## Chemical Analysis II Chemistry 254

# University of Mary Washington Spring 2011

Lecture MWF, 11:00 – 11:50 (Jepson 210)

Laboratory W & F 1:00-3:50 (Jepson 210, 310, or 309)

Instructor Dr. Charles Sharpless (Jepson 340, <u>csharple@umw.edu</u>, Phone # 654-1405)

Office Hours M (9:30-11 AM, 1:30-3 PM), W & F (10-11 AM). Appointments also welcome!

Course Website <u>http://blackboard6.umw.edu/</u>

#### **Required Course Materials**

Textbook: <u>Quantitative Chemical Analysis</u>, 7th ed., Harris, D.C.

Lab Coat, Safety Goggles, Lab Notebook (carbonless copy type), Scientific Calculator

**Course Objectives:** Chem254 provides a theoretical and hands-on introduction to some of the most common spectroscopic and chromatographic instrumentation used in chemical analyses. Our major goals are to:

- understand the basic theory underlying the construction of several common instruments
- become familiar with the operation of spectroscopic and chromatographic instruments and the influence of instrumental settings
- apply proper data analysis to evaluate the accuracy of your quantitative technique
- gain familiarity with identifying and correcting common errors and interferences

Grading

Reports (best 5 of 6) and final project (20% of lab grade)
Three 50 min. exams
Final exam

\* No late assignments will be accepted without my prior consent.

\* Missed laboratories and exams may not be made-up without my prior consent.

**Class Attendance** Attendance is mandatory for laboratories and expected for lectures, which will emphasize selected topics that may not be covered in detail in the text. For labs, important safety and procedural information will be covered, and lateness is not acceptable.

Lab Exercises and Grading There is no lab manual for this course. Experimental procedures and readings will be posted on Blackboard. You must download these before lab to properly prepare for the exercise. Furthermore, the labs will frequently cover material not yet introduced in lecture. It is therefore essential that you do the assigned readings prior to performing labs.

<u>Lab reports</u>: All reports must be double-spaced with 1.25 in. margins, numbered pages, and are to be completed *individually*. They should be between 3 and 5 pages (not including appendix) and follow the format below. A grading rubric is posted on Blackboard and *must be stapled to submitted reports*.

Title: including your name, your partners' names, dates of work, and title

Abstract: one brief paragraph describing your major results.

*Procedure*: a <u>concise</u> summary with enough detail to be reproduced by a stranger

Data and Results: presentation of your results in text and tabular or graphic format as appropriate with accompanying prose briefly describing the results.

Discussion Questions: answers to assigned questions (provided with lab)

Appendix: notebook pages including all calculations leading to results presented in report

Each lab will require slightly different report content, and we will discuss this in class or it will be made explicit in the lab assignment. Many labs will require you to submit a spreadsheet data analysis. When this is the case, it will be graded as part of the analysis & calculations.

<u>Notebooks</u>: Your lab notebook serves as a journal record of your experimental methods and data. Someone else looking at it should have no difficulty understanding what you did and should be able to follow your methods and duplicate your results. *Each experiment should start on a new page, have a descriptive title, begin with a brief statement of purpose and a procedural outline, and clearly present the raw data as it was collected.* There should also be a clear collection of calculations that were performed on the data. In cases where there are many repetitive calculations that were done via spreadsheet, the notebook should outline the calculation procedure and include at least one example calculation.

<u>Lab Safety</u>: Safe lab practices are expected of you. There are potential risks, such as exposure to hazardous chemicals and minor injuries (e.g., cuts and burns). During the first week of the semester, the safety rules will be presented and reviewed. To participate in this course, each student must sign a statement in which they acknowledge the risks associated with the course and agree to follow all safety rules and to assume responsibility for their actions in the laboratory.

**Practice Problems:** Suggested problems from the 7<sup>th</sup> edition of the textbook are listed at the bottom of the weekly schedule (below). Along with example problems from lecture, these are representative of questions you can expect on exams. The answer key is available in my office (J340), and you should feel free to drop by at office hours or other times (as long as you know I'll be there) to check your work.

