Syllabus for Chemistry 6910
Special Topics: GC-MS, LC-MS and Fluorescence
Spring Semester, 2004

Instructors:

Dr. Haiying Liu
402E ChemSci
Phone: (906) 487-3451
E-mail: hyliu@mtu.edu

Course Schedule:

Lecture: Tuesday 9:00 – 12:00 AM, ChemSci 404
Laboratory: Section 1: TR, 9:00 - 12:00 AM

Office Hours:

Mondays and Thursdays 11:00 am to 12:00 pm, Room 402E Chemistry Building.
Please send me an e-mail to schedule appointments at other times.

Course objectives:

1. To learn basic principles governing chromatographic separation techniques.
2. To develop basic skills in operating chromatographic equipment.
3. To learn to apply basic principles in order to develop a chromatographic method to achieve a particular objective.
4. To learn basic principles of fluorescence of fluorescence spectroscopy for sensing and biosensing applications

Course description:
1. HPLC and GC

Review of chromatographic principles; instrumentation; mobile phase preparation; column selection; detector selection; sample preparation (liquid-liquid & solid phase); elution survey; separation optimization; quantitative analysis;

2. Mass Spectrometry

Mainstream analytical mass spectrometric principles and methods: Introduction to mass spectrometry; Instrumentation, Mass Spectrometry with volatile compounds - GC/MS; Mass spectrometry with involatile compounds including biomolecules - LC/MS and MALDI; Quantitative mass spectrometry, Tandem mass spectrometry (MS/MS).

3. Mass Spectrometry Interfaced with LC and GC

Applications of LC-MS and GC-MS; problems associated with interfacing; LC-MS (moving belt, direct-liquid, thermospray); GC-MS (packed vs. capillary columns, jet separator, semipermeable separator, effusion separator); tandem mass spec.

4. Fluorescence

Introduction to fluorescence; Instrumentation for fluorescence spectroscopy; fluorophores; quenching of fluorescence; Fluorescence sensing.

Topics:

Principles of Fluorescence Spectroscopy

Introduction to fluorescence
Instrumentation for fluorescence spectroscopy
Fluorophores
Quenching of fluorescence
Fluorescence sensing
DNA technology

General Introduction to Chromatographic Separation

A general description of chromatography
Migration rates of solutes
Zone broadening and column efficiency
Summary of important relationships for chromatography
Applications of chromatography
Instruments for gas-liquid chromatography

Gas Chromatography
Principles of gas-liquid chromatography
Gas chromatographic columns and stationary phases
Applications of chromatography

High-Performance Liquid Chromatography

Column efficiency in liquid chromatography
Instruments for liquid chromatography
Partition chromatography
Adsorption chromatography
Ion-exchange chromatography
Size-exclusion chromatography

Mass Spectrometry - Analysis of Small Molecules

Brief History/Overview
Hardware
Sample Introduction
Ion Sources
Sector Instruments and Quadrupoles
Detectors
Resolution
Ions Formed in MS
Electron Impact Ionization
Chemical Ionization
Isotopes & Isotope Calculators
Structural Clues
Fragmentation Chemistry

Mass Spectrometry - Analysis of Large Molecules

Desorption Ionization Methods
Fast Atom Bombardment with Sector Instruments
Electrospray Ionization with Quadrupole Mass Filters
Multiply-Charged Ions
Matrix-Assisted Laser Desorption/Ionization
Time-of-Flight Mass Spectrometry
MALDI matrices and applications

Gas Chromatography-Mass Spectrometry

Vacuum systems
GC/MS interfacing
Gas chromatography methods and techniques relevant to GC/MS
Mass spectrometer operation

Liquid Chromatography-Mass Spectrometry

History of LC-MS
Particle-bean interface
Continuous-flow fast-atom bombardment
Thermospray interface
Nebulization ionization in LC-MS
Electrospray interfacing
Atmospheric-pressure chemical ionization interfaces

Text:

Handouts and lecture notes

Suggested readings:

You are encouraged to read the following journals on a regular basis: Analytical Chemistry, Journal of the American Society for Mass Spectrometry, Journal of Mass Spectrometry and Rapid Communications in Mass Spectrometry.

