

*SC/Chem 4080 :
Advanced analytical separation methods*

Course Grading

Participation and quizzes:	15%
Presentation:	25%
Test:	15%
Midterm:	15%
Final exam:	30%

Important Dates:	Jan 5 th – March 31 st (24 classes)
Final Exam:	TBD
Tests (tentative):	January 28 th , March 1 st
Presentations start:	March 8 th (2 or 3 per lecture)
Selection of Topics:	February 9 th (latest)
Reading Week (no classes):	February 13 th -19 th

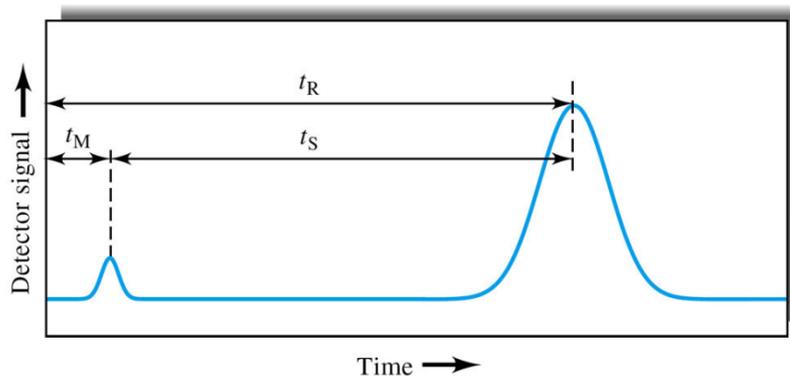
Details presentations

- Each student has to give one presentation of 20-25 min duration + discussion.
 - A list with the subjects (and dates) will be posted. Selection of topics will be based on “First come, first served” basis.
 - The presentation will consist of 4 parts:
 - Literature search (5%)
 - A “notes type” summary document for your class mates identifying the most important message of your talk (Word, Power Point, PDF) with key figure, tables and short explanations for posting on Moodle (5%). Maximum 2 pages including Figures and Tables. References can be up to one page extra.
 - A short “layman” summary (elevator conversation style) (5%)
 - The oral (Power Point) presentation and discussion (10%).
- Total: 25% of final grade.**
- Timelines:
 - i) Selection of topics: Feb 9th
 - ii) Discussion of literature search with CD: 2-3 weeks before presentation
 - iii) Discussion of presentation with CD: 10 days before presentation.
 - If necessary, make appointments with CD in due time.

- The focus of the course is advanced theory and practice of high resolution separation techniques, especially high resolution chromatography and capillary electrophoresis.
- Emphasis of the course is on the practical application of advanced theories and the problem how to optimize (conceptually and instrumentally) separation procedures with respect to different criteria (e.g. separation efficiency, detection limit, analysis time).
- At the end of the course you should be able to understand (and to some extent create) analytical procedures as integrated methods. Basis for this is understanding fundamental physico-chemical principles as well as practical limitations resulting from the need to interface sample injection, separation method and detection.

General principles of separation techniques:

- a) Driving forces, gradients, diffusion, equilibrium and kinetic limitations.
- b) Selective and unselective detection, qualitative and quantitative analysis, detector noise, and detection limit, on-column and off-column detection, sample injection and peak broadening, principle of focusing methods, sample volume and detection limit
- c) Resolution, theory of peak-broadening, peak capacity, peak overlap probabilities.
- d) Pressure, flow resistance, radial profiles and diffusion.



Capillary Electrophoresis (CE).

a) Theory, practical limitations, migration of ions in an electric field, role of run buffer, dependence of electric field on buffer and sample composition.

b) Theory of peak broadening in CE, resolution in practical applications.

c) Sample introduction (hydrostatic, electrokinetic, stacking)

