CHEM 465: Analytical Chemistry
Spring 2024

Times and Locations: **Lecture:** M W F 9:00-9:50 am (Roddy 153)
Lab: Thu 1:10-4:00 pm (Caputo 223/224)

Instructor: Dr. Maria Schiza ○ **maria.schiza@millersville.edu** ○ **Office: Caputo 219**, tel.# 717-871-7437

Office Hours: Mon & Fri 12:30-2:00 pm, Wed 2:00-3:00 pm, Thu 10:00-11:00 am
Alternative times can be scheduled by appointment or virtually through Zoom.

Course Materials and Supplies:

1. Text: *Principles of Instrumental Analysis, 7th edition*, Skoog, Holler & Crouch, 2018.
2. Safety Googles/Glasses: must be worn in the laboratory at all times.
3. Laboratory Notebook: Permanently bound notebook, approximately 7" x 9.5", quadrille-ruled.

Course Policies:

Attendance

Regular attendance is expected for all lectures and laboratories. If an absence results in a missed exam or lab, the absence must be excused by the instructor before the exam or lab can be made up. Absences are excused for illness, death in the family, and college activities. Absences due to college activities must be discussed with the instructor in advance so arrangements can be made for making up the missed work. All missed work must be made up within one week of the student's return to class.

Grading Scale:

Grade	Percentage
A	92-100
A-	90-91.9
B+	87-89.9
B	82-86.9
B-	80-81.9
C+	77-79.9
C	72-76.9
C-	70-71.9
D+	67-69.9
D	62-66.9
D-	60-61.9
F	< 60

Important dates to remember

January 23rd: Last day to drop or add a class

March 29th: Last day to withdraw with a W grade

Three (3) Exams: 30% or 300 pts (10% or 100 pts each)
Final Exam (cumulative): 12% or 120 pts – Wednesday, May 1st (8:00-10:00 am)
Laboratory: 58% or 580 pts

- **Eight (8) instructor experiments: 48% or 480 pts (6% or 60 pts each)**
- **One (1) student designed experiment: 10% or 100 pts)**

Total from Lecture and Lab = 100% or 1000 pts

Lecture:

Lectures will cover the theory and technique of spectrophotometric, electroanalytical, and chromatographic methods. Exams will include information/material covered in the class lectures, labs and homework assignments.

Recommended Homework/Assignments:

Recommended Assignments will be given periodically in class.

TENTATIVE COURSE OUTLINE

- Analytical Methods
- Molecular Spectroscopy
  - UV-Visible Spectroscopy
  - Molecular Luminescence
  - Infrared Spectroscopy
  - Raman Spectroscopy
  - Molecular Mass Spectroscopy
- Atomic Spectroscopy
  - Spectrophotometric Methods
  - Atomic Absorption Spectroscopy
  - Atomic Emission Spectroscopy
  - Atomic Mass Spectrometry
- Surface Characterization by Spectroscopy/Electron and Atomic Force Microscopy
- Chromatography
  - Gas Chromatography
  - High Performance Liquid Chromatography
  - Supercritical Fluid Chromatography
  - Capillary Electrophoresis
- Electroanalytical Chemistry
  - Potentiometry
  - Coulometry
  - Voltammetry

- Other Methods (Thermal, Radiochemical, Automated, Particle Size Determination)

Laboratory:

The laboratory of the class will require more time outside of the class than many of your previous courses. Each student group will be assigned a specific experiment each week. However, during each laboratory period groups will be working on different experiments. Therefore, it will not be possible for the instructor to give an extended pre-lab discussion. In addition, the experiment assigned for a particular week may come well in advance for the discussion of that topic in lecture. It will be necessary for the student to have an understanding of the experiment and of the instrument before coming to lab.

All wet chemistry is to be done in the Analytical Lab and not at the location of the instrument to be used. At the end of each lab, all instruments should be cleaned and left in the manner in which they were found or better. All chemicals for each experiment must be returned to their proper place. Laboratory work should be done during the scheduled laboratory period. If additional time is needed, arrangements must be made with the instructor. Permission must be obtained from the instructor each time you use the laboratory outside of regularly scheduled hours. No student is to work in the laboratory alone.

Whenever time and the amount of sample permit, multiple samples should be analyzed (3 suggested). In addition, multiple trials (3 suggested) should be made of each sample. This will allow you to comment on any variation between samples as well as between measurements.

You are upper classmen and, therefore, expected to conduct yourselves in a safe and appropriate manner in the laboratory. This laboratory is designed to instruct you in the basic instrumental techniques used in research and industry. You are expected to learn independence of thought and action in the laboratory.