**Final Project**: At the end of the semester, you will complete a self-directed project in teams. This will count as 20% of your lab grade. Graded portions of the projects include a presentation and a paper. Experimental work will occur during the last three weeks of the semester. Serious planning must occur well before the work begins. This means paying careful attention to detail in the areas of sampling, sample preparation, instrumental method validation, and obtaining high quality data.

*Project Topics*: Topics may be drawn from several sources including: a) recent chemistry literature (i.e., any ACS journal); b) chemical education literature (i.e., the Journal of Chemical Education); or c) an independently planned experiment. You may want to develop a teaching lab, verify or adapt a published analytical technique, or perhaps assemble and demonstrate a novel instrumental device. We will discuss projects in detail on March 7<sup>th</sup>. Key dates to note are:

3/25: three possible projects must be selected and submitted in outline form

3/30 – 4/1: discussion of possible topics with Dr. Sharpless, proposal development

4/4: proposals due

Failure to meet these deadlines will result in a 5% penalty to your grade for each missed deadline. Extensive delays in meeting deadlines may result in a failing grade for the project.

**Students with Disabilities** If you require accommodations due to a disability, visit the Office of Disability Services online (http://www.umw.edu/disability/) for information about available resources. You will need to request accommodations through this office as soon as possible. If you have allergies to any chemicals or other emergency medical information, or have any other special needs, please notify me.

**Honor System:** <u>All work submitted for grading must be your own and be pledged as such</u>. It is your duty as students and mine as faculty to uphold the Honor Code. Suspected honor violations will be addressed according to the policy established by the Honor Council.

While working in the laboratory, you will need to work together to collect data. Occasionally helping each other with a task or to understand the procedures is acceptable. However, it is not acceptable if one student is doing all the labor, or repeatedly issuing instructions to or aiding his/her partner. If you find that you need help understanding the material in lab (or lecture), please come see me.

Spring 2011 Weekly Schedule (Subject to change). assigned chapters in parentileses (7 – et	subject to change): assigned chapters in parentheses (7 <sup>th</sup> ed.)
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Week Of	М	W	F	LAB	
Jan 10 <sup>th</sup>	calibration (5)	calibration (5)	Meet in J108	Direct calibration (J210)	
Jan 17 <sup>th</sup>	MLK Day	calibration (5)	spectroscopy (18)	Double Beam UV-Vis (J309)	
Jan 24 <sup>th</sup>	spectroscopy (18)	spectroscopy (18)	spectroscopy (18)		
Jan 31 <sup>st</sup>	spectrometers (20) omit 20-4	spectrometers (20) omit 20-4	spectrometers (20) omit 20-4	Atomic Absorption (1309)	
Feb 7 <sup>th</sup>	spectrometers (20) omit 20-4	Exam 1 (ch's 5, 18, 20)	atomic spec. (21)		
Feb 14 <sup>th</sup>	atomic spec. (21)	atomic spec. (21)	mass spec. (22) omit 22-2 and 22-4	ICP-Atomic Emission (J309)	
Feb 21 <sup>st</sup>	mass spec. (22) omit 22-2 and 22-4	mass spec. (22) omit 22-2 and 22-4	ТВА		
Feb 28 <sup>th</sup>	SPRING BREAK				
Mar 7 <sup>th</sup>	project orientation	Exam 2 (ch's 21, 22)	separations (23) deadline for all course withdrawals	GC-MS (J210)	
Mar 14 <sup>th</sup>	separations (23)	separations (23)	GC (24)		
Mar 21 <sup>st</sup>	GC (24)	GC (24)	HPLC (25)		
Mar 28 <sup>th</sup>	HPLC (25)	project discussions	project discussions	HPLC (3310)	
Apr 4 <sup>th</sup>	HPLC (25) proposals due	MS in HPLC (25 and 22-4)	IC & CE (26) omit 26-3 and 26-4		
Apr 11 <sup>th</sup>	Exam 3 (ch's 23, 24, 25)	IC & CE (26) omit 26-3 and 26-4	IC & CE (26) omit 26-3 and 26-4	Projects	
Apr 18 <sup>th</sup>	project presentations	project presentations	project presentations		
Apr 25 <sup>th</sup>	FINA	L EXAM: Friday, Apr	il 29 <sup>th</sup> , 12:00 – 2:30 F	PM (Jepson 210)	