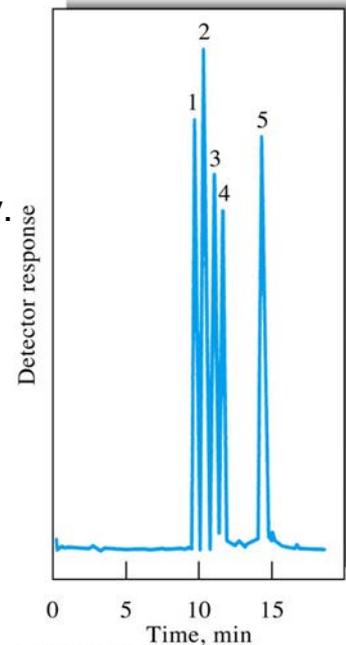
d) Detection (sensitivity, peak broadening, coupling to advanced detection methods)

e) Effect of sample matrix, dependence between buffer, sample size and composition and peak shape

f) Indirect detection

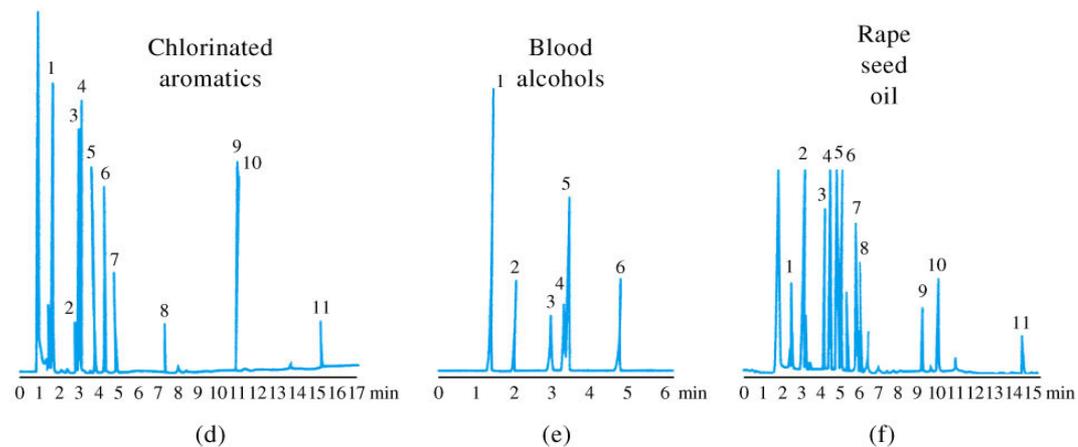
g) Micellar electrokinetic chromatography and capillary electro chromatography.

h) Applications of CE



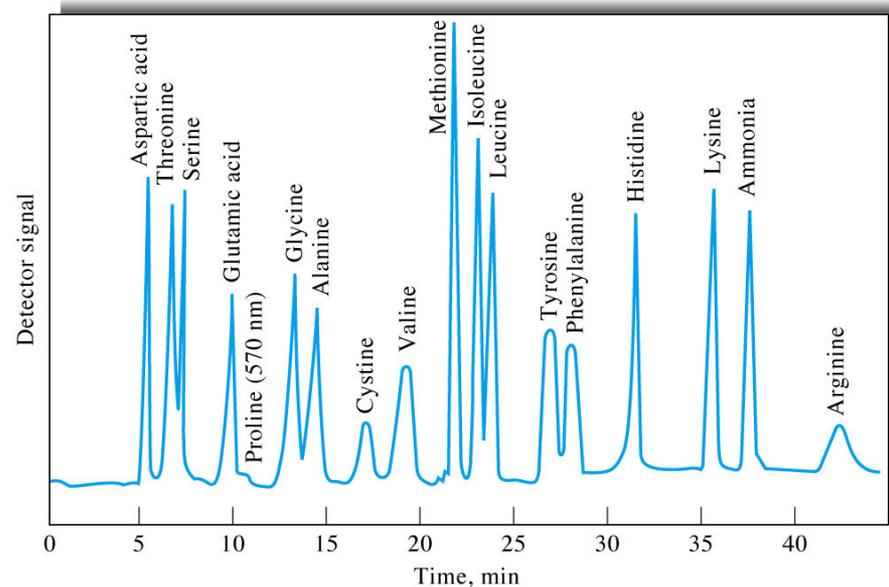
High resolution gas chromatography

- a) Advanced theory of chromatography (zone migration and thermodynamic properties, non-equilibrium effects and peak-broadening, diffusion)
- b) Sample injection (split, splitless, peak-broadening due to sample injection).
- c) Temperature programming and retention time
- d) Selection of stationary phases and column dimensions.
- e) Interfacing with advanced detectors, selective detection.
- f) Applications of “hyphenated” GC-techniques.



