Instrumental Analysis

Chemistry 3210 (4 credit hours)

Spring 2014

MWF 9:00 – 9:50 am

Boettcher Center West 124

Instructor: Dr. Brian Majestic

Email: brian.majestic@du.edu

Office: SGM 151

Telephone: (office) (303) 871-2986

 (cell) (480) 204-9515 (please only call until 8 pm!)

Office Hours: MW 8-9 am, Tr 10-11 or make an appt during any scheduled lab time

**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.

**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 18 of the class periods. Each quiz may be on past lectures or on reading for the day’s lecture. **There will be ~18 quizzes and only the top 14 will count towards your final grade**. If you take < 14 quizzes, then I will have to average in “zeroes.” There are no make-ups for missed quizzes (including illness, family emergencies, and institutional excuses). The dropped quizzes allows for a reasonable amount of illness, bad hair days, missed alarm clocks, etc. Even with circumstances beyond your control, it is likely that if you miss 2 weeks of classes, then you have much larger issues on your plate and will likely need to drop the course.

**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 4 at 8:00 am and 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):** 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 Blackboard. It is important to note that **a failing grade in the lab will automatically correspond to a failing grade in the course**.

**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), 8th Ed. Daniel C. Harris is required. This is the same textbook from Equilibrium Systems for most of you. Other instrumental textbooks will also work, 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 **prior to class**. 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>.

**Important Dates:**

* Mon, March 24: Classes begin
* Sun, May 4: Last day to drop w/out approval
* Mon, May 26: Memorial Day (no class)
* Fri, May 30: Last day of class
* Wed, June 4: Final Exam (8:00 – 9:50 am)

**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

**Capillary electrophoresis** (time permitting)

Objectives of CE

Principles of ion migration

The electroosmotic flow

Separation methods

Detection methods

CE-MS systems

Applications