

Syllabus for Chemistry 250A (1 credit)
Instrument Proficiency for Scientists - GCMS
Fall 2009

Instructor

Duane Weisshaar Office: GSC 254 e-mail: duane.weisshaar@augie.edu
Phone 274-4812 class website: <http://faculty.augie.edu/~dew>
Office hours: 11-12 daily and by appointment

Class schedule

Lab activities and literature work averaging about 2-3 hours per week in lab.
Principal locations of course work: GSC 240, 259, and 265.
Regular attendance is expected.
Work in groups of two is permitted and encouraged.

Course description

This course will enable students to develop proficiency in using intelligently the Gas Chromatograph Mass Spectrometer. Students will develop pertinent background through reading, computer based training, and class discussion. Students will develop proficiency in the actual operation of the GCMS instrument and interpretation of data. Corequisite: Chemistry 202.

Instruments

Work will be centered around the HP GCD and Headspace Sampler.

Supplies

Safety goggles with full splash guards (wear them consistently in lab work)
Bound lab notebook (notebook from a previous lab is acceptable).

Resources (no texts to purchase)

Use these resources in place. **Do not remove them!** If you wish, photocopy key sections at your own expense.

Primary Resources

1. Instrument manuals for GCD and Headspace Analyzer.
2. *GCD System Tutorial* on computers Chem13 and Chem19 in GSC 240.
3. *GCD MS Fundamentals* on computers Chem13 and Chem19 in GSC 240.
4. *Basic Gas Chromatography-Mass Spectrometry: Principles and Techniques*, Karasek and Clement, Elsevier: New York, NY, 1988 (QD 79 .C45 K37 1988, in the Seminar Room with the ACOL and Advanced Analysis materials).
5. *Gas Chromatography*, Willet, ACOL, John Wiley & Sons: New York, NY, 1987 (QD79 .C45 W55 1987, Seminar Room ACOL collection).
6. *Mass Spectrometry*, Davis and Frearson, ACOL, John Wiley & Sons: New York, NY, 1987 (QD96 .M3 D38 1987, Seminar Room ACOL collection).
7. *Practical Aspects of Gas Chromatography/Mass Spectrometry*, Massage, John Wiley & Sons: New York, NY, 1984 (QD79 .C45 M46 1984, Seminar Room ACOL Collection).
8. *Spectrometric Identification of Organic Compounds*, Silverstein and Bassler, 1st or 2nd ed., John Wiley & Sons: New York, NY, 1964 and 1967 and other pertinent spectral interpretation books used in organic.



Auxiliary Resources

9. Dr. Weisshaar's Courses Home Page at URL <http://faculty.augie.edu/~dew> and associated links.
10. Analytical Sciences Digital Library <http://asdlb.org/> - a peer-reviewed digital library for the analytical sciences.
11. *J. Chem. Ed.* searchable index on the web at URL <http://data.jche.divched.org/~cesearch/index.php> (link provided on the course web site).
12. Chemistry 311 materials by the window in the Chemistry Seminar Room.
 - a. Lab and Writing Resources - experimental procedures.
 - b. Articles on Instrumental Methods.
 - c. Analysis and Instrumental Methods texts and lab manuals.
13. Do your own search for others, include SciFinder. Share good links with your instructor.

Deadline

Completion of all work and submission of reports is Friday, Dec. 4. In place of a final exam, the class will meet at 2:00 pm on Wednesday, Dec 9, to share experiences and evaluate the course.

Grading

Criteria for grades of C, B, and A are outlined below. Plus/minus grades will be determined by the quality of the work submitted. Submit components as you complete them.

To earn a grade of C:

1. Each person maintain a current and satisfactory laboratory notebook.
2. Be present in lab each week (log your time in your notebook).
3. Complete the *GCD System Tutorial* and the *GCD MS Fundamentals* training modules (GSC 240). Take notes documenting your progress in your notebook. Attain at least 70% on each master exam (print certificate and show to instructor). This component must be completed before working on the instrument itself.
4. Study and take notes on the instrument manual and at least one of the Primary Resources texts on GCMS.
5. Submit a safety report for the intro experiment (handout) which includes hazards, handling, and disposal information for the reagents used. Be specific for your lab situation. Get the instructor's OK on this information BEFORE you begin lab work and follow your specs.
6. Schedule a time with the instructor for an introduction to the instrument. Create a new folder on C: on the GC computer to collect your data files and name it. Save all your GC data files in this directory.
7. Successfully complete the intro experiment.
8. Consult with the instructor to identify a suitable sample. Submit a safety report for analyzing this sample. When the safety report is approved, develop a GCD method to achieve baseline separation of all components in the sample. (Be sure to give a unique name to your method - don't overwrite a previous method). Adjust all parameters to produce a quality chromatogram - strive for a balance between separation of all components and reasonable analysis time. Identify the major components (correlate with the ingredients list on the sample) and account for the three most abundant mass fragments in the spectrum for each of these components (write the mechanism for their formation). Print out the final method, chromatogram, and spectra of each major component. Write a brief summary of the steps you took to arrive at the final method, explaining your rationale for the changes you made along the way (outline form is OK). This report (brief summary and fragment formation mechanisms) must be submitted before you move to the exercises required for a B.

To earn a grade of B - requirements for a C plus:

9. Explore the other Primary Resources and some of the Auxiliary Resources. In a four page paper (double spaced, 1" margins) summarize the pertinent aspects of GCMS and a little on two other often-used GC detectors, the thermal conductivity detector and the flame ionization detector - focus on the fundamental theory of GC and the three detectors and the basic instrumental design and operation. Submit a hard copy and an electronic copy of this report, include your initials in the filename.
10. Quantitate a selected component (consult instructor) in your sample using the calibration curve approach. Set up a **multisample method** to run all the standards and unknowns automatically. Include at least four concentrations in the calibration curve, with at least three injections for each concentration and the unknown. Using a spreadsheet and linear regression, determine the 95% confidence interval for the concentration of the component. Submit a Summary Statistics Report (like the one used in Analysis) for this analysis along with a plot of the calibration curve (data and regression line). Submit a hard copy and an electronic copy of this report and your spreadsheet, include your initials in the filenames. This report must be submitted before you move to the exercises required for an A.
11. Measure the split ratio (consult the manual for directions). This can be done while samples are running.
12. Compile a list of corrections and suggested modifications for the GCD operations manual. Submit a hard copy and an electronic copy of this report, include your initials in the filename.

To earn a grade of A - requirements for a B plus:

13. Study and take notes on the Headspace Sampler Manual and its set of condensed directions.
14. Demonstrate to the instructor your proficiency with the Headspace Sampler.
15. Consult with the instructor to identify a sample suitable for headspace analysis. Submit a safety report for this analysis. After the safety report is approved, develop a GCD method incorporating the Headspace Sampler to achieve a quality chromatogram for the selected sample. Identify the major components (correlate with the ingredients list on the sample) and account for the three most abundant mass fragments in the spectrum for each of these components (write the mechanism for their formation). Print out the final method, chromatogram, and spectra of each major component. Write a brief summary of the steps you took to arrive at the final method, explaining your rationale for the changes you made along the way (outline form is OK), include the fragmentation formation mechanisms.
16. Compile a list of corrections and suggested modifications for the Headspace operations manual. Submit a hard copy and an electronic copy of this report, include your initials in the filename.