

The American Chemical Society  
Presents



# *Microwave Enhanced Sample Preparation: General, Analytical, and Environmental Applications*

*A Hands-On Lecture-Laboratory Short Course  
Monday–Friday, July 30–August 3, 2001*

*Duquesne University  
Pittsburgh, Pennsylvania*

## **Important!**

Enrollment in this hands-on course is strictly limited to 20 participants to ensure that everyone receives individual attention. We urge you to **register today** to guarantee you will have a reserved seat in this cutting-edge course.

## Who Should Attend

Analytical chemists, environmental chemists, clinical and forensic chemists, analysts; chemists involved in sample preparation for spectrometry, spectroscopy and chromatography, trace and ultra-trace elemental analysis, or sample preparation involving organic extractions; clinicians; technicians; and laboratory supervisors who are responsible for sample analyses. Participants should be familiar with basic analytical techniques.

## 17 Key Ways You'll Benefit from Attending This Course

- Learn to develop a sample preparation procedure for your own sample matrix.
- Understand why traditional pressure/temperature relationships do not hold in most microwave equipment and how to use these unique relationships to your advantage.
- Master fundamental concepts in quality assurance, method standardization and transfer of methods.
- Acquire understanding and laboratory experience with ASTM, SEMI, EPA, NCCLS, and other standard methods.
- Participate in hands-on laboratory sessions with a variety of instruments.
- Gain laboratory exposure to the state-of-the-art in laboratory microwave equipment—moderate pressure, high pressure and atmospheric pressure equipment and vessels.
- Learn fundamental microwave decomposition and extraction procedures for environmental, industrial, biological, botanical, geological, aqueous, and other sample types.
- Review the use of microwave extraction as an alternative to Soxhlet.
- Learn how microwave energy is absorbed by reagents and sample molecules to produce heat.
- Gain a theoretical understanding of why microwave sample decomposition and extraction is superior chemically as well as faster and more efficient than conventional methods.
- Receive reference materials and a comprehensive review of acid dissolution chemistry.
- Understand the advantages and alternatives of specific reagents and acid combinations.
- Gain clean chemistry techniques that permit lower analytical and laboratory blanks and provide improvement in quality assurance of analysis.
- Gain insight into predicting sample behavior in the microwave environment.
- Gain expertise in optimizing equipment for your application requirements and specifications.
- Evaluate alternative features and options you may need from manufacturers.
- Receive a comprehensive literature review of microwave enhanced chemistry applications.

## Special Course Bonus!

All attendees will receive a copy of the ACS's new, best-selling Professional Reference Book, *Microwave-Enhanced Chemistry: Fundamentals, Sample Preparation, and Applications* (1997) a \$109.95 value!

## Here's What You'll Learn in This Hands-On Course

- New laboratory microwave methods and equipment designs.
- Many non-intuitive fundamental and advanced concepts in microwave sample preparation.
- How to apply this knowledge to developing the decomposition and extraction procedures.
- How microwaves interact with solvents, acids, samples, and materials.
- How to evaluate national and international standard microwave methods including EPA (3015A, 3031, 3050B, 3051A, 3052, NPDES), ASTM (D4309-91, D4643-93, D5258-92, E1358-90, and F1317-90), Standard (3030K), SEMI 3038, French (V03-100), Chinese (C303-01T) methods, and draft and proposed new standard methods.
- Microwave decomposition procedures for botanical, biological, environmental, clinical, geological, metallurgical and specialized industrial matrices.
- Ways to approach and test the development of new decomposition procedures.
- How to work more safely to reduce the chance of unexpected and unwanted reactions.
- Recent microwave decomposition and extraction developments.
- The current state of the art in equipment, control, and practical techniques from major equipment manufacturers.

### **Bring Your Own Sample to Analyze!!**

You may bring a well characterized sample representative of your sample types to use for interactive classroom method development . For safety reasons, instructors must approve the sample type. You will help evaluate the sample matrix, its potential chemical difficulties, analytes, the instrumental technique, and numerous other factors. The instructors will guide you through a systematic approach to propose a method of microwave preparation. During the final laboratory period, selected samples will be digested according to the class developed procedures. This exercise will help you acquire the skills necessary to continue developing independent sample preparation methods—safely and effectively using many different kinds of laboratory microwave equipment configurations.

