the teams; numbers indicate the laboratory experiments to be performed

EXPERIMENTS:

- Introduction to the Instrumental Laboratory: statistical methods and analytical figures of merit
- Electronics and Chem. Instrument.: Op. amps, circuit review and measurement of pH
- Potentiometry with Ion Selective Electrodes: determination of fluoride in consumer products
- Cyclic Voltammetry: study of electrode mechanisms by cyclic voltammetry
- Anodic Stripping Voltammetry: determination of lead and cadmium in unknown samples
- Capillary Gas Chromatography: quantitative analysis of mixtures by gas chromatography
- Gas Chromatography-Mass Spectrometry: identification and quantification of flavoring agents
- Atomic Absorption Spectroscopy: flame atomization, determination of copper and lead
- HPLC: separation of urinary compounds by reversed phase chromatography
- Ultraviolet-Visible Absorption Spectroscopy: determination of the pKa of an indicator

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Materials:

1. Lab Notebook: Students should **purchase a 100-page notebook (carbonless copy)**. Procedures for the experiment are to be summarized in the notebook **BEFORE** the scheduled lab period. All data are to be recorded directly in the notebook in ink. Make certain to maintain an organized and legible notebook.
2. Safety: **Goggles are to be worn in the laboratory at ALL times.** In addition, workbenches are to be kept clear of clutter (books, backpacks, etc.). Appropriate clothing is to be worn in the laboratory - no sandals, ballet shoes or open-toed shoes, no shorts or sleeveless shirts. Long hair should be tied back. **NO FOOD OR DRINKS IN THE LAB!**

LABORATORY NOTEBOOK GUIDELINES

Your lab notebook is intended to be a substantive record of work performed in the laboratory, in which you must record the data/results obtained from your experiments. It should contain sufficient information so that **anyone** reading your notebook can reproduce your experiments, and evaluate your conclusions. In general, scientific notebooks follow a basic format provided below.

Notebook Format:

1. Table of Contents - Leave a few pages at the beginning of you notebook so that you can list the individual experiments, the dates on which they were performed, and the pages on which the relevant procedures and data may be found.
2. Experiments - **Each lab exercise should be dated and initialed on every page.** In addition, the following items should be included:
 - a) Title - Should be listed both in the Table of Contents and on the first page of the experimental section.
 - b) Objective - A brief (2 to 3 sentences) description of the purpose and goals of the analyses to be performed.
 - c) Procedures - An itemized list of the sequential steps performed, including information relevant to the preparation of samples, standards, and reagent solutions, and the equipment utilized.
 - d) Data - **All data obtained during the analysis should be permanently recorded, in ink, directly in the notebook! Do not write data on pieces of scrap paper!** The data should be clearly labeled (including appropriate units) so that it is obvious what the data represents, i.e. tare weight, weight of tare + sample, etc. If you anticipate accumulating a lot of data, use data tables for convenient data entry. *Preparation of these tables ahead of time can save time during the lab.* If you make a mistake in entering data, **do not erase**, rather, draw a single line through the erroneous value(s) and enter the correct value(s) either above or next to the mistake(s). Include graphs when appropriate.
 - e) Calculations - Provide any necessary calculations for the experiment (i.e., solvent volume calculations to provide a specific concentration solution from a certain amount of solute) in

your laboratory notebook. Your calculations should be clear enough so that it is obvious how the final result was achieved.

f) Observations - Report any useful, unexpected or necessary observations (e.g., solution turned pink momentarily, followed by a return to clear). When possible provide potential explanations for unexpected phenomena.

Original pages of your notebook MUST BE TURNED IN IMMEDIATELY FOLLOWING THE LAB.

The duplicate pages in the notebook can then be used by you to complete the laboratory report and any associated analyses.

LABORATORY REPORT GUIDELINES

For each of the ten laboratory experiments performed in this class, a written report of the results obtained and answers to any questions posed in the laboratory protocol should be answered. These reports should be typed up or be hand-written legibly. Any portion of the report that is not clear or legible to the grader or the instructor will be marked incorrect. These reports should be clearly organized and include answers and results to the **“Treatment of Data”** and **“Questions”** sections of the laboratory protocol, **as well as** any other questions raised throughout the other portions of the laboratory protocol.

Instrument Development (Chem 661 Students Only):

Students enrolled in Chem 661, will spend the lab periods designing and building either an HPLC, CV, UV-Vis, fluorimeter or other piece of instrumentation to operate with specific figures of merit. At the end of the semester, samples will be provided by the instructor for validation of the instruments response. Grades for the project will be given for weekly progress on the development (including weekly written assignments and questions) as well as the final validation of the instrument and the students ability to interpret the data output to answer the specific question(s) posed during the initial week of the lab.