

004:209 Separations

Spring 2008 – Monday, Wednesday and Fridays; 9:30 – 10:20 a.m., C139 PC

Instructor: Don Cannon, Ph.D.;
431 CB (office location);
335-2761 (office telephone #);
don-cannon@uiowa.edu (email)

Office hours: Mon. (1:30 to 3:00 pm); Wed. (11:00 to 12:30 pm); *and by appointment*

Required Textbook: James M. Miller, “*Chromatography: Concepts and Contrasts*,” 2nd Edition, Wiley-Interscience, 2005.

Other Resources: Course website is on ICON (icon.uiowa.edu).

J. Calvin Giddings, “Unified Separation Science,” Wiley, 1991.
Daniel C. Harris, Quantitative Chemical Analysis, 6th Edition, 2002.
Other Analytical Textbooks (i.e. Skoog’s “Principles of Instrumental Analysis”)

Research Journals (searchable via SciFinder, etc.)

Analytical Chemistry

The Analyst

Journal of Chromatography A

Journal of Chromatography B

Journal of Separation Science

Journal of Analytical Chemistry

Analytica Chimica Acta

Analytical Biochemistry

Analytical and Bioanalytical Chemistry (Fresenius' J. Anal. Chem.)

Trends in Analytical Chemistry

Talanta

Electrophoresis

Lab on a Chip

General Internet (i.e. www.google.com);

Analytical Sciences Digital Library (www.asdlib.org);

Course Objectives:

Chemical separations have far-reaching applicability to both academic research and industrial processes. *Separations can be generally defined as the selective enrichment of individual target compounds, for either **analytical** or **preparative** purposes.* The goal of this advanced graduate level course is to first teach the basic concepts behind many of these separation techniques. The field has aptly termed this approach the ***unified separations theory***. This unified separations theory allows for discussion of the underlying principles inherent to many areas of chromatographic and electrophoretic separations.

Knowledge of these basic concepts will provide sufficient theoretical understanding of the wide range of separation techniques, such that you will be able to choose, optimize and conduct appropriate separation methods necessary for your current or future research endeavors. Although analytical separations will be the main focus of the specific separation techniques presented in this course, preparative topics such as extractions and column chromatography will be briefly discussed. Specific analytical separation methods that will be discussed and compared include gas chromatography, liquid chromatography and electrophoresis. The Miller textbook was chosen for this course not only for the unified separations theory approach, but also for the chapter on Industrial and Governmental regulations on chromatography, which will serve as an excellent reference for those planning on careers in these fields.

The latter part of the class will rely on the knowledge gained through the unified separations theory, allowing the class to delve into specific separation topics of interest to the participants. These special topics will require the critical reading and review of the research reports in the current literature. Since this is an advanced graduate class with discussion as an integral component, peer to peer learning will further be encouraged through short student-led discussions and the design of an eLearning module. The short student-led discussions, supplemented with a prepared presentation, will be on a separation topic of interest to the presenter, either technology or application motivated. Scientific digital libraries have been gaining in number and effectiveness. The latter-half of the course will also focus on the evaluation of existing internet resources as a preparatory step in the design of eLearning modules to be submitted to the peer-reviewed on-line community of Analytical Sciences Digital Library (ASDL – an NSF sponsored project now in collaboration with the Analytical Division of the ACS). While helping to make important connections in the material being presented, this activity will also give exposure to effective educational resources that are being used by other educators across the world.

Tentative Separations Course Outline:

- I. Introduction to Separations
 - a. history
 - b. definitions, classifications and symbols
 - c. distributions
- II. Physical Forces and Molecular Interactions
 - a. types of forces (i.e. intermolecular, interionic, and sorption)
 - b. physical exclusion
 - c. physical models
- III. Band Broadening and Kinetics
 - a. physical characteristics of columns and stationary phases
 - b. rate theory (van Deemter theory)
- IV. Achieving and Optimizing Separations
 - a. zone broadening kinetics
 - b. zone migration thermodynamics
- V. Preparative Separations
 - a. extractions
 - b. Craig counter current distributions
 - c. classification of preparative column chromatography
- VI. Gas Chromatography
 - a. theories and classifications (i.e. column characteristics)
 - b. instrumentation
- VII. Liquid Chromatography
 - a. theories and classifications (i.e. column characteristics)
 - b. instrumentation
- VIII. Electrophoresis
 - a. principles of electrokinetics (both electrophoresis and electroosmosis)
 - b. various modalities
- IX. Microfluidics (a subtopic in the field of Lab on a Chip)
 - a. integration of tasks (sample handling, preparation, separation and detection)
 - b. benefits to miniaturization (will also discuss nanofluidics)
- X. Special Topics (both technique and application inspired).
 - a. topics chosen by both instructor and student (*references will be provided*)
examples: field flow fractionation, preconcentration methods, chiral separations, hyphenated techniques (LC/MS), enzyme and immuno assays via CE, flow cytometry, etc.
 - b. Student-led Discussions
 - c. ASDL elearning modules

Grading: three midterm exams; special topics literature project including written and oral synopses, four homework problem sets; one final project (emphasis on ASDL eLearning Modules); plus/minus grading system will be used.

