

EHT 525 Environmental Chemical Analysis Fall 2009

Faculty:

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Office Hours:

9:00 – 4:00

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Course Description

A one semester course that teaches the principles and applications of instrumental analysis for environmental chemists. The course is designed for first-year graduate students or senior undergraduates.

Learning Objectives

Students will acquire knowledge of the instrumental tools suitable for the chemical analysis of environmental samples. They will learn of the appropriate procedures and method for the collection, preservation and pretreatment of samples for instrumental analysis and the principles of the techniques that are applied to produce accurate, sensitive and reproducible analytical results.

Prerequisites for the course

Two years of college chemistry or the consent of the instructor.

Course Outline

INTRODUCTION

Nature and scope of environmental Chemical Analysis.
Criteria for selecting an analytical method
Sampling of water, air, soils sediments and sludges
Sample pretreatment
Reliability of analytical data

ATOMIC SPECTROMETRY

Principles of atomic spectroscopy: atomic emission, atomic absorption and atomic fluorescence; energy level diagrams; hollow-cathode lamps; spectral line broadening; detection using photomultiplier tubes.

Flame atomic absorption spectrometry: nebulizers, air - acetylene burners, nitrous oxide - acetylene burners, releasing agents, flame chemistry, worked examples, calibration, method of standard additions.

Background absorption errors and correction techniques:
Continuum (deuterium and tungsten lamp) systems; Zeeman effect systems (transverse vs longitudinal) and pulsed (Smith-Heijfje) systems.

Graphite-furnace AAS: wall atomization, and transient signals, gas-phase and condensed-phase interferences. L'vov platform, furnace temperatures, pyrolytic graphite and STPF theory, applications.

Inductively-coupled plasma atomic emission spectrometry (ICP-AES), RF plasmas, dc plasmas, arc/spark sources, monochromator,

interferences.

Elemental analysis using Inductively Coupled Plasma Source- Mass Spectrometry (ICP/MS). Interface designs, DRC, orthogonal sampling of ions, limits of detection for multielement analysis.

Mass analyzers. Nominal and exact mass measurements.

Sample introduction, ionization and element measurement. Collision cells and interference issues.

Sample collection and preparation: solids homogenization; ball mills; digestion procedures; wet ashing; dry ashing; pre-concentration and extractions.

CHROMATOGRAPHY

Theory (2): principles of chromatography; terminology, classification of chromatographic methods; plate theory, rate theory, column efficiency; factors affecting chromatographic separation.

GC Instrument (2): principle, retention volumes; gas-liquid chromatography, gas-solid chromatography; instrument description; carrier gas; injectors; columns, pack column, capillary column; thermostated oven; detectors, FID, ECD, TCD, TID; applications.

HPLC Instrument (4): general instrument description; solvent delivery system; sample injection system; columns; types of LC, partition chromatography, adsorption chromatography, ion chromatography, size-exclusion chromatography; detectors, absorbance detector, fluorescence detector, refractive-index detector, electrochemical detector, conductivity detector/suppressor; applications.

MASS SPECTROMETRY

Mass Spectrometry - historical development of instrumentation, mass spectroscopic terminology, units, exact molecular weight, nominal mass, mass resolution. Components of a mass spectrometer. Vacuum systems, electronics, data system.

The electron impact source, ion detection and magnetic mass spectrometers.

Mass filters, quadrupole mass spectrometers, ion traps and time of flight instruments.

Molecular spectra and identification of pure compounds by mass spectrometry. Solid inlet probe and gas sampling probe. Other sample ionization techniques.

Gas Chromatography / mass spectrometry - development of the inlet and interface. Instrumental consideration for data acquisition and processing. Consideration for GC/MS with high and low resolution mass spectrometers. Calibration of mass and response functions, use of internal standards.

Gas Chromatography / low resolution MS - The use of mass spectral libraries for identification of organic compounds. Full scan, selected ion monitoring experiments. Quantitation of volatile organic compounds using GC/MS.

Gas Chromatography / high resolution MS - full scan exact mass measurement, selected ion monitoring, quantitation. Applications of GC/HRMS in environmental analysis.

Gas Chromatography / mass spectrometry - Processing of GC/MS raw data to obtain qualitative and quantitative results. Reconstructed ion chromatograms, library matching, quantitation. Quality control of GC/MS analyses. Maintenance of MS instrumentation.

Liquid Chromatography /mass spectrometry – interface requirements and methods used. Ionization methods and sample introduction.

Text: Suggested reading – not required. “Principles of Instrumental Analysis”, 6th Edition
Skoog/Holler/Crouch ISBN-10: 0-495-01201-7. Published by Cengage Learning .

Assessment:

Grading A-C

Mid-term Examination 45%

Take Home Question 5%

Final Examination 50% (questions set on second half of semester lectures)

