

Virtual Instrumental Analysis  
Chemistry 3210 (4 credit hours)  
Spring 2021  
Location: anywhere and everywhere

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Office Hours: Will announce online. Office hours will take place via Zoom or by Phone. I am open to whatever is more convenient to you.  
The Office hour url is:  
<https://udenver.zoom.us/my/brianmajestic>

**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, time permitting, electrochemistry.

**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.

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.

**Course Format:** Lectures will take place synchronously, via Zoom. The links will be posted in the course Canvas site. Additionally, the lectures will be recorded so that they can be accessed (in Canvas) for the duration of the quarter.

**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 min) quizzes will be administered randomly throughout the quarter, but you can expect one on at least 15 of the class periods. Each quiz may be on past lectures or on reading for the day's lecture. **There will be ~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." **These quizzes will be timed quizzes on Canvas and will be available for 8-10 min only, beginning at 9:00 Colorado time.** There are no make-ups for missed quizzes. The dropped quizzes allows for a reasonable amount of illness, bad hair days, missed alarm clocks, etc. If you are not able to make this timing, then please let me know so we can work something else out.

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

**Final Exam (20 % of your grade):** The Final Exam will be administered on Monday, June 7 at 12:01 am, and will be available for 48 hrs. 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 month of the quarter.

**Laboratory (20 % of your grade):** This quarter, some of you are working in the lab and some are working virtual. Either way, 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 partner 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.

**Virtual:** There will be 4 laboratory experiments. These will be a combination of providing data and running instrument simulations. 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. 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), 9<sup>th</sup> 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](mailto: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, along with the lecture videos. 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](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

Derivative spectrometry

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  
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