

CHEM 465
ANALYTICAL CHEMISTRY

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COURSE DESCRIPTION: Theory and practice of modern analytical techniques in chemical separations and instrumental analysis. 3 hours lecture, 3 hours lab. Offered in spring. Prerequisite: ENGL 110 and Prerequisite or Corequisite: CHEM 342. Four credit hours. General Education W course.

Lecture: MWF 9:00 – 9:50 in Roddy 153 Lab: Thursday 1:00 – 3:50 in Caputo 223 & 224

COURSE MATERIALS:

1. Text: Principle of Instrumental Analysis, 7th Edition; Skoog, Holler & Crouch, 2018.
2. Safety Goggles: Goggles must be worn in laboratory at all times.
3. Laboratory Notebook: Permanent bound quadrille-ruled notebook.

OFFICE HOURS:

Monday, Thursday, Friday 10:00– 11:00; Tuesday 1:00-2:00; Wednesday 11:00-12:00 Feel free to come by any time that I am in my office. If you have difficulty finding a time to meet with me, make an appointment to see me.

ATTENDANCE:

Regular attendance is expected for all lectures and laboratories. If an absence results in a missed exam or lab, a Request For Excused Absence Form (attached to the end of the syllabus) must be submitted to the instructor. Absences are excused for illness, family emergencies and college activities. Absences due to college activities must be discussed with the instructor in advance and arrangements made for making up the missed work. All missed work must be made up within one week of the student's return to class.

EVALUATION:

Exam:	30% (2 exams)	<u>Letter Grade</u>	<u>Percentage</u>
Final Exam:	20%	A	92 – 100
Laboratory:	50%	A-	90 – 92
		B+	88 – 90
		B	82 – 88
		B-	80 – 82
		C+	77 – 80
		C	68 – 77
		C-	65 – 68
		D+	64 – 65
		D	61 – 64
		D-	60 – 61
		F	< 60

TENTATIVE EXAM DATES:

Exam 1: March 1
Exam 2: April 14
Final Exam: Wednesday May 3 at 8:00 -10:00

LECTURE:

Lectures will cover the theory and technique of spectrophotometric, electroanalytical, and chromatographic methods. Exams will be taken from information covered in the lectures, albs, and homework assignments.

LABORATORY:

The laboratory portion of the class will require more time outside of 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 a pre-lab discussion. In addition, the experiment assigned for a particular week may come well in advance of the discussion of that topic in lecture. It will be necessary, therefore, 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 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 schedule 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 upperclassmen 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, bear your name, the date, and the experiment title. All data must be recorded in the notebook using 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 obliterate the data (also do not use white out, tear out pages, recopy data, or tape in new pages). Draw a single line through the bad data. Include a note explaining why the data was in error.

The notebook is a record of your work as it is done. A running log of what you are doing in the lab should be kept in the notebook. The notebook should be kept in such a way so that the instructor can turn to any experiment and tell exactly what you did during an experiment. All data must include the appropriate units and be labeled to identify the data. All instrument parameters and recorder settings must be recorded in the notebook for each trial. All calculations, graphs, and table should 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 describing the reagents to be used. This should consist of a table listing the reagent's name, how the reagent is to be used in the experiment, any hazards associated with the reagent, and the proper disposal method in the lab. This is to be completed before the student begins the experiment.

LABORATORY REPORT:

These are formal reports and should be word-processed in a professional manner. Reports should be approximately five pages of text excluding the title page, figures, and tables. Each lab report should contain the following (label and identify each section of the report):

1. Title Page: Name, partner's name in parentheses, date, experiment title and abstract (short summary of the goal of the experiment, the technique used and the major results).
2. Theory: Both the theory essential to an understanding of the chemical principles on which the measurement is based 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 (2-3 pages).
3. Instrumentation: 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. The name and model of the instrument should also be included (1-2 pages).

4. **Methods and Materials:** Give a reference for where the procedure may be found. Include changes to the procedure and a description of the sampling method (a short paragraph).
6. **Data Presentation and Results:** 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 conclusion 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.
7. **Conclusions and Discussion:** The conclusion 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. 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 statistical analysis of your data should be included as well as a discussion of the sensitivity and detection limits of the instrument (1 page).
8. **Graphs:** Use proper labels for axes titles, and units. When spectra, chromatographs, or voltammograms are attached to a lab report, they should be labeled with a figure number and discussed in the text. 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 a table or as several plots overlaid on one page to show changes that occur.
9. **References:** Report in correct bibliographic style (minimum of 2 outside references). References should be properly cited in the theory and instrumentation sections.

Lab reports will be due one week from the scheduled date for the experiment. **LATE LAB REPORTS WILL RECEIVE A GRADE OF F.** The only exceptions will be for extreme circumstances such as major illness or family emergencies. Notebooks should be signed and evaluated by the instructor at the end of each period.