Laboratory Notebook:

The laboratory notebook is a permanent record of your work in the laboratory. You must have your notebook with you in order to work in the lab. All notebooks must be permanently bound and begin with a table of contents. All entries should be in ink. Each page must be consecutively numbered, with your name, date, and experiment title on the top of the page. All data must be recorded in the notebook using the correct significant figures. Never write data on another sheet of paper with the idea of transferring it to the notebook. Notebooks should be relatively neat and orderly; however, data should never be recopied in the notebook. If an error is made, do not use white out, tear out pages, recopy data or tape new pages. Simply draw a single line through the bad data and include an explanation why the error was made.

The notebook is a record of your work as it is performed. A running log of what you are doing in the lab should be kept in the notebook. The notebook should be kept is such way so that the instructor can turn to any experiment and tell exactly what you did during an experiment. All data must include units and appropriate labels. All instrumental parameters should be recorded for each trial. All calculations, tables and graphs must be included in the notebook.

Each laboratory experiment in the notebook must begin with a brief statement of the purpose of the experiment and a section with the materials used. This should consist of a table listing the chemicals names, the potential hazards associated with each chemical (information from SDS), and their proper waste disposal procedure. This is to be completed before the student begins the experiment. **Notebooks need to be signed by the instructor before each student leaves the laboratory.**

Laboratory Report:

These are formal reports and should be typed in a professional manner (**double spaced-12 font-Times New Roman style**). Reports should be approximately four-five pages of text excluding the title page, figures and tables, and references. Each lab report should contain the following (label and identify each section of the report):

1. **Title page/Abstract:** Name, partner(s) name in parentheses, date(s). Date(s) should include the date of the experiment performance and the date that the lab report is submitted (due). An experiment title and an abstract should be included. An abstract is a summary paragraph of the goal of the experiment, the method used, the principles on which the measurements are based on, and the type of techniques and instruments used as well as the major results of the experiment. (**a short paragraph**).
2. **Theory-Background:** Both the theory essential to an understanding of the chemical principles on which the measurement is based on and the theory necessary for understanding the equations used in the interpretation of the data should be discussed. A discussion of all chemical reactions taking place should also be included. Figures used in this section need to be original and include captions. (**1-2 pages**).
3. **Instrumentation:** Instrumentation diagrams should be original and include captions. Begin with a block diagram of the instrument with all major components labeled. This should be based on the instrument that you actually used. An explanation of the function of each component of the instrument should follow in the text. The name and specific model of the instrument should also be included (**1-2 pages**).
4. **Methods and Materials:** Give a list of materials used (using a descriptive paragraph – not a bulleted format) as well as the reference for where the procedure was found. Include changes to the procedure and a description of the sampling method (**a short paragraph**).
5. **Results (including data tables and graphs):** Results that relate directly to the purpose of the experiment should be clearly stated. Data tables and graphs should be employed whenever possible for presenting results. Label data clearly, include units, and maintain significant figures. Only data and graphs necessary for an explanation of the results need to be included (failed trials do not need to be reported). All tables and graphs included in the report must be referred to and explained in the conclusions and discussion section. An estimation of the statistical uncertainty associated with your reported results in terms of standard deviation and/or 95% confidence limits should be included.

   Tables/Graphs: For graphs use proper labels for the axes, and proper units. Include captions for any table, and graph in your report. Discuss all tables, and graphs in the conclusions/discussion section. Also spectra, chromatographs, or voltammograms that are attached to the lab report, should be labeled with a figure number and discussed in the conclusions/discussion section. Only those figures that are needed for the discussion need to be included. Rather than attach many pages of data, the important points of the data should be summarized in the table or as several plots overlaid on one page to show changes that occur.
6. **Conclusions and Discussion:** The conclusions should state or describe the results that relate directly to the purpose or goal of the experiment. Evidence then must be presented to either support or refute the quality of your results. This argument should include an indication of how good (or bad) you think your results are and why you think this is the case. This discussion should use your data, comparison of results from multiple trials and multiple samples, data scatter in graphs, other observation during the experiment, and the expected range of results obtained from the literature to help convince the reader of the quality of your results and conclusion. A quantitative a statistical analysis of your data should be included as well as a discussion of the sensitivity and detection limits of the instrument (**1 page**).
7. **References:** Report in correct bibliographic style (**minimum of 2 outside references**). References should be properly cited in the theory and instrumentation sections or wherever appropriate.

Lab reports will be due one week from the scheduled date for the experiment. NO LATE REPORTS WILL BE ACCEPTED. Notebooks should be signed and evaluated by the instructor at the end of each period.

