

CHEMISTRY 447
PHYSICAL METHODS OF ANALYSIS
(FALL, 2001)

FACULTY:

Mark E. Meyerhoff (room 3306)

(Office hours: 9 – 10 AM, M, W, F)

GENERAL COURSE INFORMATION:

Classes: M,W,F 8-9 AM, room 2000

Selected evening lectures/problem sessions on certain Mondays, 5:00-6:30 PM

Exams: 4 (each worth 25% of grade)

Schedule for first 3 exams (6-8 pm): October 2, October 30, November 27

The 4th exam, given during final exam week, will cover material from November 27 to remainder of term—plus ACS exam type multiple choice questions covering topics from entire course!

Required Textbook: D. A. Skoog, F. J. Holler and T. A. Nieman, "Principles of Instrumental Analysis," 5th Edition, Saunders, 1997.

Problem Sets/Old Exams: Several problem sets and/or old exams will be posted on the course Web site throughout the term---only for practice, not to be turned in or graded. Answers will be posted and/or reviewed during evening problem sessions.

Web-Site: www.umich.edu/~chem447 ---All course announcements, lecture notes, problem sets, old exams, etc. will be posted on this site.

Library Reserve List: see attached

*additional handouts and journal articles will be assigned for reading throughout the term.

Chapter numbers refer to chapters in Skoog, Leary, and Nieman where relevant material can be found!

A. Analytical Measurements and Measurement Systems (6 lectures)

1. Introductory Material—Definition of Terms, Basic Instrument Designs (Chapter 1)
2. Evaluation of a measurement system (Chapter 1)
 - a. selectivity, sensitivity
 - b. calibration methods--standards and standard addition
 - c. recovery studies and matrix effects
3. Basic instrumentation principles (Chapters 2, 3 and 5)
 - a. basic electrical measurements; voltage dividers, RC circuits, RC filters, etc.
 - b. operational amplifiers
 - c. noise and noise reduction techniques (FT methods, signal averaging, lock-in amplifiers, etc.)

B. Electroanalytical Methods (8 lectures)

1. Fundamental principles and introductory concepts (Chapter 22)
 - a. thermodynamics of redox rxns; Nernst equation
 - b. kinetics of electron transfer rxns (mass transfer, diffusion)
 - c. types of cells and electrodes
2. Potentiometry (Chapter 23)
 - a. direct and indirect potentiometry with ISEs, pH measurements
 - b. junction potentials and other sources of errors
 - c. gas sensors and miniature sensors
2. Voltammetric/Amperometric methods (Chapter 25)
 - a. current-voltage curves, diffusion currents
 - b. polarography (DC, pulse and differential pulse)
 - c. oxygen sensors
 - d. amperometric titrations
 - e. anodic stripping and cyclic voltammetries

C. Optical Methods of Analysis (9 lectures)

1. Properties of radiation and origin of atomic and molecular spectra (Chapters 6, 13)
3. Components of Optical Instruments (Chapter 7)
 - a. sources of radiation (including lasers)
 - b. dispersion by gratings and prisms
 - c. slit-width and resolution
 - d. radiation transducers, PM tubes, etc.

3. Atomic spectroscopy (Chapters 8, 9, 10)
 - a. comparison of emission, absorption and fluorescence methods
 - b. radiation sources and sample cells for given approaches
 - c. high-temperature plasma devices (ICP)
 - d. nebulizers and furnaces
4. Molecular spectroscopy (Chapters 14, 15, 16)
 - a. UV-Vis instruments
 - b. molecular fluorescence and phosphorescence
 - c. raman spectrometry
 - d. infrared instruments—sources and detectors

C. Mass Spectrometry-Atomic and Molecular (4 lectures) (Chapters 11 and 20)

1. Basic principles
2. Ionization methods
 - a. electron impact
 - b. chemical ionizations
 - c. field ionization
 - d. fast atom bombardment
 - e. laser ionization
 - f. plasma ionization
 - g. electrospray
3. GC and LC interfaces
4. Mass analyzers
 - a. magnetic sector
 - b. quadrupole
 - c. time-of-flight
 - d. ion cyclotron resonance
 - e. ion traps, etc.
5. MS-MS

E. Bioanalytical Methods (3 lectures) (Handouts)

1. General concepts
 - a. equilibrium vs. non-equilibrium (kinetic) chemical measurements
2. Specific approaches, principles, and instrumentation
 - a. enzymatic analysis (for enzyme activity; for substrate analysis)
 - b. enzyme electrodes and other biosensors (glucose electrode)
 - c. principles of modern enzyme and fluorescence immunoassays

F. Separation Methods (10 lectures)

1. General overview (Chapter 26)
 - a. stationary phases and mobile phases
 - b. types of analytical methods (including planar chromatography)
2. Partition theory and column efficiency theory(Chapter 26)
 - a. HETP
 - b. capacity factor, selectivity factor, resolution
3. Liquid chromatography (HPLC) (Chapter 28)
 - a. stationary phases
 - b. detectors
 - c. reversed phase vs. normal phase
 - d. ion-exchange system
 - e. size exclusion
4. Gas Chromatography (Chapter 27)
 - a. principles and theory of gas-solid vs. gas-liquid
 - b. instrumentation
 - c. columns and stationary phases
 - d. detectors (thermal conductivity, flame ionization, etc.)
 - e. applications
5. Electrophoresis (CZE, etc.) (Chapter 30)
6. Hyphenated methods
 - a. GC-MS
 - b. GC-FTIR
 - c. LC-MS and LC-MS-MS