Lab Report Evaluation:	Notebook	5 points
	Format and Style	15
	Theory	20
	Instrumentation	20
	Data Presentation and Results	20
	Conclusions	15
	References	5

Lab Report Rewrites: You may rewrite and resubmit a maximum of 4 graded lab reports if you wish to improve the grade. *Both the old graded report and the new rewrite should be paper clipped together* and submitted within one week from the day the graded report is returned to you. Only one rewrite of each lab report will be allowed. Grades due to late reports cannot be made up by submitting a rewrite.

COURSE TOPICS

1. Signal to Noise Ratio
2. Spectroscopy
 - Major Instrument Components: Sources, Monochromators, Detectors
 - Quantum-Mechanical Model of Absorption, Emission and Fluorescence
 - Beer's Law
 - Atomic Spectroscopy: Emission, Absorption, ICP
 - Molecular Spectroscopy: UV-Vis; Fluorescence; FTIR; Raman
3. NMR: Theory of nuclear absorption; FTNMR

4. Mass Spectroscopy: Ion Sources
Magnetic Deflection, Quadropole; Time-of-Flight
5. Electrochemistry:
Potentiometry: pH electrodes, ion-selective electrodes, reference electrodes
Voltammetry: Cyclic voltammetry; polarography
6. Chromatography: theory of separation on a column
GC: instrument components; capillary vs. packed column
HPLC: instrument components
Partition, adsorption, ion-exchange and size exclusion

Millersville University Policies

ADA Program (Office of Learning Services) Americans With Disability Act | Millersville University (if you have a disability that requires accommodations under the Americans with Disabilities Act, please present your letter of accommodations and meet with me as soon as possible so that I can support your success in an informed manner. Accommodations cannot be granted retroactively. If you would like to know more about the Millersville University Office of Learning Services-please contact the office at 717-871- 5554)

- Academic Honesty Policy link [Governance Manual \(millersville.edu\)](#); for additional information please see the following: [What is Academic Integrity? | Millersville University](#)
- Attendance Policy link: [Class Attendance Policy | Millersville University](#)
- Inclusion Statement: [Millersville University Inclusion Statement | Millersville University](#)
- Land Acknowledgement: [Land Acknowledgement | Millersville University](#)
- Policy on Delays and Cancellations link [Policy on Delays & Cancellations | Millersville University](#)
- Preferred Name FAQs link [Preferred Name FAQs | Millersville University](#)
- Privacy Rights under FERPA link [Annual Notification of Student Rights Under FERPA | Millersville University](#)
- Student Conduct and Community Standards Handbook link [studentcodeofconduct.pdf \(millersville.edu\)](#)
- Title IX Reporting Requirements and the Faculty member: Millersville University is committed to maintaining a safe education environment for all students. In compliance with Title IX of the Education Amendments of 1972 and guidance from the Office for Civil Rights, the University requires faculty members to report incidents of sexual violence shared by students to the University's Title IX Coordinator. 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 sexual violence or any other abuse of a students who was, or is, a child (under 18 years of age) when the abuse allegedly occurred to the person. Information about Title IX, resources and reporting can be found at: [What is Title IX | Millersville University](#)

**LABORATORY SCHEDULE SPRING 2023
ANALYTICAL CHEMISTRY**

Group		I	II	III	IV	V
Jan	19	-----CHECK-IN ----Intro to Lab-----				
	26	1	2	3	4	5
Feb	2	2	3	4	5	1
	9	3	4	5	1	2
	16	4	5	1	2	3
	23	5	1	2	3	4
Mrch	2	-----SEM and XRF-----				
	9	-----BREAK-----				
	16	6	7	8	9	10
	23	7	8	9	10	6
	30	-----Lab Tour-----				
Apr	6	8	9	10	6	7
	13	9	10	6	7	8
	20	10	6	7	8	9
	27	-----POSTER PRESENTATIONS & Check out-----				

- 1 = Atomic Absorbance Spectroscopy - Flame
- 2 = Raman Spectroscopy
- 3 = Fluorimetry
- 4 = FTIR Infrared Spectroscopy
- 5 = UV-Visible Spectroscopy
- 6 = High Performance Liquid Chromatography
- 7 = Cyclic Voltammetry
- 8 = Ion Chromatography
- 9 = Gas Chromatography/Mass Spectroscopy
- 10 = Liquid Chromatography/Mass Spectrometry

Request for Excused Absence

Student Name:

Dates of Absence:

Reason for Absence (circle one): Illness, Family Emergency, University Activity

I request this absence be excused and that: (check all that apply)

_____ my final exam grade be substituted for the missed test.

_____ I be allowed to make-up the missed laboratory experiment. The lab must be made-up and the laboratory report submitted for grading within one week of returning to class.

Attach documentation to support the request for an excused absence.