Instrumental Analysis
Chemistry 3210 (4 credit hours)
Spring 2023
Location: Sturm 479

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Course Description: This is a second course in analytical chemistry for Chemistry majors and other advanced students with a Chemistry emphasis. The purpose of this class is to provide students with an overview of modern chemical instrumentation: what the devices are, how they work, what design features are important, and applications to chemical problems. The class will begin with a review of some fundamental issues of importance in all instrumental analytical chemistry, and will subsequently provide a brief overview of the major areas of spectroscopy, chromatography, and mass spectrometry.

Course Objectives: The student should complete this course having obtained an overview of modern instrumental analytical chemistry. The student should be familiar with modern chemical instrumentation and its underlying physical/chemical principles. The student should be capable of selecting appropriate techniques for solving specific problems in analytical chemistry, and should have an understanding of pertinent interferences, limitations, quality assurance practices, and procedures for evaluating the accuracy and precision of the resulting data. Additionally, the student should be able to re-create block diagrams of important instrumentation, and recall the importance of each component.

Sometimes the lectures will be somewhat general and it will be up to the student to fill in the gaps to truly understand the details. BOTTOM LINE: Pace yourself and continuously work from beginning to end. Even if you think a topic is easy, I recommend that you put in extra time to make sure you understand the details that may not be presented in lecture. This especially applies to the first part of the course as it is applicable to everything we do. Students are expected to put in 2-3 hrs outside of class per credit. The lecture section of CHM 3210 is a 3-hr course, so the class is designed such that ~ 6-9 hrs of your time outside of class is focused on this course.

Grading: To help assess the knowledge you have gained from Chemistry 3210, you will be evaluated based on your performance in several areas:

Homework: No graded homework. However, I strongly recommend reviewing questions from the book.

Quizzes (30 % of your grade): Short (~ 5-7 min) quizzes will be administered randomly throughout the quarter, but you can expect one on at least 15 of the class periods. Each quiz is generally on past lectures. **There will be at least 15 quizzes and only the top 12 will count towards your final grade**. If you take < 12 quizzes, then I will have to average in "zeroes." There are no make-ups for missed quizzes. However, if you know that you have an extended absence, please contact me – we can work out an alternate arrangement. The dropped quizzes allows for a reasonable amount of illness, bad hair days, missed alarm clocks, etc.

Exams (30 % [15 % each] of your grade): Two 50 min exams will be administered during class-time.

Final Exam (20 % of your grade): The Final Exam will be administered on Wednesday, June 7 at 8:00 am. The Exam is cumulative throughout the quarter. The Final Exam is not optional and the time can only be changed if you meet the University criteria for moving the Final Exam date. If you do qualify for this, then please let me know in the first week of the quarter.

Laboratory (20 % of your grade): One exception: It is important to note that **a failing grade in the lab will automatically correspond to a failing grade in the course**.

In person: You have one 3-hr lab section each week. The labs are only loosely coordinated with the lecture, so this will sometimes require significant preparation on your part! Be sure to give yourself plenty of time to familiarize yourself with the techniques and pre-lab assignments. **The lab cannot be completed until the pre-lab is complete!** To stay on good terms with your lab partners and your TA, make sure you take the time to do this. Note that some pre-lab assignments are more involved than just taking 10 min before lab to finish. There is a separate lab hand-out for each assignment – all of which are posted on Canvas.

Final Grades: This class is NOT based on a curve. Thus, you are not competing against each other. If everyone exceeds the required threshold, then everyone can earn an "A" in this course. The letter grades are detailed below.

A	> 92.99 %
A-	> 89.99 %
B+	> 86.99 %
B	> 82.99 %
B-	> 79.99 %
C+	> 73.99 %
C	> 68.99 %
C-	> 64.99 %
D+	> 61.99 %
D	> 57.99 %
D-	> 53.99 %
F	< 53.99 %

I reserve the right to make downward adjustments to this scale (i.e. adjustments in the direction of leniency). In no event will the actual scale used be adjusted upward from that described above.

Resources:

Textbook: Quantitative Chemical Analysis (QCA), 9th Ed. Daniel C. Harris is required. Other instrumental textbooks will also work (like Skoog), if you happen to have one from somewhere else.

Me: Email me anytime with questions about the course or the material. If my office hours do not fit your schedule, then I can easily make myself available. My email address is brian.majestic@du.edu. Just email me.

Lecture Notes and Powerpoint Slides: Most lectures will be a Powerpoint presentation. These will be posted on Canvas. Lecture will move at a relatively rapid pace, so it will benefit you to download these and have them handy prior to lecture.

Each Other: You are more than welcome to collaborate with each other in groups. However, each paper turned in **must be your own work**. Please review the **DU Code of Academic Honesty** as you will be strictly held to this. It is also encouraged that you form study groups. As in the "real-world," Chemistry 3210 does not need to be an individual experience. Most of the time, very little gets done without the help of others and team-work is a great skill to learn.