Evaluation for Laboratory Notebook:	
Notebook	5 points
Evaluation for Laboratory Report:	
Format and Style/Grammar and Spelling	15 points
Abstract/Theory-Background	15 points
Instrumentation/Methods and Materials	15 points
Results/Data Presentation/Tables and Graphs	30 points
Conclusions and Discussion	15 points
References	5 points
TOTAL:	**100 points**

Lab Reports Rewrites/Revisions (5):

A lab report is due a week after each experiment is finished. Lab report rewrites/revisions will be allowed and accepted for 5 total experiments. Rewrites/Revisions will be allowed for the first 3 experiment Lab Reports (performed before Spring Recess), the experiment that you will submit as a Team report (after Spring Recess) and the experiment presented as a poster at the end of the semester. The first two lab reports, the team report, and the poster will be reviewed by the instructor. The third lab report (before Spring Recess) will be reviewed by a peer of yours before final submission.

You may rewrite and resubmit the first three graded lab reports, the team report, and the poster if you wish to improve your grade **(as long as the lab report was submitted on time originally and the first draft was similar in content to the final draft).** Both the old lab report and the new rewritten lab report should be paper clipped together and submitted within a week from the day the graded report is returned to you. **Only one rewrite will be allowed.** The poster should be submitted to the instructor for review 3 days after the completion of the experiment. Recommendations for revisions will be send to you by e-mail. Final poster presentations will be scheduled for the last week of the semester. All written lab reports (including the poster) are evaluated based on a rubric designed for written lab reports.

Oral Lab Report Presentations (2):

Two lab reports will be orally presented and evaluated using a rubric designed for oral presentations.

Student Designed Experiment: Proposal/Design/Performance

Each group of students will propose, design, and perform an experiment of their own by the end of the semester. The experiment selected can be adopted from the literature. This should be an analytical experiment that can be performed using existing materials and instrumentation at the MU chemistry department. **The proposal/design of this experiment is due before Spring Recess. It will be orally presented in lab and will be evaluated by your peers and the instructor.** It has to be approved by the instructor ahead of time in order for the group to have the appropriate time, materials, and equipment to perform the experiment after Spring Recess. **This will require some advance planning.**

Tentative Laboratory Schedule Spring 2024
Analytical Chemistry

Groups:	1	2	3	4
Jan 18	**CHECK-IN ---- Introduction to Lab**			
Jan 25	UV-Vis/part 1	FTIR-ATR (Instructor Review)	AA-F (Instructor Review)	RS (Instructor Review)
Feb 1	UV-Vis/part 2 (Instructor Review)	UV-Vis/part 1	FTIR-ATR (Instructor Review)	AA-F (Instructor Review)
Feb 8	RS (Instructor Review)	UV-Vis/part 2 (Instructor Review)	UV-Vis/part 1	FTIR-ATR (Peer Review)
Feb 15	AA-F (Peer Review)	RS (Peer Review)	UV-Vis/part 2 (Peer Review)	UV-Vis/part 1
Feb 22	FTIR-ATR (Oral Report I)	AA-F (Oral Report I)	RS (Oral Report I)	UV-Vis/part 2 (Oral Report I)
Feb 29	**Student Designed Experiment /Proposal Presentations**			
Mar 7	**Spring Recess**			
Mar 14	**Oral Report I - from Experiments on February 22nd**			
Mar 21	**SEM**	**SEM**	**SEM**	**SEM**
Mar 28	HPLC (Team Report) - (Instructor Review)	GC/MS/SPME (Team Report) -(Instructor Review)	Fluo (Team Report) - (Instructor Review)	Your Analytical Experiment*(Team Report) -(Instructor Review)
Apr 4	Your Analytical Experiment*	HPLC	GC/MS/SPME	Fluo
Apr 11	Fluo (Oral Report II)	Your Analytical Experiment* (Oral Report II)	HPLC (Oral Report II)	GC/MS/SPME (Oral Report II)
Apr 18	GC/MS/SPME (Poster) - (Instructor Review)	Fluo (Poster) - (Instructor Review)	Your Analytical Experiment* (Poster) -(Instructor Review)	HPLC (Poster) – (Instructor Review)
Apr 25	**CHECK-OUT---- Poster Presentations**			

-UV-Vis: UV-Visible Spectroscopy
-Fluo: Fluorimetry
-FTIR-ATR - Infrared Spectroscopy-Attenuated Total Reflection
-RS: Raman Spectroscopy
-AA-F: Atomic Absorption Spectroscopy - Flame
-HPLC: High Performance/Pressure Liquid Chromatography
-SEM: Scanning Electron Microscopy
-GC/MS/SPME: Gas Chromatography/Mass Spectroscopy with Solid Phase Microextraction Fibers
-Your Analytical Experiment Proposal/Design/Performance and Laboratory Report*
Alternative Lab-CV: Cyclic Voltametry