Graded Item	Quantity	Total Points
Midterm Exams (3 @ 100 pts each)	3	300
Special Topics Project (includes both written and oral components)	1	200
Problem Sets (50 pts each)	4	200
Final Project	1	200
TOTAL		900

Exam Schedule:

Midterm 1:	February 27, 2008 (Wednesday)
Midterm 2:	April 2, 2008 (Wednesday)
Midterm 3:	May 2, 2008 (Friday)
Final Project Due:	May 16, 2008 (Friday); by 5:00 p.m.

Discussion Schedules: To be determined. Topics will be chosen (and approved by instructor) by March 1st. Presentations will be approximately 15 – 20 minutes in length, including class questions and discussion. Information to be presented includes a brief description of the separation technique followed by a review and critical report of the specific literature paper.

Class Expectations: “The College guideline is for one semester hour of credit to be approximately equivalent of three hours of work (class time & out-of-class preparation) each week over the course of a whole semester. In a typical lecture/discussion course, each hour of class normally entails at least two hours of outside preparation for the average student (e.g., in a three-credit-hour lecture course, standard out-of-class preparation is six hours per week). This standard is the basis on which the Registrar's Office assigns hours of University credit for courses.”

Classroom Policies: "Your responsibilities to this class and to your education as a whole include attendance and participation." Attendance and participation are required. The fulfillment of your attendance and participation responsibilities will be assessed by the instructor during the semester. This assessment will be specifically discussed with individuals if negative performance is observed before points are deducted. “You have a responsibility to the rest of the class, and to the instructor, to help create a classroom environment where all may learn. At the most basic level, this means that you will respect the other members of the class and the instructor, and treat them with the courtesy you hope to receive in turn."

Students with Disabilities: "I would like to hear from anyone who has a disability which may require seating modifications or testing accommodations or accommodations of other class requirements, so that appropriate arrangements may be made. Please contact me during my office hours." *Office of Student Disability Services*

Academic Fraud, Dishonesty, and Cheating: ALL WORK performed by students in this course is expected to be your own. Students are expected to be honest and honorable regarding completion of assignments and in test-taking situations. Answers to the problem sets are expected to be the product of your own efforts. No credit will be given if work that is turned in is determined to not be your own. Plagiarism and other forms of academic fraud is not acceptable and will result in severe penalties as outlined in the College's policy on plagiarism and cheating in the Student Academic Handbook: http://www.clas.uiowa.edu/students/academic_handbook/. Also of note is the service of Turnitin.com, a plagiarism detection service (turnitin@uiowa.edu).

Understanding Sexual Harassment: Sexual harassment is reprehensible and will not be tolerated by the University. It subverts the mission of the University and threatens the well-being of students, faculty, and staff. Visit this [site](http://www.sexualharassment.uiowa.edu/) (<http://www.sexualharassment.uiowa.edu/>) for definitions, assistance, and the full University policy.

Complaints: All students have the right to report any complaints about classroom activities or instructor actions, including English competency. Information regarding these policies and procedures is available in the Schedule of Courses and in the College's Student Academic Handbook: http://www.clas.uiowa.edu/students.academic_handbook/.

Departmental Contact:

Department office: 305 Chemistry Building
Department Chair (DEO): Professor David F. Weimer
DEO telephone: 319-335-3124
DEO email: david-weimer@uiowa.edu

Cross Enrollment: "This course is given by the College of Liberal Arts and Sciences. This means that class policies on matters such as requirements, grading, and sanctions for academic dishonesty are governed by the College of Liberal Arts and Sciences. Students wishing to add or drop this course after the official deadline must receive approval of the Dean of the College of Liberal Arts and Sciences. Details of the University policy of cross enrollments may be found at: <http://www.uiowa.edu/~provost/deos/crossenroll.pdf>."

Reacting Safely to Severe Weather: If severe weather is indicated by the UI outdoor **warning system**, class members will seek shelter in the innermost part of the building, if possible at the lowest level, staying clear of windows and of free-standing expanses which might prove unstable. The class will resume after the severe weather has ended. See the Operations Manual section **16.14.i**.

Tentative Lecture Schedule:

