Analytical Separations

CHEM 516

Iowa State University

Instructor: Jared L. Anderson

Phone: 294-8356

Office: 0210 Hach Hall

E-mail: andersoj@iastate.edu

Office Hours: 2:00 - 3:30 M W, other hours possible by appointment

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 compliment 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%