G. Automated Methods of Analysis (2 Lectures) (Chapter 33)

1. Continuous Flow and Flow-Injection Analysis
2. Microtiter Plates
3. Centrifugal Analyzers
4. Multilayer Film Systems

Instructor: M. E. Meyerhoff

Books on Reserve in Science Library

1. D.A. Skoog, et al. "Principles of Instrumental Analysis", Saunders, 1997, 5th Ed.
2. H.H. Willard, L.L. Merritt, J.A. Dean and F.A. Settle, "Instrumental Methods of Analysis", Wadsworth, 7th Edition, 1988.
3. D.C. Harris, "Quantitative Chemical Analysis", W. H. Freeman, 1995, 4th Ed.
4. K. A. Rubinson and J. F. Rubinson, "Contemporary Instrumental Analysis", Prentice-Hall, 2000.
5. H.A. Strobel and W.R. Heineman, "Chemical Instrumentation: A Systematic Approach", John Wiley, 3rd. Edition, 1989.
6. Robinson, "Atomic Spectroscopy", Dekker, 1990.
7. J. Wang, "Analytical Electrochemistry", Wiley-VCH, 2000, 2nd Ed.

M. E. Meyerhoff—Biographical Sketch

Mark E. Meyerhoff is Professor of Chemistry in the Department of Chemistry at the University of Michigan. He received his Ph.D. from the State University of New York at Buffalo in 1979. Following post-doctoral work at the University of Delaware, he joined the faculty at Michigan as an Assistant Professor in the Fall of 1979. His research interests are in the field of analytical chemistry, particularly the development of new ion-, gas-, and bio-selective electrochemical sensors suitable for whole blood measurements of clinically important analytes. He also has active research programs in the areas of novel non-separation immunoassay methods, immobilized metalloporphyrin phases for liquid chromatography, and the development and characterization of nitric oxide (NO) releasing polymeric materials for biomedical applications. He and his collaborators have authored more than 225 original research papers on these topics. Professor Meyerhoff is on the editorial/advisory boards of *Clinical Chemistry*, *Biosensors & Bioelectronics*, *Electroanalysis*, *Analytica Chimica Acta*, and *Applied Biochemistry and Biotechnology*. He also serves as a consultant or is on the Scientific Advisory Boards of the following companies: Medtronic Perfusion Systems, Instrumentation Laboratories, Bolton Medical, Monsanto Inc., GDS Technologies, and Selective Technologies.

Recent Publications:

U. Ruedel and M. E. Meyerhoff, "Redox Modulated Solute Retention on Metalloprotoporphyrin-Silica Stationary Phases," *Anal. Chim. Acta*, 392, 191-200 (1999).

L. C. Chang, M. E. Meyerhoff, and V. C. Yang, "Electrochemical Assay of Plasminogen Activators in Plasma Using Polyion Sensitive Membrane Electrode Detection," *Anal. Biochem.*, 266, 116-124 (1999).

G. M. Annich, J. P. Meinhardt, K. A. Mowery, m B. A. Ashton, S. I. Merz, R. B. Hirschl, M. E. Meyerhoff, and R. H. Bartlett, "Reduced Platelet Activation and Thrombosis in Extracorporeal Circuits Coated with Nitric Oxide Release Polymers," *Crit. Care Med.*, 28, 915-920 (2000).

K. A. Mowery, M. H. Schoenfisch, J. E. Saavedra, L. K. Keefer and M. E. Meyerhoff, "Development of Hydrophobic Thromboresistant Polymer Films Via Nitric Oxide Release," *Biomaterials*, 21, 9-21 (2000).

M. H. Schoenfisch, K. A. Mowery, M. V. Rader, N. Baliga, J. A. Wahr and M. E. Meyerhoff, "Improving the Thromboresistivity of Chemical Sensors via Nitric Oxide Release: Fabrication and In Vivo Evaluation of NO Releasing Oxygen Sensing Catheters," *Anal. Chem.*, 72, 1119-1126 (2000).

S. Dai, Q. Ye, E. Wang and M. E. Meyerhoff, "Polymer Film Modified Microtiter Plates for Optical Detection of Polycations: Response Mechanism and Bioanalytical Applications," *Anal. Chem.*, 72, 3142-3149 (2000).

J. Sang Kim, J. Pike, D. Coucouvanis, and M. E. Meyerhoff, "Enzyme Electrode with Enhanced Specificity Using Outer Polymeric Membrane Doped with Substrate Selective Ditopic Carrier," *Electroanalysis*, 12, 1258-1262 (2000).

S. Chen, J. C. Fetzer and M. E. Meyerhoff, "Retention Behavior of Large Polycyclic Aromatic Hydrocarbons on Protoporphyrin-Silica Stationary Phases," *Fresenius J. Anal. Chem.*, 369, 385-392 (2001).

S. Dai and M. E. Meyerhoff, "Non-Separation Binding/Immunoassays Using Polycation-Sensitive Membrane Electrode Detection," *Electroanalysis*, 13, 276-283 (2001).

Q. Ye and M. E. Meyerhoff, "Rotating Electrode Potentiometry: Lowering Detection Limits of Non-Equilibrium Polyion-Sensitive Membrane Electrodes," *Anal. Chem.*, 73, 332-336 (2001).

H. Zhang, S. I Merz, G. M Annich, K. Osterholzer, J. Miskulin, R. H. Bartlett and M. E. Meyerhoff, "Nitric Oxide Releasing Silicone Rubbers with Improved Blood Compatibility: Preparation, Characterization, and *In Vivo* Evaluations," *Biomaterials*, in press, 2001