Supplementary Resources: In addition to the textbook and class presentations, you will have the opportunity (and expectation) to consult other resources. These will consist of websites (instrumentation manufacturers, laboratories, trade associations, government sites, user groups, and list-servers), electronically available journals, and paper-based journals. Most journal articles are available in electronic format and may be printed and/or stored in journal publication format as *.pdf. Two examples of where to look are as follows: **A) the ACS journals (pubs.acs.org) are available from any DU-based URL; and B) many other journals are available free of charge from www.sciencedirect.com, a service which the DU library subscribes to.** Sciencedirect is available without a personal account from any campus-based URL; however, you can access it from off-campus through the DU library's website with proxy identification. Databases such as Google Scholar (scholar.google.com) can be a good place to start with a keyword search for material on a specific subject or author.

Academic Integrity: You will notice that, in the lectures, I credit any sources which provide images, content, or text. In CHEM 3210, we take cases of academic dishonesty very seriously. Note that academic dishonesty is not limited to plagiarism or copying another student's work. It also includes behaviors such as giving false reasons for missing a lab or exam or hiding the fact that other students are knowingly practicing academic dishonesty. All students are expected to abide by the University of Denver Honor Code. These expectations include the application of academic integrity and honesty in your class participation and assignments.

The DU Honor Code is found here:

http://www.du.edu/studentlife/advising/fsem_handbook_docs/UniversityofDenverHonorCode.pdf

Lecture and Testing Accommodations: I will make every effort to accommodate students diagnosed with a learning disability. I will do this in complete confidence. I do, however, request that any student requiring these accommodations inform me the first week of class. For further information, please see the University Disability Services's website: http://www.du.edu/disability/dsp/index.html.

Course Outline (This is VERY rough and should only be used as a guide - subject to change)

Material for Exam 01:

Introduction: What's in a data point?

Quantitative vs qualitative measurements
Basic Instrumental Components
Sample considerations
Analyte considerations
Review of basic statistics
Precision, bias, and accuracy
Errors in chemical analysis: random and systematic errors
Propagation of uncertainty
Proper use of significant figures
Calibration processes and practices: external calibration, method of standard additions, internal standardization
S/N ratio
Detection limits
Types of noise and S/N improvement

Intro to Spectroscopy

The electromagnetic spectrum
Energy, frequency, wavelength, wavenumbers
Properties of light
Energy level diagrams
Types of atomic spectral measurements: emission, absorption, fluorescence
Spectrometric processes: absorption, emission, fluorescence, phosphorescence
Spectral purity and spectral resolution
Monochromators and polychromators
Bandwidth, slit width, resolving power, and resolution
Emission spectrometry and absorption spectrometry
Beer's Law and its limitations
Multicomponent analysis
Total internal reflection and fiber optics
Attenuated total reflection and diffuse reflectance
Diffraction and diffraction gratings
The monochromator
Light sources for the UV, visible, and IR
Light detectors

Spectroscopy for Elemental Analysis

Atomic linewidths
Atom cells: the flame, ETV furnace as atom cells
Sample introduction processes
Properties of flames
Atomization in flames
Flame hardware
Electrothermal atomization and hardware
Specialized atomization techniques
Instrumentation for AAS
Interferences, problems in AAS; background correction methods
Techniques and applications of AAS
Atomic fluorescence spectroscopy
Advantages and disadvantages of high-energy plasma sources
The inductively coupled plasma
The three-electrode DC plasma jet
Microwave induced plasmas
DC and RF glow discharges
Instrumentation for atomic emission spectroscopy
Techniques and applications of ICP spectroscopy
Arc, spark, and flame emission sources
Nature of X-Rays
Sources
Absorption
Monochromators, filters, and detectors
X-Ray fluorescence instrumentation
XRF techniques and applications
X-Ray diffraction instruments and applications
Electron microprobes and X-ray microanalysis

Vibrational Spectroscopy

Molecular basis for IR, Raman
IR sources and detectors
FTIR spectrometers
Sample presentation methods
Applications to structural elucidation
Quantitative analysis via IR absorption spectrophotometry
The near-IR region: instrumentation and applications
Raman scattering and Raman spectra
Dispersive and FT Raman spectrometers
Applications of Raman spectroscopy

Material for Exam 02:

Mass spectrometry

Overview, purposes of MS
Ion sources
Ion detectors
Mass analyzers
Magnetic sectors

Quadrupoles
Time-of-flight
FT/ICR, ion traps
Orbitraps
Applications: structural elucidation
Interpretation of electron impact mass spectra of organic compounds

Introduction to chromatographic separations

Purposes; classification of methods
Examining the chromatogram
Equilibria - the basis for retention
Partitioning, resolution, dispersion
Effect of mobile phase flowrate
Qualitative identification via chromatography
Quantitative analysis via chromatography
Zone broadening: the van Deemter Equation and HETP

Liquid chromatography

Types of separation methods
Role of chemical variables
Effects of temperature
Particle size, column size, pressure drop, and HETP
Instrumentation
Detection methods
LC-MS systems
Planar chromatography
Preparative liquid chromatography
Applications

Gas chromatography

Applicability
Retention volumes and partition coefficients
Instrumentation
Types of columns
Sample injection methods
Detection methods
Qualitative identification and retention indices
GC-MS systems
Applications of gas chromatography
Sample preparation for gas chromatography