Chromatography & Mass Spectrometry, Chem 543 7033 (3 credits)

Instructor: Alena Kubatova, Abbott Hall (301), 777-0348  
E-mail: alena.kubatova@UND.edu  
Lectures: Tuesdays 9:30-10:45 AM, Thursdays 10-11:45 AM  
Laboratory periods: The lab times are scheduled by appointment, the lab tasks are scheduled to be completed within a total of ca. 22 h.  
Office hours: We 2-3 PM AH 301, other times by appointment

Chromatography and Mass Spectrometry are considered to be most frequently used analytical techniques. However, as a number of users increases the quality and understanding often declines. This course will cover advanced theory as well as the practical training in chromatographic techniques including also fundamentals of instrumentation in mass spectrometric detection. The course (Chem 543, 3 credits) is the follow-up of undergraduate (Chem 443) ensuring deeper understanding of theoretical background and topics in general. Although basics will be covered as part of discussions student are expected to have a basic knowledge of chromatography and mass spectrometry prior to the course.

The class is run in a flipped classroom format. That is, students will be expected to prepare using the online lecture and reading materials as well as some texts available in the library for each ‘lecture session’. They will be required to bring solved take-home quizzes to lectures, so the answers will be discussed in class. Further, students will present two presentations based on the articles published in LC GC Chromatography.

Students are expected to work in collaborative environment with undergraduate students. The first assignment will focus on helping and providing feedback to undergraduate students with the literature review assignment. The teaching experience is expected to enhance graduate students awareness in common mistakes in writing and thus helping them to improve their own writing.

Since the analytical chemistry is a practical science requiring hands on experience, there will also be two lab assignments, in which students will individually perform practical measurements and demonstrate their understanding on applied examples. These lab assignments will prepare students to perform measurements independently from the start-up of the instrument to the optimization of basic operation parameters.

Both laboratory assignments are part of departmental training and can be found under tab instrumental training. First assignment prepares user to work with gas chromatograph focusing on basic operation of GC. The second laboratory targets operation of high resolution mass spectrometer.

Topics Covered
- Quantitation and data processing
- Fundamental concepts and theory of chromatography
- Gas chromatography: Injectors, detectors, column selection
- Sample preparation
- Mass spectrometry
  - Ionization sources
  - Mass analyzers
  - Basics of El interpretation
- Liquid chromatography
• Hyphenated techniques, i.e., GC/MS, LC/MS
• Ion chromatography, size exclusion chromatography
• Bio applications

**REQUIRED MATERIALS**
A bound laboratory notebook and a calculator

**SAFETY REQUIREMENT**
Prior to any lab work, students will be required to complete the online Chemistry Department Safety training. Failure to complete the safety training will prevent students from entering the lab, so they cannot complete the assignments.

**WEBSITE SUPPORT**
**Blackboard:** Students are expected to check Blackboard online regularly for announcements and submit all their work through the blackboard site

**Chromacademy:** [http://www.chromacademy.com](http://www.chromacademy.com) This website is essential to follow the required lecture materials. You will need to register on the Chromacademy website by selecting a tab with free University sponsorship (do NOT select LITE version on Free membership!!)

Other useful sites:
- [http://chemwiki.ucdavis.edu/Analytical_Chemistry](http://chemwiki.ucdavis.edu/Analytical_Chemistry)

**RECOMMENDED REFERENCE MATERIALS**
The following materials will be used as reference throughout the course in addition to pertinent peer reviewed papers.

1. Quantitative Chemical Analysis by Daniel Harris, Freeman [http://bcs.whfreeman.com/qca8e](http://bcs.whfreeman.com/qca8e)
**TENTATIVE SYLLABUS CHEM 543**

**FALL 2016**

**PAPER REVIEW**

As 1st assignment graduate students will work with several undergraduate students and provide feedback on their peer reviewed research papers. This will be carried out in class and monitored through the blackboard, where each graduate students will provide feedback to 5-6 undergraduates. Graduate students are expected to provide feedback within three days after the submission of the papers. The participation on this assignment will be part of a grading score.