Note: The samples must be well characterized and of a non-toxic and non-explosive nature. Characterized means that the matrix is approximately known as well as the analytes and trace components. Samples of questionable composition will not be analyzed for safety concerns. It is not necessary for every participant to bring a sample, but you are encouraged to bring the information regarding your samples for classroom method development.

## **Earn University Graduate Course Credit**

All course attendees can earn three graduate course credits, in addition to ACS CEU credits, from the Duquesne University Department of Chemistry and Biochemistry or Environmental Science and Management program in the Bayer School of Natural and Environmental Sciences. To take advantage of this option, you will have to pay an additional tuition fee and you will be required to take a final exam. You can also take the course for a letter grade, pass/fail or audit basis. For more information, please call Mary Ann Quinn at (412) 396-6339.

## **Program Agenda**

Check-in will begin at 8:00 a.m. on July 30. The course will be taught from 8:30 a.m. to 5:00 p.m. each day. The course is divided approximately in half between formal instruction and laboratory sessions.

**Introduction**

- Why microwave decomposition?
- Overview of solvent chemical interactions

**Theoretical Overview**

- What are the important heat absorbing mechanisms and what influences them

**Basic Equipment**

- Design of microwave systems
- Vessel design and limitations
- Temperature measurement and reaction control systems
- Equipment configurations—alternatives and options

**Power and Temperature Relationships**

- Predicting conditions by understanding energy interactions
- Why partial pressure does not hold in the microwave—new fundamentals
- Role that heat loss plays during decomposition and chemical reaction
- Important equations and the mechanisms they represent
- Using temperature and pressure for control

**Encapsulation and Transfer of Microwave Procedures**

- Standardizing procedures within and between laboratories

**Clean Chemistry: Role and Control of the Analytical Blanks for ICP-MS and Ultra Trace**

- Applying clean chemical principles and achieve ultra-trace analysis
- Practical, cost effective clean chemistry methods and improve data

**Applications, Literature Examples and Current Research**

- Standard sample preparation procedures you can use
- Environmental applications and standard methods
- Geological and metallurgical matrix decomposition approaches
- Inorganic matrix decompositions
- Biological, botanical, and organic matrix decomposition for trace and ultra trace
- Organic extraction mechanisms, implementations and alternatives
- Discussion of applications that may be of interest
- Development of procedures for your sample

**Current and Emerging Research in Microwave Sample Preparation**

- Total microwave sample processing
- Focused microwave digestions
- Speciated extraction and analysis
- Organic extraction
- Integration with instruments

**Safety in Microwave Chemistry and Sample Preparation**

- Pressure can be evaluated, managed and manipulated
- Safety devices and how to apply them
- Problems that can be avoided
- Equipment handling for safety and efficiency
- Equipment failure and alternative approaches
- Case studies of previous accidents
- General discussion of concerns and application to your sample

**Development of Dissolution for Class Samples (Interactive Exercise)****Application of Microwave Theory to Individual Matrices, Samples, and Analytes****Ongoing Support for Microwave Sample Preparation**

- Sample preparation and reference materials, SAMPLEPREP®
- World Wide Web support and continued support (<http://www.sampleprep.duq.edu/sampleprep/>)

## About the Laboratories

Five to seven laboratories will be performed by the participants. Laboratories will also be structured toward the interests of the participants.

### Topics

- Individualized samples and method development
- Closed and open vessel alternatives
- EPA, ASTM, environmental, and other standard methods
- Speciation extractions
- Microwave organic extraction
- Clean chemistry techniques
- Decomposition of specific matrices
- Large samples, special analysis and/or unique applications

## Course Site/Housing

The course will be held in Mellon Hall, Duquesne University, Pittsburgh, PA. Participants should meet on Monday, July 30th, in the Mellon Hall lobby no later than 8:30 a.m. for check-in and orientation. A campus map will be included with your course confirmation material. Parking is available on campus for \$5.00 a day.