High Performance Liquid Chromatography (HPLC)

- a) Interaction between solvent, stationary phase and analyte.
- b) Selection of column dimension and stationary phase.
- c) Solvent, solvent mixtures and solvent gradients
- d) Sample injection, sample size, peak width and detection limits.
- e) Interfacing with advanced detectors, selective detection.
- f) Applications of advanced HPLC.



Books

- D.R. Baker, Capillary Electrophoresis, John Wiley and Sons, Toronto, 1995
- Chromatography Today, C. F. Poole, S. K. Poole, Elsevier, New York, 1991
- Unified Separation Science, J. C. Giddings, Wiley and Sons, New York, 1991
- H.M. Mc Nair, J. M. Miller, Basic Gas Chromatography, New York : Wiley, 1998

E-resources (Library)

- Chromatography : Concepts and Contrasts (2nd Edition), Miller, James M.; Wiley-Interscience; 2005 (eCopy)
- Ion chromatography [electronic resource] / James S. Fritz, Douglas T. Gjerde. 3rd, completely rev. and enl. ed. Weinheim ; New York : Wiley-VCH, 2000.
- Modern practice of gas chromatography [electronic resource]. 4th ed. / edited by Robert L. Grob, Eugene F. Barry. Hoboken, N.J. : Wiley-Interscience, 2004.
- HPLC [electronic resource] ; A practical guide. Cambridge : Royal Society of Chemistry, RSC Distribution Services 1999
- Capillary electrophoresis [electronic resource] : methods and protocols / edited by Philippe Schmitt-Kopplin. Springer Protocols E-books - York University.
- Capillary Electrophoresis Guidebook : Principles, Operation, and Applications, Kevin D. Altria (ed), Methods in Molecular Biology | Volume No.: 52

There are also some very useful chapters in the CHEM 3080 textbook: Principals of Instrumental Analysis, D. A. Skoog,

For those student who have not taken 3080, the most important chapters (with respect to this course) are 1 and 26, 27, 28
(for the students who have taken 3080 refreshing your memory still may be a good idea).

Other useful books

- R. Kuhn, S. Hofstetter-Kuhn, Capillary Electrophoresis: Principles and Practice, Springer-Verlag, New York, 1993, QP519.9 C36 K84 1993, Chapters 1-3, (4), useful tables etc. for practical purposes
- P. D. Grossman, J.C. Colburn (Ed.), Capillary Electrophoresis: Theory and Practice, Academic Press, Toronto, 1992 QP519.9 C36 C37 1992 Good articles by experts on specific subjects, detailed theory, Chapters 1, 2, (3)
- P. Jandik, G. Bonn, Capillary Electrophoresis of Small Molecules and Ions, VCH Publishers, Inc., New York, QD79 E44 J36 1993, Good overview, often very detailed, some quite useful information for more detailed understanding, Chapters 2, 3
- Tandem separation techniques, R.P.W. Scott, John Wiley and Sons, New York, 1997
- QD 79 C4 S 3837 1997
- R.P.W. Scott, Liquid chromatography for the analyst, Marcel Dekker, Inc. New York, 1994, QD 79 C454 S37 1994
- D. Parriott, Ed., A practical guide to HPLC detection, , Academic Press, Inc., Toronto, 1993, QD 79 C 454 P69 1993
- G. Patonay, HPLC Detection, newer methods, Ed. VCH Publishers, New York, 1992 QD 79 C454 H64 1992
- Chromatography : a science of discovery / edited by Robert L. Wixom, Charles W. Gehrke ; associate editors, Deborah L. Chance, Thomas P. Mawhinney, Hoboken, N.J. : Wiley, 2010.
- Chromatography : Fundamentals and applications of chromatography and related differential migration methods / edited by E. Heftmann. 6th ed. Amsterdam ; Boston : Elsevier, 2004.