**PRESENTATIONS**

Students are expected to signup and present two 15 min PowerPoint presentations. The topics will be posted in the assignment section on the blackboard. The electronic version of presentation, electronic links to papers presented should be submitted to the instructor by 4 PM a day prior to the day of presentation. **Failure to signup (deadline are specified in timeline below), present within specified dates, and providing electronic materials on time will results in loss of 5 points per incident.**

**Evaluation of presentation**

Presentation should include:

- reference to the paper presented (title page)
- the reason for presentation
- problem/material discussed in the article
- basics of analytical methods used including introduction to these methods even if not covered in the article
- factors affecting those methods
- conclusive optimal conditions – their advantages and disadvantages
- critical evaluation of the method by student

The presentation should be presented in a clear comprehensive manner so other students can learn from it. The questions regarding the presentation may appear in the exams. Each student must show understanding the material covered. The presentation will be posted on the blackboard in the form of PDF file.

**LABORATORY ASSIGNMENTS**

The laboratory assignments will cover two topics:

1) Beyond user - advanced use of GC

2) Optimization of electrospray conditions with high resolution mass spectrometry

The goal of these labs is to develop background in application of chromatographic - mass spectrometric methods. So, students not only learn to operate instrumentation, but they will be able to start from "scratch" that is starting the instruments and performing initial setup. The labs will mimic research investigation of optimal analytical conditions and should be reported in the form of paper for peer reviewed publication.

Labs will be performed upon completion of the safety training by students working individually

The lab reports will consist of the MS Word and MS Excel files and must be submitted by specified deadline through Blackboard. The detailed instruction for the labs are available on the website under instruments policies folder.

1st Lab GC training [http://arts-sciences.und.edu/chemistry/kubatova-research-group/gc_training02.cfm](http://arts-sciences.und.edu/chemistry/kubatova-research-group/gc_training02.cfm)

2nd Lab ESI MS TOF training [http://arts-sciences.und.edu/chemistry/kubatova-research-group/ms_training.cfm](http://arts-sciences.und.edu/chemistry/kubatova-research-group/ms_training.cfm)
**Grading**

The grading will be based on the percentage distribution and points for individual assignments specified below.

**Letter Grades:** A (≥90%), B (89-80%), C (79-70%), D (69-55%), F (<55%)

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<thead>
<tr>
<th></th>
<th>Number of activities</th>
<th>Points per assignment</th>
<th>Total points possible</th>
<th>%</th>
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<tbody>
<tr>
<td>Preparation/participation</td>
<td>16</td>
<td>3</td>
<td>48</td>
<td>8</td>
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<tr>
<td>Paper Review Feedback</td>
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<td>20</td>
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<td>Lab assignments/reports</td>
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<td>Presentations</td>
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<td>Midterm Exam</td>
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<td>21</td>
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<tr>
<td>Final Exam</td>
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<td>250</td>
<td>35</td>
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<td>Week</td>
<td>Date</td>
<td>Topic</td>
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<td>Section</td>
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<tr>
<td>1</td>
<td>23-Aug</td>
<td>Intro to class</td>
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<td>2</td>
<td>30-Aug</td>
<td>Data handling and quantitation</td>
<td>Quant Chem Analysis by Harris</td>
<td>Calibration: external and internal standards, limits of detection</td>
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<td>6-Sep</td>
<td>Working with Scifinder</td>
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<td>4</td>
<td>13-Sep</td>
<td>GC intro</td>
<td>Chromacademy</td>
<td>GC Introduction/Chromatographic Parameters and Band Broadening</td>
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<td>20-Sep</td>
<td>GC Injection</td>
<td>GC Theory of Instrum. Rate theory</td>
<td>Sample Introduction</td>
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<td>22-Sep</td>
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<td>Materials Provided by Instructor</td>
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<td>27-Sep</td>
<td>GC column selection</td>
<td>GC Theory of Instrum.</td>
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<td>29-Sep</td>
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<td>Gas supply and Pressure control</td>
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<td>4-Oct</td>
<td>GC detectors</td>
<td>GC Theory of Instrum.</td>
<td>GC detectors</td>
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<td>11-Oct</td>
<td>Midterm Exam</td>
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<td>9</td>
<td>18-Oct</td>
<td>2D GC Sample preparation</td>
<td>Multidimensional GC Sample Preparation</td>
<td>Multidimensional Chromatography SPE overview &amp; mechanisms</td>
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<td>25-Oct</td>
<td>GC-MS</td>
<td>MS</td>
<td>Fundamental GC-MS – Introduction E-learning mod.</td>
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<td>1-Nov</td>
<td>LC-MS</td>
<td>MS</td>
<td>Fundamental LC-MS – Intro, 10 Ways to Break Your LC-MS.</td>
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<td>LC-MS</td>
<td>LC-MS</td>
<td>Materials Provided by Instructor &amp; Analysis of biomolecules</td>
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<td>8-Nov</td>
<td>MS instruments &amp; interpretation</td>
<td>MS</td>
<td>MS interpretation – General strategies</td>
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<td>12</td>
<td>15-Nov</td>
<td>LC Introduction &amp; Instrumentation</td>
<td>LC</td>
<td>Theory of HPLC: Introduction &amp; Reverse phase</td>
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<tr>
<td>13</td>
<td>22-Nov</td>
<td>LC Separation</td>
<td>LC</td>
<td>Theory of HPLC: Normal phase &amp; Gradient</td>
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<td>14</td>
<td>29-Nov</td>
<td>LC separation</td>
<td>LC</td>
<td>Theory of HPLC: Ion chromatography</td>
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<td>15</td>
<td>6-Dec</td>
<td>LC detectors</td>
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<td>Instrumentation of HPLC: Detectors</td>
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<td>15</td>
<td>12-Dec</td>
<td>Final Exam</td>
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DISABILITY STATEMENT

