CHEM 349 Instrumental Analysis
Syllabus

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Office: HN1302/1303
Office Hours: M, Th, 10-11 or by appt

The course will concentrate on various aspects of instrumental methods used in chemistry and biochemistry. Classroom discussions and hands-on experiments that characterize and analyze chemical and biochemical samples will give students the theoretical underpinnings and practical experience with the use of a vast array of modern instrumentation: UV-vis, IR, Fluorescence, MS, GC-MS, atomic force microscopy, ICP, X-ray, NMR, TEM, and more. NMR will be covered in slightly more detail than the other methods, and many examples of theory will be illustrated using NMR. Students will gain understanding in both the theoretical basis and the mechanical and electrical design of each type of instrumentation. The factors that influence the different analytical method’s capabilities and limitations will be discussed.

This is a laboratory course! In addition to ro er laboratory attire including eye protection, ord rocessing, s read sheet, and critical thinking skills are required.

Class: 4 hours, twice a week: 1 hour lecture and 3 hours of lab

Evaluations:

<table>
<thead>
<tr>
<th>Evaluation</th>
<th>Points</th>
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<tbody>
<tr>
<td>Lab notebook (checked weekly)</td>
<td>50</td>
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<tr>
<td>Class assignments</td>
<td>100</td>
</tr>
<tr>
<td>3 lab reports (50 points each)</td>
<td>150</td>
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<tr>
<td>5 Quizzes (10 points each)</td>
<td>50</td>
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<tr>
<td>Oral Presentation</td>
<td>50</td>
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<tr>
<td>Midterm Exam</td>
<td>100</td>
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<tr>
<td>Final Exam</td>
<td>100</td>
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<tr>
<td>Total</td>
<td>600 points</td>
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Cell phones are not allowed in class. You may not use your phone calculator.
Bathroom breaks will not be allowed during quizzes or exams. Don't ask.

Blackboard: Lectures and other materials posted, email communications via Blackboard
**Required Textbook:**

(A copy is available on reserve at the Hunter library.)

- A large portion of the course lectures will follow the “Skoog” book. Listed below are several other textbooks available that cover much of the same material.
- Texts and references focused on the individual techniques, particularly NMR, will be made available through handouts and/or the Hunter Library electronic resources.

**OTHER GENERAL INSTRUMENTAL ANALYSIS TEXTS:**

*Undergraduate Instrumental Analysis*, Sixth Edition.  
Publisher: CRC Press; (2004)  
**ISBN-10:** 0824753593; **ISBN-13:** 978-0824753597, $45-60 paperback (Kindle ed. available)

*Chemical Analysis: Modern Instrumentation Methods and Techniques,*  
Publisher: Wiley; 2 edition (2007)  
**ISBN-10:** 0470859032; **ISBN-13:** 978-0470859032 $45-60 paperback

**NMR RESOURCES:**

*Rule, Gordon* "Fundamentals of Protein NMR Spectroscopy" (2006). pdf download at library.hunter.cuny.edu


Basic of NMR website by Joseph Hornak at RIT. [http://www.cis.rit.edu/htbooks/nmr/inside.htm](http://www.cis.rit.edu/htbooks/nmr/inside.htm)

NMR relaxation mechanisms by Hans Reich at [https://www.chem.wisc.edu/areas/reich/nmr/08-tech-01-relax.htm](https://www.chem.wisc.edu/areas/reich/nmr/08-tech-01-relax.htm)

**X-ray Crystallography:**


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**Required Materials**

- lab notebook, goggles, scientific calculator.
- These must be in your possession during all class periods.
- Failure to arrive with these items will result in zero points for the day.
- And students may be asked to leave the laboratory if they do not have these items.
- Proper Laboratory attire is mandatory. A lab coat is recommended.

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### COURSE SCHEDULE SPRING 2018

Laboratory Schedule is **Tentative**. Lecture and Lab topic may not match due to instrument availability.