A limited block of hotel guest rooms has been reserved at the Ramada Plaza Suites, 1 Bigelow Square, Pittsburgh, PA 15219. Telephone (800) 225-5858 or (412) 281-5800. The rate is \$85 single or double suite. We urge you to call as soon as possible, mentioning that you will be attending the Duquesne/American Chemical Society program. Valet parking is available for \$6.00 a day (includes in and out privileges). The Ramada Plaza Suites is about two blocks from the Duquesne University Campus and about 15 miles (20 minutes) from the Greater Pittsburgh Airport. Airport limousine service is available (about \$12).

## Fee

ACS member: \$1,650; Non-member: \$1,850

(Includes ACS Short Course tuition, an extensive course manual, a professional reference book, laboratory supplies, reference material and lunch each day. A guide of activities for Pittsburgh during the week of the course will also be available.)

## About The Faculty

**Dr. H. M. (Skip) Kingston** is Professor of Analytical Chemistry at Duquesne University in Pittsburgh, Pennsylvania. For the past 20 years, Dr. Kingston has been actively involved in advancing microwave sample preparation through fundamental research and method development including standardizing these procedures and holds a registered trademark for SAMPLEPREP. He has taught the ACS laboratory microwave courses for the past 14 years. From 1976–1991 he was a Supervisory Research Chemist in the Analytical Research Division of the National Institute of Standards and Technology. He developed, co-edited, and co-authored the first and second ACS Professional Reference Books on microwave sample preparation, published in 1988 and 1997. A pioneer in the development of new analytical methods, he is the recipient of three R&D 100 awards for the development of the “Microwave Dissolution system” (1987), “Chelation Chromatography System” (1989), and “Speciated Isotope Dilution Mass Spectrometry” (1996). In sample preparation research he has received the 1988 “Pioneer in Laboratory Robotics” award, the Department of Commerce Bronze Medal in 1990, the 1990 NIST Applied Research Award, the 1991 Award of Merit from the Federal Laboratory Consortium for Technology Transfer, and the 1995 Duquesne University President’s Award for Excellence and the 2001 Bayer School of Natural and Environmental Sciences Award for Excellence in Scholarship and the 1998 EPA Service to Others Award. Dr. Kingston has participated in drafting or updating more than 12 national and international standard methods for agencies and organizations such as the EPA, SEMI and ASTM based on microwave enhanced chemistry. Dr. Kingston also serves on a number of academic, industrial, and government advisory boards. Dr. Kingston is one of the highest ranked instructors in the ACS Continuing Education Program.

### Assistant Instructors and Laboratory Instructors

**Ms. Sejal Iyer** is currently working on her doctoral research in analytical chemistry with Dr. Kingston. Her research focuses on microwave extraction of environmental and clinical materials. She is developing integrated extraction methods that enhance the overall efficiency of sample preparation in this area. Ms. Iyer is responsible for the microwave demonstration laboratory manual and the laboratory experience in the course and also has conducted an upgrade of the Web Site Sampleprep Web for the course.

**Dr. Ye Han** is an analytical chemist now with Metara Inc. and has done research in Dr. Kingston’s Research Group for five years focusing on ICP-MS, and the development of new standard EPA speciated methods. Dr. Han has developed ultra-trace clean chemistry for semiconductor, clinical and medical research.

**Mr. Mike Moses** is Regional Support Manager at Milestone, Inc. He has 15 years of extensive experience in all types of digestion and sample preparation techniques and has developed and written a comprehensive library of more than 900 applications for conventional and microwave decomposition.

**Mr. Mizanur Rahman** is currently working on his PhD. in analytical chemistry with Dr. Kingston. His research focuses on species extraction and analysis. He has experience with mercury analysis and has participated in the group developing the draft EPA method in this area.

**Dr. Amit Chatterjee** is a Research Scholar with Dr. Kingston’s Research Group at Duquesne University. He is conducting research on mass spectrometric ultra-trace elemental and speciation analysis on semiconductor processes.