If you need accommodations in this course because of a disability, if you have emergency medical information to share, or if you need special arrangements in case the building must be evacuated, please make an appointment to discuss these issues with your instructor soon as possible.

ACADEMIC INTEGRITY

Students are expected to display academic honesty as defined in the UND Code of Student Life http://und.edu/student-affairs/code-of-student-life/index.cfm. Academic dishonesty will result in a loss of all points for the graded material on which the transgression occurred. A second violation will result in a failing grade for the course.

NON-DISCRIMINATION STATEMENT

As part of its commitment to providing an educational environment free from discrimination, UND complies with Title IX of the Education Amendments, which prohibits discrimination and harassment based upon sex in an institution’s education programs and activities. Title IX prohibits sexual harassment, including sexual violence, of students at UND-sponsored activities and programs whether occurring on-campus or off-campus. Title IX also protects third-parties, such as visiting student athletes, from sexual harassment or violence in UND’s programs and activities and protects employees from sexual harassment and discrimination. Prohibited harassment includes acts of verbal, nonverbal or physical aggression, intimidation or hostility based on sex, even if those acts do not involve conduct of a sexual nature; sex-based harassment by those of the same sex; and discriminatory sex stereotyping. UND will take prompt action to investigate and resolve reports of sexual harassment or sexual violence in accordance with Title IX. UND’s Title IX coordinator is Donna Smith, Director of Equal Employment Opportunity/Affirmative Action, 401 Twamley Hall, 264 Centennial Drive Stop 7097, Grand Forks, ND 58202-7097, 701-777-4171, donna.smith@und.edu. Retaliation against any person who initiates an inquiry or complaint or participates in the investigation of a complaint is prohibited. Such conduct will be further cause for disciplinary action.

Brief Information about Reporting

Any student who has been impacted by sexual violence (sexual assault, domestic violence, dating violence, stalking) or gender-based misconduct is encouraged to report and seek appropriate resources on campus. Please contact the Title IX Coordinator (Donna Smith, donna.smith@und.edu or 701-777-4171) to discuss your options. To view the policy and additional resources, please visit: http://und.edu/finance-operations/university-police/sexual-violence-programs.pdf.

How to seek help when in distress: We know that while college is a wonderful time for most students, some students may struggle. You may experience students in distress on campus, in your classroom, in your home, and within residence halls. Distressed students may initially seek assistance from faculty, staff members, their parents, and other students. In addition to the support we can provide to each other, there are also professional support services available to students on campus through the Dean of Students and University Counseling Center. Both staffs are available to consult with you about getting help or providing a friend with the help that he or she may need. For more additional information, please go to http://und.edu/und-cares/.