Laboratory Report Submission-Due Dates

Groups:	1	2	3	4
Jan 18	**CHECK-IN ---- Introduction to Lab**			
Jan 25	UV-Vis/part 1	FTIR-ATR (Written report due on Feb 1 - **instructor review**)	AA-F (Written report due on Feb 1 - **instructor review**)	RS (Written report due on Feb 1 - **instructor review**)
Feb 1	UV-Vis/part 2 (Written report due on Feb 8 - **instructor review**)	UV-Vis/part 1	FTIR-ATR (Written report due on Feb 8 - **instructor review**)	AA-F (Written report due on Feb 8 - **instructor review**)
Feb 8	RS (Written report due on Feb 15 - **instructor review**)	UV-Vis/part 2 (Written report due on Feb 15 - **instructor review**)	UV-Vis/part 1	FTIR-ATR (**peer review**) - (due on Feb 22 to the peer, Feb 29 back to the student, & Mar 14 to the instructor)
Feb 15	AA-F (**peer review**) - due on Feb 22 to the peer, Feb 29 back to the student, & Mar 14 to the instructor)	RS (**peer review**) - due on Feb 22 to the peer, Feb 29 back to the student, & Mar 14 to the instructor)	UV-Vis/part 2 (**peer review**) - (due on Feb 22 to the peer, Feb 29 back to the student, & Mar 14 to the instructor)	UV-Vis/part 1
Feb 22	FTIR-ATR (**Oral Report I** – Mar 14)	AA-F (**Oral Report I** – Mar 14)	RS (**Oral Report I** – Mar 14)	UV-Vis/part 2 (**Oral Report I** – Mar 14)
Feb 29	**Student Designed Experiment/Proposal Presentations**			
Mar 7	**Spring Recess**			
Mar 14	**Oral Report I - from Experiments on February 22nd**			
Mar 21	SEM (due on Mar 28 to the instructor)	SEM (due on Mar 28 to the instructor)	SEM (due on Mar 28 to the instructor)	SEM (due on Mar 28 to the instructor)
Mar 28	HPLC (**Team Written Report** due on Apr 4 to the instructor)	GC/MS/SPME (**Team Written Report** due on Apr 4 to the instructor)	Fluo (**Team Written Report** due on Apr 4 to the instructor)	Your Analytical Experiment* (**Team Written Report** due on Apr 4 to the instructor)
Apr 4	Your Analytical Experiment* (due on Apr 11 to the instructor)	HPLC (due on Apr 11 to the instructor)	GC/MS/SPME Your (due on Apr 11 to the instructor)	Fluo (due on Apr 11 to the instructor)
Apr 11	Fluo (**Oral Report II**– presented in class the week of Apr 18)	Your Analytical Experiment* (**Oral Report II**– presented in class the week of Apr 18)	HPLC (**Oral Report II**– presented in class the week of Apr 18)	GC/MS/SPME (**Oral Report II**– presented in class the week of Apr 18)
Apr 18	GC/MS/SPME (**Poster**- presented on Apr 25)	Fluo (**Poster**- presented on Apr 25)	Analytical Experiment* (**Poster**- presented on Apr 25)	HPLC (**Poster**- presented on Apr 25)
Apr 25	**CHECK-OUT---- Poster Presentations**			

Summary Table:

LABORATORIES [1,2,3,4]	LAB REPORTS
Experiment 1	Instructor reviewed/1 rewrite
Experiment 2	Instructor reviewed/1 rewrite
Experiment 3	Peer reviewed/1 rewrite
Experiment 4	Team Oral Presentation I
Experiment 5	Instructor reviewed/no rewrite
Experiment 6	Team Written Report Instructor reviewed/1 rewrite
Experiment 7	Instructor reviewed/no rewrite
Experiment 8	Team Oral Presentation II
Experiment 9	Team Poster Presentation/1 rewrite

1. One lab before Spring Recess is 2 weeks long
2. One lab after Spring Recess is based on a student group proposal/design
3. There is one peer reviewed lab report. **Part of your lab report grade will be based on the quality of your peer review.**
4. There are 5 opportunities for groups to work together as a team: a proposal/designed & proposed experiment presentation, two oral report presentations, a team written report, a poster presentation.

**All presentations will be formal and use Power Point.

Millersville University and its faculty are committed to assuring a safe and productive educational environment for all students. In order to meet this commitment, comply with Title IX of the Education Amendments of 1972, 20 U.S.C. §1681, et seq., and act in accordance with guidance from the Office for Civil Rights, the University requires faculty members to report to the University's Title IX Coordinator incidents of sexual violence shared by students. The only exceptions to the faculty member's reporting obligation are when incidents of sexual violence are communicated by a student during a classroom discussion, in a writing assignment for a class, or as part of a University-approved research project. Faculty members are obligated to report to the person designated in the University Protection of Minors policy incidents of sexual violence or any other abuse of a student who was, or is, a child (a person under 18 years of age) when the abuse allegedly occurred.

Information regarding the reporting of sexual violence, and the resources that are available to victims of sexual violence, is available at www.millersville.edu/titleix