<table>
<thead>
<tr>
<th>Month</th>
<th>Mon</th>
<th>Thu</th>
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<tbody>
<tr>
<td><strong>Mar 2018</strong></td>
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</tr>
</tbody>
</table>
| 29 | Data processing/Analysis, Figures of Merit  
Reading: Skoog Ch 1-2 | 1 | Electricity, Oscillation, Phase, Noise  
Reading: Skoog Ch 2, Ch 5 |
| 5 | Spectroscopy  
Reading: Skoog Ch 5-6 | 9 | UV-vis  
Reading: Skoog Ch 7 & 13 |
| **12 No Class**  
(College closed - Lincoln's B-day) | | 15 | UV-vis cont.  
Reading: Skoog Ch 7 & 13 |
| **20 Tuesday Class** - College closed Monday | Fluorescence - Reading: Skoog Ch 8 & 15 | 22 | Fluorescence  
Reading: Skoog Ch 8 & 15 |
| 26 | FTIR  
Reading: Skoog Ch 7 & 16 | 1 | NMR: Introduction  
Reading: Skoog Ch 19, Rule Ch 1 and 2 |
| 5 | NMR: finish intro, start vector model  
Reading: Rule Ch 1 | 9 | NMR: finish vector model  
Reading: Handout |
| 12 **Tentative Midterm date** | | 15 | NMR Coupling  
Reading: Rule Ch 7 |
| 19 | Heteronuclear NMR  
Reading: Rule 10 | 22 | NMR |
| 26 | NMR Relaxation *(abstract due!)*  
Reading: Rule 19/Handout, Reich web page | 29 | NMR |
| **Apr 2018** | | **SPRING BREAK** |
| 2 | | 5 | **SPRING BREAK** |
| 9 | NMR - gradients, | 12 | Multidimensional NMR  
Reading: Rule 9 |
| 16 | Chromatography  
Reading: Skoog 26 | 19 | HPLC  
Reading: Skoog 28 |
| 23 | GC-MS  
Reading: Skoog 27 | 26 | MS  
Reading: Skoog 11 & 20 |
| 30 | X-ray diffraction  
Reading: Handout | 3 | ICP  
Reading: Skoog 8, 9 & 10 |
| **May 2018** | | **STUDENT PRESENTATIONS** |
| 7 | TEM  
Reading: Skoog 21 | 10 |  
| 14 | **STUDENT PRESENTATIONS** | 17 | Reading Day |
| 21 | | 24 | **FINALS** |
Outcomes for Instrumental Analysis 349:

This course will introduce the student to the theories and principles of instrumental methods of chemical analysis. Upon successful completion of this course, a student should be able to:

- distinguish between analog and digital signals.
- understand and apply the concepts of signal, noise, sensitivity, detection limit, resolution, dynamic range, and selectivity to an instrumental analysis.
- calibrate an instrumental method.
- be knowledgeable about electromagnetic sources and detection systems used in instrumental methods of chemical analysis.
- choose a suitable instrumental method of analysis for a given analyte and matrix.
- understand the underlying principles of basic instrumental methods such as atomic absorbance and emission; IR, UV, visible, and fluorescence spectroscopy; mass spectrometry; nuclear magnetic resonance; gas and liquid chromatography.

Evaluations: Design and assay an unknown by diverse instrumental methods. Describe an instrumental method, including dynamic range, and appropriate sample preparation.

Assessments of evaluations and other assessments: via 1-on-1 interactions with students and their ability to work independently on a spectroscopic evaluation.

Lab Notebooks

You are to use a laboratory notebook to record data and observations. The development of good notebook "habits" (keeping a well organized, complete record of your lab work) will be important to your success in this course. A day-to-day written record of laboratory work is essential in research. The notebook is needed so that you or others may repeat your work successfully (or avoid making the same mistakes). A careful record enables you to make intelligent corrections in the procedure to increase the chance of success in future work. The notebook can also provide a permanent record of instructions you received, which you can refer to later if necessary.

Guidelines for the Laboratory Notebook
- Bound.
- Two-three pages at the front are dedicated to a table of contents.
- Each page is dated and numbered.
- Written in ink as experimental work proceeds (not the next day).
- Incorrect entries are crossed out with a single line.
- Blank pages have a single diagonal line indicating that they are intentionally left blank.
- Contains drawings of experimental apparatus, descriptions of how the work was actually carried out (when changes are made in the lab manual procedure, you should indicate
what you did, not what the lab manual said), and experimental observations (color changes, temperatures, measurements, problems encountered). Includes information about reagents (FW, density, formula, manufacturer, grade) and instrumentation (Make, Model, parameters, solvents, columns, sample cells, flow rates, wavelength ranges).

