Chem 868: Topics in ANALYTICAL CHEMISTRY, Winter 2015

Modern Liquid Chromatography

Monday, 7:30-8:50 PM, Disque 307 Wednesday, 6-7:20 PM, Disque 307

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Office Hours: Feel free to call or e-mail me, or to *stop by in the early to mid-afternoon on any day of the week except Thursday and Friday. I will also be available in the late afternoons on Mondays and Wednesdays from 4:00 - 5:30 pm.* In addition, I am willing to schedule a special appointment for anyone who can't see me during the above office hours.

Course objectives

A. To gain a better appreciation for and/or a quantitative understanding of a variety of aspects of modern liquid chromatography, including

1. general principles of chromatography performed under constant experimental conditions

- 2. quantitative analysis using external or internal standards
- 3. instrumental aspects of high/ultra performance liquid chromatography (HPLC/UHPLC)
 - 4. various physical and chemical aspects of state-of-the-art stationary phases
 - 5. reversed-phase liquid chromatography (RPLC), the most popular separation mode

6. principles of chromatography when the mobile phase strength is varied during the separation, i.e, under gradient elution conditions

7. other separation modes applicable to liquid chromatography

8. approaches to chiral separations, i.e., the separation of enantiomers, diastereomers, and other stereoisomers

9. nonlinear/preparative chromatography

10. chromatographic peak integration and the inherent errors associated with the measurement of overlapping peaks

B. To increase your knowledge and ability to perform quantitative calculations pertaining to chromatographic separation theory and quantitative analysis

C. To further improve your understanding of HPLC via the use of a free Java-based simulator for RPLC

Text(s): Please purchase the *required text* <u>Introduction to Modern Liquid Chromatography</u>, 3rd edition, written by Snyder, Kirkland, and Dolan and published by Wiley (2010, ISBN 978-0-470-16754-0 (hardback)).

The following book is *strictly optional* although some students have found it to be very **useful**: V. Meyer, <u>Practical High Performance Liquid Chromatography</u>, 5th edition, Wiley, 2010, ISBN 978-0-470-68217-3 (paperback).

Journal articles: Numerous journal articles cited in the textbook have been pre-downloaded and organized into a compressed EndNote library that will be available via Bb Learn. Similarly, review articles published in pertinent journals such as the Journal of Chromatography A and Trends in Analytical Chemistry have been loaded onto compressed EndNote libraries that will be available via Bb Learn.

Other reading materials: Additional supplementary information will be posted on the Bb Learn website for this course. Examples include selected chapters from the <u>Advances in</u> <u>Chromatography</u> series (now up to volume 52); <u>Chromatographic Integration Methods</u> (Dyson), 2nd edition; <u>Chromatography</u> (Heftmann), 2nd edition; and <u>Multidimensional Liquid</u> <u>Chromatography</u> (Cohen and Schure).

Other resources: A free Java-based HPLC simulator will be used to illustrate a number of important concepts and practical aspects of reversed-phase liquid chromatography. It can be utilized within a web browser or independently as a stand-alone app. It is available from the following website: http://www.hplcsimulator.org/simulator.php

Additional resources will be announced during the term.

Grading: Your course grade will be determined as outlined below. The percentages are approximate $(\pm 5\%)$. At least 20% of the mid-term will be take-home.

Selected homework (computer simulations/problem sets)	24 %
Mid-Term Exam (take-home)	22 %
Review topic/article presentations	20 %
Attendance at one of two CFDV seminars during Jan. and Feb.	10 %
Final Exam (March 16 or 18 (TBD), 6-8 PM, Disque 307)	24 %

Grading scale: The standard Drexel grading scale is employed, i.e., $\ge 97 = "A+"$, 93-96 = "A", 90-92 = "A-", etc.

Reading assignments: For every topic that we cover in class that is also discussed in the required textbook, I expect you to read the corresponding chapter or section without me having to make a formal assignment. However, a cumulative summary of your reading assignments is incorporated into the syllabus that is posted on Bb Learn; I will try to remember to update the syllabus if substantial changes are made in the reading assignments.

Computer Exercises/Problem Sets: Up to 3 problem sets will be assigned, collected, and graded during the semester. Problem sets must be submitted in the following format or they will be returned to the student for no credit: (i) 1-2 page summary sheet of the answers to the problems *in the order assigned*: (ii) detailed solution to each of the problems *in the order assigned*. Students are strongly encouraged to use some type of spreadsheet (Google, Microsoft Excel, etc.) for statistical or repetitive calculations.