- Jan 23 – Introduction to Separations course
- Jan 25 – Introduction to separations and chromatography (Chapter 2)
- Jan 28 – Defining chromatograms and thermodynamic theory of efficiency (Chapter 2)
- Jan 30 – Defining chromatograms and thermodynamic theory of efficiency (Chapter 2)
- Feb 1 – Measuring parameters from real chromatograms (Appendix B)
- Feb 4 – Band broadening and kinetics (Chapter 3)
- Feb 6 – Band broadening and kinetics (Chapter 3)
- Feb 8 – Physical interactions and equilibria important for separations (Chapter 4)
- Feb 11 – Physical interactions and equilibria important for separations (Chapter 4)
- Feb 13 – Physical interactions and equilibria important for separations (Chapter 4)
- Feb 15 – Optimization and achievement of separations (Chapter 5)
- Feb 18 – Preparative chromatography (Chapter 14)
- Feb 20 – Preparative chromatography (Chapter 14)
- Feb 22 – Preparative chromatography (Chapter 14)
- Feb 25 – Review for exam
- Feb 27 – Midterm Exam 1**
- Feb 29 – Return Exam – start chromatography comparison (Chapter 6)
- Mar 3 – Chromatography comparisons / GC introduction (Chapter 6 & 7)
- Mar 5 – GC columns / SPs / Retention index (Chapter 7)
- Mar 7 – GC injectors / detectors / practical considerations (Chapter 7)
- Mar 10 – LC introduction (Chapter 8)
- Mar 12 – LC (Chapter 8)
- Mar 14 – LC (Chapter 8)
- Mar 15 – Mar 23 Spring Break**
- Mar 24 – CE (Chapter 13)
- Mar 26 – CE (Chapter 13)
- Mar 28 – CE (Chapter 13)
- Mar 31 – Review for exam
- Apr 2 – Midterm Exam 2**
- Apr 4 – Return exam / introduction to special topics

- Apr 7 – Special topics
- Apr 9 – ASDL
- Apr 11 – Special topics
- Apr 14 – 2 Student-led discussions
- Apr 16 – Special topics
- Apr 18 – 2 Student-led discussions
- Apr 21 – 2 Student-led discussions
- Apr 23 – Special topics
- Apr 25 – 2 Student-led discussions
- Apr 28 – Special topics
- Apr 30 – Review and ASDL
- May 2 – Midterm Exam 3**
- May 5 – Return exam / eLearning modules
- May 7 – eLearning modules
- May 9 – eLearning modules
- May 16 – ASDL eLearning modules due

Spring 2006 Student-led Discussions

2-D comprehensive GC (Dimandja, J.-M.,D., “GC x GC,” *Anal. Chem.*, **2005**, 76, 167A-173A.)

SPE (Gontanals, N., Marce, R.M., Borrull, F., “New hydrophilic materials for solid-phase extraction,” *Trends Anal. Chem.*, **2005**, 24, 394-406.)

silica-based packing materials (Krupczynska, K., Buszewski, B. and Jandera, P., “Characterizing HPLC Stationary Phases,” *Anal. Chem.*, **2004**, 76, 226A-234A.)

Two mobile phase chromatography (Dolan, J.W., “The Hazards of Adjusting Gradients,” *LC-GC Europe*, **2002**, 15, 706-710.)

self-assembled carbon nanotubes (Kartsova, L.A. and Makarov, A.A., “Properties of Carbon Materials and Their Use in Chromatography,” *Rus. J. Appl. Chem.*, **2002**, 75, 1725-1731.)

SEC (Striegel, A.M., “Multiple Detection in Size-exclusion Chromatography of Macromolecules,” *Anal. Chem.*, **2005**, 77, 104A-113A.)

molecular imprinted technology chromatography (Haupt, K., "Molecularly Imprinted Polymers: The Next Generation," *Anal. Chem.*, **2003**, 75, 376A-383A.)

industrial applications (Sutherland, I., Hawes, D., Ignatova, S., Janaway, L., Wood, P., "Review of Progress Toward the Industrial Scale-up of CCC," *J. Liq. Chromatogr. & Rel. Technol.*, **2005**, 28, 1877-1891.)

microdialysis (Plock, N. and Kloft, C., "Microdialysis – theoretical background and recent implementation in applied life-sciences," *Eur. J. Pharm. Sci.*, **2005**, 25, 1-25.

LC enantiomeric separations (Haginaka, J., "Pharmaceutical and biomedical applications of enantioseparations using liquid chromatographic techniques," *J. Pharm. Biomed. Anal.*, **2002**, 27, 357-372.)

DNA electrophoresis (Slater, G.W. et al., "Theory of DNA electrophoresis (~1999-2002½)," *Electrophoresis*, **2002**, 23, 3791-3816.)

MEKC (Terabe, S., "Micellar Electrokinetic Chromatography," *Anal. Chem.*, **2004**, 76, 240A-246A.)

CE / biomedical and pharmaceutical apps (Paez, X. and Hernandez, L., "Biomedical Applications of Capillary Electrophoresis with Laser-induced Fluorescence Detection," *Biopharm. Drug Dispos.* **2001**, 22, 273-289.)

CE ultrathin layer gel electrophoresis (Guttman, A. and Ronal, Z., "Ultrathin-layer gel electrophoresis of biopolymers," *Electrophoresis*, **2000**, 21, 3952-3964.)

microdialysis in microfluidic devices (Wang, P.-C., DeVoe, D.L. and Lee, C.S., "Integration of polymeric membranes with microfluidic networks for bioanalytical applications," *Electrophoresis*, **2001**, 22, 3857-3867.)

LC-CE-MS (Stroink, T., et al., "On-line multidimensional liquid chromatography and capillary electrophoresis for peptides and proteins," *J. Chromatogr. B*, **2005**, 817, 49-66.)