- Includes results, equations and calculations.
- Includes file path and data file names.
- Spectra, chromatograms, calibration curves, etc are taped or stapled to pages in the notebook.
- Includes explanations, interpretations, questions that arise, future plans.

LABORATORY REPORTS:

A lab report will be submitted for each of the experimental modules. Requirements for individual reports will vary and will be specified on handouts. Regardless of other requirements, each report should have a cover page and be written using a word processor and other relevant plotting software (e.g., Excel).

The writing and style of presentation should be of high quality, and be in the following format.

**Cover page** listing the title of the experiment, the name of the students, and the date

**Abstract:** (ca. 100 words)

**Introduction:** (1-2 paragraphs with the references/texts you used to prepare for the experiment) Experimental (how you did the experiment and why)

**Results:** the data in tabular and/or plotted

**Discussion and Conclusions:** (try to keep it less than a page or two) answering the specific questions in the handout, what were the problems, what could have been done better.

Please make sure the numbers are formatted correctly, and you use the above section titles. Each figure, graph, and table should be numbered. The references/bibliographys should be as found in an American Chemical Society Journal

STUDENT LECTURE

The last 2 lectures of the semester have been reserved for student lectures. Each student will select and research an instrumental technique **not** presented in this course. This will allow you do delve into a subject of your own interests. You will be allowed twenty minutes in which to present the theory, instrumental details and applications. You can use the book for a start, but you should also have ca. 10 literature sources (two of which may be websites). This is a Power Point presentation. You should prepare 1-page handouts. The final exam may include material(s) from these presentations, so you should prepare one question for potential use in the exam.

Your presentation will be evaluated for content and oral communication. See syllabus for deadlines.
Hunter College require statements for syllabi

1. Academic Integrity Statement: “Hunter College regards acts of academic dishonesty (e.g., plagiarism, cheating on examinations, obtaining unfair advantage, and falsification of records and official documents) as serious offenses against the values of intellectual honesty. The College is committed to enforcing the CUNY Policy on Academic Integrity and will pursue cases of academic dishonesty according to the Hunter College Academic Integrity Procedures.”

2. ADA Statement: “In compliance with the ADA and with Section 504 of the Rehabilitation Act, Hunter College is committed to ensuring educational access and accommodations for all its registered students. Hunter College's students with disabilities and medical conditions are encouraged to register with the Office of AccessABILITY for assistance and accommodation. For information and appointment contact the Office of AccessABILITY located in Room E1214 or call (212) 772-4857 /or VRS (646) 755-3129.”

3. Hunter College Policy on Sexual Misconduct: “In compliance with the CUNY Policy on Sexual Misconduct, Hunter College reaffirms the prohibition of any sexual misconduct, which includes sexual violence, sexual harassment, and gender-based harassment retaliation against students, employees, or visitors, as well as certain intimate relationships. Students who have experienced any form of sexual violence on or off campus (including CUNY-sponsored trips and events) are entitled to the rights outlined in the Bill of Rights for Hunter College.

   a. Sexual Violence: Students are strongly encouraged to immediately report the incident by calling 911, contacting NYPD Special Victims Division Hotline (646-610-7272) or their local police precinct, or contacting the College's Public Safety Office (212-772-4444).

   b. All Other Forms of Sexual Misconduct: Students are also encouraged to contact the College's Title IX Campus Coordinator, Dean John Rose (jtrose@hunter.cuny.edu or 212-650-3262) or Colleen Barry (colleen.barry@hunter.cuny.edu or 212-772-4534) and seek complimentary services through the Counseling and Wellness Services Office, Hunter East 1123.

CUNY Policy on Sexual Misconduct Link:
http://www.cuny.edu/about/administration/offices/la/Policy-on-Sexual-Misconduct-12-1-14-with-links.pdf"