

# Analytical Separations

## CHEM 516

### Iowa State University

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Purpose and Goals: Since the time that Tsweet discovered chromatography at the turn of the century, the field has changed immensely. At that time, chemists were largely limited to techniques such as crystallization, liquid-liquid extraction, and distillation methods for the separation of their components. Despite the fact that chromatography is a century old, the field is still alive and thriving. As an analytical chemist, quality control and quality assurance are of ultimate concerns. Modern separation techniques coupled with detection methods assuring high sensitivity are essential in any governmental, industrial, or academic laboratory. This course will provide a look into the past century of chromatography and present the basics of modern chromatography. In addition, separation techniques based on extraction as well as electrophoretic separation techniques such as capillary electrophoresis will also be considered. Larger scale separations of volatile compounds via distillation will also be discussed. The ultimate goal of this course is to provide students with an advanced understanding of how the various separation techniques work and which techniques are ideal for specific analytes or situations. For those students enrolled in the laboratory section of this course, the laboratory will complement the theoretical discussions with practical applications.

Text: Currently, no graduate-level text offers a complete and comprehensive survey of all separation techniques that we will discuss in this course.

However, a nice reference book covering the chromatography section of this course follows and is highly recommended.

- “*The Essence of Chromatography*” Colin F. Poole, © Elsevier 2003 ISBN: 0 444 50199 1

References to certain texts, online references, and papers will be given periodically throughout the semester in the course notes.

Course Outline: The following outline demonstrates the order of the topics discussed in this course.

- I. Introduction to Chromatography
  - a. Theory
  - b. Peak shape and band broadening
  - c. Van Deemter Plots
  
- II. Gas Chromatography (GC)
  - a. Theory
  - b. Instrumentation design
  - c. Column components and stationary phases
  - d. Methods of detection
  - e. Golay Equation
  - f. Rohrschneider-McReynolds column evaluation
  - g. Linear Free Energy Relationships
  - h. Chromatogram evaluation
  
- III. High Performance Liquid Chromatography (HPLC)
  - a. Theory
  - b. Instrumentation design

- c. Column components and stationary phases
  - d. Methods of detection
  - e. Mobile phase characteristics
  - f. Linear Free Energy Relationships
  - g. Chromatogram evaluation
- IV. Supercritical Fluid Chromatography (SFC)
- V. Thin Layer Chromatography (TLC)
- VI. Gel Permeation Chromatography/Size Exclusion Chromatography (GPC & SEC)
- VII. Affinity Chromatography
- VIII. Chiral Chromatography
- a. Chiral recognition theory (three point interaction model)
  - b. Gas Chromatography
  - c. Liquid Chromatography
  - d. Miscellaneous techniques
- IX. Ion Exchange Chromatography (IEC)
- X. Countercurrent Chromatography (CCC)
- XI. Field Flow Fractionation (FFF)
- XII. Electrophoretic Methods of Separation
- a. Capillary Electrophoresis
  - b. Capillary Electrochromatography (CEC)
- XIII. Separation techniques based on extraction
- a. Liquid-liquid extraction (LLE)

- b. Countercurrent extraction
- c. Solid Phase extraction
  - i. Solid-phase microextraction
  - ii. Liquid-phase microextraction
  - iii. Stir bar sorptive extraction

#### XIV. Distillation theory and methods

Academic Honesty/Dishonesty: Examples of such acts of academic dishonesty are listed within the Graduate Handbook. Any act of dishonesty will be immediately reported to the student's academic program.

Grading: The overall course grade will be determined according to the following breakdown. The final exam will not be comprehensive in nature.

Hour exam I – 33 1/3%; Hour exam II – 33 1/3%; Hour exam III (Final exam) – 33 1/3%

#### **ISU's Principles of Community**

Students are responsible for living the tenets established in ISU's Principles of Community: Respect, Purpose, Cooperation, Richness of Diversity, Freedom from discrimination, and the Honest and respectful expression of ideas. Visit ISU's [Principles of Community webpage](#)

#### **Student Health and Wellness**

Iowa State University is committed to proactively facilitating all students' well-being. We welcome and encourage students to contact the following on-campus services for their physical, intellectual, occupational, spiritual, environmental, financial, social, and/or emotional needs:

- Student Wellness call 515-294-1099 or via website (<http://studentwellness.iastate.edu>)
- Thielen Student Health Center call 515-294-5801 (24/7 Medical Advice) or via website (<http://www.cyclonehealth.org>)
- Student Counseling Services call 515-294-5056 or via website (<https://counseling.iastate.edu>)
- Recreation Services call 515-294-4980 or via website (<http://recservices.iastate.edu>)
- Students dealing with heightened feelings of sadness or hopelessness, thoughts of harm or suicide, or increased anxiety may contact the ISU Crisis Text Line (Text ISU to 741-741) or contact ISU Police Department 515-294-4428.

**Responsible Employee.** Iowa State University is committed to creating an educational, work, living, and campus environment that is free from all forms of sexual harassment, sexual assault, dating violence, domestic violence, stalking, and unlawful discrimination and harassment on the basis of protected class. As a responsible employee, I am responsible for reporting all incidents

of prohibited sexual harassment, including sexual assault, stalking, and dating and domestic violence, to the university's Title IX coordinator. Students can choose to discuss their experiences confidentially with the following resources: ACCESS (Assault Care Center Extending Shelter and Support) at 515-292- 0500; ISU Student Counseling Services at 515-294-5056; and Thielen Student Health Center at 515-294-5801.

**Expectations for mutual respect and professionalism.** You are expected to treat your instructor and all other participants in the course with courtesy and respect. Your comments to others should be factual, constructive, and free from harassing statements. You are encouraged to disagree with other students, but such disagreements need to be based upon facts and documentation (rather than prejudices and personalities). It is the instructor's goal to promote an atmosphere of mutual respect in the classroom. Please contact the instructor if you have suggestions for improving the classroom environment. It is preferable if students discuss issues directly with the instructor; however, students may also leave a note in the instructor's mailbox or via Inbox in Canvas.

**ISU's Inclusive Language Policy.** All university publications and communication, whether oral or written, shall use inclusive language and illustrations. Inclusive language refers to language that makes every attempt to include comprehensively all groups in the community. Whenever possible, selection of academic materials will also reflect efforts to uphold this university policy." Visit the Policy Library's [Inclusive Language website](#).

**Course usability, disability and design.** I am committed to creating a course that is inclusive in its design. If you encounter barriers, immediately let me know so that we can determine if there is a design adjustment to make or if an accommodation might be needed to overcome the design's limitations. I am always happy to consider creative solutions as long as they do not compromise the intent of the assessment or learning activity. You are also welcome to contact the Student Accessibility Services via phone at 515-294-7220 to begin this conversation or to establish accommodations for this or other courses. I welcome feedback that will assist me in improving the usability and experience for all students.