#### **Practice Problems**

Unit 1: Ch 5: Ex. A, B, C; Prob. 6, 7, 9, 12, 25, 28, 29
Ch 18: Ex. B, C; Prob. 3, 4, 6, 7, 8, 9, 10, 19, 20, 21, 24
Ch 20: Ex. A, C; Prob. 1, 2, 3, 5, 6, 8, 10, 11, 25, 26, 27, 29
Unit 2: Ch 21: Ex. C, D; Prob. 1, 5, 6, 7, 9, 12, 13, 18
Ch 22: Ex. A; Prob. 1, 2, 5, 6, 19, 20, 21, (22 for fun)

### *Unit 3*: Ch 23: Ex. B, F; Prob. 2, 3, 5, 8, 11, 15, 17, 21, 24, 27, 29, 30, 34, 37, 42 Ch 24: Ex. B, C; Prob. 2, 3, 4, 5, 6, 7, 9, 11, 13, 18, 20, 25, AND Ch 23-19 and 23-25 Ch 25: Ex. B, D; Prob. 1, 3, 4, 6, 7, 9, 10, 12, 17, 21, 24, 33, 34, 36, 37a Ch 22: Prob. 23, 25, 26

For final: Ch 26: Ex. C; Prob. 1, 3, 4, 12a, 13, 22, 23, 27, 32, 33

#### RELEASE FORM FOR CHEMISTRY LABORATORY STUDENTS

Please read carefully before signing.

I, \_\_\_\_\_\_, fully understand that there are hazards associated with performing new and unfamiliar procedures in a chemistry laboratory. I further understand that by signing up for CHEM \_\_\_\_\_\_ I must be especially careful to follow all safety rules and procedures while working in the laboratory. In addition to abiding by the Departmental safety rules, I agree that...

- (1) I will receive appropriate safety training, and afterwards I will follow all proper safety guidelines for working in the chemistry laboratory;
- (2) I will not work alone in the laboratory, and I will always have at least one other person, who has also had appropriate safety training, present with me in the laboratory when I am performing any procedure:
- (3) I will not perform unauthorized experiments, meaning that all experimental procedures will be approved before I perform them and that the quantities of reagents used will be no greater than the amounts delineated in the approved procedure (but they may be scaled down to lesser amounts);
- (4) I take full responsibility for my actions and for any injuries that I may incur as a result of performing the approved experimental procedures, and I will not hold the University of Mary Washington, the Chemistry Department, or Dr. Sharpless responsible should an injury occur;
- (5) If I take any action that puts me or anybody else at unnecessary risk of injury, I will immediately be dismissed from the laboratory and lose the privilege of participating in the laboratory portion of the course;
- (6) If I lose my right to work in the laboratory, I am solely responsible for lost time and any subsequent loss of grade points resulting from deadlines that cannot be met;
- (7) I will not remove any chemicals or equipment from the laboratory;
- (8) I will report any accidents immediately to Dr. Sharpless and the departmental safety officer, Dr. Crowder;
- (9) I will report any damaged or malfunctioning equipment or safety violations to Dr. Sharpless, to the departmental safety officer, Dr. Crowder, and to the department chair, Dr. Giancarlo.

Sianed:	
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Date: \_\_\_\_\_

Adapted from Dr. Bleil, Dakota State University, CHEM 241 Intermediate Organic Chemistry Safety Waiver Form.