Late problem sets will be penalized up to 5% per weekday unless there is a legitimate reason (as determined by the instructor).

Exams: If you cannot attend a scheduled exam, you must notify me in advance. If no such notification is given before the exam and no *acceptable* excuse is given afterwards, you will receive a zero for the exam.

Review topic/article presentations: Your assignment is to select, from a list of chapters in the serial monograph <u>Advances in Chromatography</u> or review articles published in the <u>Journal of Chromatography A</u> posted on Bb Learn, an approved review topic/chapter/article and then present an oral summary via an 15-18 minute PowerPoint presentation, followed by 3-5 minutes of questions from other students and the instructor.

Attendance at CFDV seminars: The Chromatography Forum of the Delaware Valley (CFDV), a local professional society devoted to separation science, has monthly seminars on Tuesdays at 7:30 PM; please consult the website (<u>http://www.cfdv.org</u>) for more details. Social hour and dinner prior to the seminar are strictly optional; full-time students are entitled to a 50% discount on dinner, i.e., the cost is \$15 with the discount. Please plan to attend one of the two seminars on January 27 and February 24, whichever is of greater interest to you.

Students who are unable to attend the CFDV seminars because they are taking a class on Tuesday evenings will be asked to summarize a book chapter, research article, or review article published recently by the CFDV speaker and suggested by the instructor.

Communication by Bb Learn and e-mail: It is VERY important for me to be able to communicate with each member of the class quickly and efficiently. Reading and homework assignments, usually announced in class, are posted on the Bb Learn Calendar for Chem 531; use the "Create printable view" feature of the Calendar to generate a cumulative list. Please be sure to *check Bb Learn at least 3 times a week*, e.g., every weekend <u>and</u> each night before our class so that if you have a question about an assignment, you could ask me about it in class the next day.

You are also welcome to submit a question about Chem 531 to me at jfoley@drexel.edu. If the number of questions submitted by email is not too large, I will reply by email and distribute the answer to the entire class (without revealing your identity!).

If you use an email address or alias other than your official Drexel email address ("firstname.middlename.lastname@drexel.edu"), it is now university policy that <u>it is your</u> responsibility, not the responsibility of the university or the instructor, to arrange for email to be forwarded appropriately, i.e., it is your responsibility to make sure that you receive all email messages that are sent to your official email address.

Academic Policies

Please review the following links provided below

- Academic Misconduct, Cheating, Fabrication, Plagiarism, Withdrawal of a Degree http://www.drexel.edu/provost/policies/academic_dishonesty.asp
- Academic Honesty Policy
 http://www.drexel.edu/studentlife/judicial/honesty.html
- Course Drop Policy
 http://www.drexel.edu/provost/policies/course_drop.asp
- Office of Disability Services http://www.drexel.edu/oed/disabilityResources/
- Course Change Policy

The instructor may make changes to the course during the term at his discretion. If changes are made to the course, students will be notified via BbLearn, email, and will be made aware verbally at the beginning of the next scheduled class.

Cumulative reading assignments

1. Lecture notes - all posted modules

2. Snyder, Kirkland, and Dolan, <u>Introduction to Modern Liquid Chromatography</u>, 3rd edition, Wiley, 2010

Preface

Glossary

Chapter 1. Introduction: all sections, with particular emphasis on sections 3 and 4

Chapter 2. Basic concepts and the control of separation: sections 1, 2, 3.1, 3.2, 4, 5.1-5.3, 7.1-7.2, and 7.5-7.7).

Chapter 3. Equipment: sections 1-7, 8 (skim), 9

Chapter 4. Detection

Chapter 5. The column: sections 1-5

Chapter 6. Reversed-phase chromatography for neutral samples: sections 1-3, 5, 6

Chapter 7. Ionic samples: reversed-phase, ion-pair, and ion-exchange chromatography

Chapter 8. Normal-phase chromatography: sections 1, 3, and 6

Chapter 9. Gradient elution: sections 1, 2.1, and 2.2

Chapter 11. Qualitative and quantitative analysis

Chapter 13. Biochemical and synthetic polymer separations: section 8

Chapter 14. Enantiomer separations: sections 1-5, 6.1, 6.4, 6.5, and 6.7

Chapter 15. Preparative separations

Chapter 16. Sample preparation: sections 1-8