Prerequisites:

A strong foundation in Analytical Chemistry (at least one year at the undergraduate level) or permission of the instructor.

Method of evaluation: The following components determine your final grade:
Homework assignments 50%, Classroom contributions 10%, Lab report 40%.

Grating system:

<table>
<thead>
<tr>
<th>Grade</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>≥ 90%</td>
</tr>
<tr>
<td>AB</td>
<td>89%-85%</td>
</tr>
<tr>
<td>B</td>
<td>84%-80%</td>
</tr>
<tr>
<td>BC</td>
<td>79%-75%</td>
</tr>
<tr>
<td>C</td>
<td>74%-70%</td>
</tr>
<tr>
<td>CD</td>
<td>69%-65%</td>
</tr>
<tr>
<td>D</td>
<td>64%-60%</td>
</tr>
</tbody>
</table>
Project Experiments

The final month of labs will be devoted to project experiments. You will work in groups of at least 2 or 3. One project will involve environmental analysis, both qualitative and quantitative using GC-MS. The other project will involve sequencing a protein or nucleotide using ESI-MS-MS. Students may select a protein of interest to them or the professor will select something commercially available.

One student in the group will be appointed as group leader and will be responsible for coordinating the groups activities and serve as liaison to the professor.

The process of preparing for this experiment is divided into several parts.

1. Identifying literature relevant to the project
2. Researching the required methods.
3. Devising a detailed plan of approach with lists of required chemicals and equipment and an outline of the operations to be performed and any safety precautions required.
4. Conducting the research.
5. Interpreting the results.
6. Presenting the Results.

Detailed Plan for Project One per group.

1. Bibliography and a 1-2 paragraph summary of each article and its relation to the project. Summaries must be written in your own words. Do not just submit the abstract of the paper. It should be clear that you have read and understood the articles. Only published, peer reviewed articles are acceptable.

2. Detailed plan of approach. The specific steps in the protocol should be explained in detail and rationalized with reference to the literature. Details of sample collection, storage, preparation, and interpretation of results must be addressed in addition to the analysis method.
3. Appendix - Formal Protocol for the analysis. This will include a list of required chemicals, equipment, instrumentation, etc., outline of procedure, and safety precautions required for the equipment and chemicals to be used.

4. Writing folder containing all photocopies used in preparing the proposal, research notes, and anything else accumulated relevant to the project.

5. Detailed time log for each member of the group with a summary table.

**Written Project Report** - Each individual will prepare a journal style report on the project. Each report will include, Introduction and Literature Background, Experimental, Data and Results, Conclusions, and Bibliography. The group may prepare a single Experimental and Data section. Each individual will complete all of the other portions of the report individually and independently.

**Evaluation** - The group will be evaluated based on the total time spent on the project and the quality of the proposal and final presentation. Individual grades will be assigned for the written reports.

**Detailed project log.** Each individual must keep a detailed log of their activities including the time, duration (day, hour, and minute), identity of group member doing the work, and the specific tasks accomplished. Each student should obtain a small notebook for this log. All work will be recorded, library time, group discussion time, individual work time, etc. The notebooks are a complete journal of all individual activities. At the end of the project, a detailed time sheet for the project must be submitted showing the total time the group spent on the project and the individual contributions. The time summary for each student, logs which support it and a copy of the summary table below will be submitted with the written proposal and again at the end of the project.

<table>
<thead>
<tr>
<th>Student</th>
<th>Individual hours</th>
<th>Pair hours</th>
<th>Group Hours</th>
<th>Total Hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>B</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Totals</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Individual hours are time spent working alone, Pair hours refers to time when two people are working together, and Group hours represent time when three people are working together.

**Evaluation-** The overall evaluation of the Project Lab will be based on these factors.

1. Summary of Intended Project (Group)
2. Accuracy and completeness of experimental protocol (Group).

3. Final report. (Individual)

All members of the group will share evenly in the group grades as long as the time contributions of the members are within 10% of the average for the group. Students who do less or more than their share will be rewarded according to their efforts.

Please note you do not have to be completely successful in your analysis to obtain an excellent grade for the project. What is required is for you to spend your time efficiently and effectively. Reports which include no results and those with partial results should provide a detailed explanation of what would be required to complete the project in their final report.