

Gas Chromatography/Mass Spectrometry

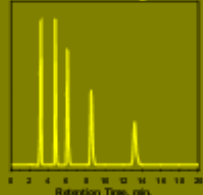
(4 Day On-Site Short Course)

Course Outline

Goals: To provide a comprehensive overview of the basic operating principles of high resolution gas chromatography, basic mass spectrometry and the application of these techniques in tandem to analyze drugs. The basic course outline is presented below.

Offerings: This four day on-site short course can be taught in one of two different modes depending on the individual preference of the sponsor. In the first mode, a balanced treatment of the topics outlined below will be presented. In the second mode, the different topics will be emphasized or deemphasized depending on the skill level of the attendees. In this latter mode, an initial questionnaire and skills assessment (i.e., taking about 15 to 20 minutes) will be carried out. Any information obtained will be used to help provide a customized treatment of the material will be

- A. Fundamentals of Separation Design
 1. Overview - Differential Migration Methods
 2. Separation Mechanisms/Techniques
 3. Causes of Non-Ideal Effects
 4. Analyte Considerations
 5. Choosing a Separation Strategy
 - B. Analyzing Legal and Illicit Drugs
 1. Important Physical/Chemical Properties
 2. Structural Considerations
 3. Method Selection and Reliability
 - C. High Resolution Gas Chromatography
 1. Basic Theory
 - a. Solute Migration
 - b. Band Spreading and Solute Resolution
 - c. Non-Ideal Effects
 - d. Instrumentation and Performance
 2. Columns
 - a. Fundamental Considerations
 - b. Column and Phase Selection
 - c. Column and Phase Stability
 - d. Separation Design
 - e. Optimizing Performance
 - f. Recognizing and Minimizing Problems
 3. Instrumentation
 - a. Inlet Systems
 - b. Oven and Temperature Control
 - c. General and Specialty Detectors
 - d. GC/MS
 4. Separation Design
 - a. Sample Considerations and Preparation
 - b. Choosing and Optimizing Conditions
 - c. Temperature and Pressure Programming
 - d. Assay Performance and Ruggedness
 5. Routine Maintenance and Troubleshooting
- D. Basics of Mass Spectrometry
 1. Basic Theory
 - a. Ionization Mechanisms
 - b. Ion Separation Mechanisms
 - c. Detection
 2. Instrumentation
 - a. Spectrometer Configurations
 - b. Ionization Sources
 - c. Analyzer Designs
 - d. Interfaces and Solute Transfer
 - e. Detection
 - f. Spectrometer Design vs. Performance
 3. Optimizing and Maintaining Performance
 - a. Ionization Techniques
 - b. Tuning and Calibration
 - c. Qualitative vs. Quantitative Information
 4. Structure Elucidation
 - a. Fragmentation Mechanisms
 - b. Information and Ionization Approach
 - c. Variability and Interferences
 - E. Methods Development/Drug Analysis
 1. Sample Considerations
 - a. Quantity and Homogeneity
 - b. Storage and Analyte Stability
 - c. Sample Preparation and Matrix Effects
 - d. Derivatization
 2. Quantitation
 - a. Standards
 - b. Calibration Procedures
 - c. Interferences
 - d. Recovery
 - e. Post Processing Procedures



Gas Chromatography/Mass Spectrometry

(4 Day On-Site Short Course)

- F. Remote Operation of Instrumentation
1. System Setup
 - a. Hardware Requirements
 - b. Software Requirements
 2. Security Issues

Course Instructors

Roger K. Gilpin is the Mead Distinguished Professor and Executive Director of Brehm Research Laboratories, Wright State University. Prior to this, he was Dean of the College of Science and Mathematics at WSU. He also is Director of the Consortium for Environmental and Process Technologies and President and a Co-Founder of Select-O-Sep, LLC.

Dr. Gilpin received his B.S. degree in Chemistry from Indiana State University in 1969 and his Ph.D. degree in Analytical Chemistry from the University of Arizona in 1973. From 1973 to 1978 he was employed as Senior Scientist and then as Group Leader of Analytical Chemistry in the Research Division of McNeil Laboratories. In 1978, Dr. Gilpin joined the faculty of Kent State University and was Professor and Chairman of the Department from 1985 to 1996.

Professor Gilpin's research interests are in fundamental and applied aspects of gas and liquid chromatography, environmental, pharmaceutical, and biomedical analysis of physiologically important compounds, chromatographic and spectrometric studies of chemically modified surfaces, characterization of the interfacial properties of materials, fundamental and applied aspects of electrospray and other mass spectrometric ionization techniques, and other liquid based capillary separation techniques.

He has published about 200 papers and presented over 450 talks at national and international scientific conferences, is Associate Editor of the *Journal of Chromatographic Science* and serves on its Editorial Advisory Board, was Section Editor for the Pharmaceutical Analysis Section of the Encyclopedia of Analytical Chemistry and is a member of the Special Emphasis Panel for NIH related to technology transfer where he has served for 22 years. He has been the principle author on the annual reviews of "Pharmaceutical and Related

Drugs," published in *Analytical Chemistry* on a continuous basis since 1973.

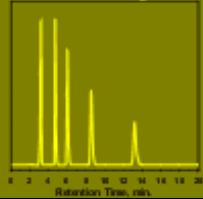
Joseph G. Solch is Laboratory Manager of Brehm Research Laboratory, Wright State University and a technical consultant to the Terran Corporation and Analytical Innovations, Inc. Likewise, he is Vice-President and a Co-Founder of Select-O-Sep, LLC.

Prior to this he earned his undergraduate degree in Chemistry and his graduate degree in Analytical Chemistry from Wright State University in 1974 and 1979, respectively. He has over 25 years of experience in the operation, maintenance, and application of high resolution gas chromatograph, published approximately 70 manuscripts, and presented a similar number talks at scientific conferences.

His research interests are in fundamental and applied aspects of gas chromatography and mass spectrometry, the utilization of high resolution GC/MS techniques for assaying physiologically important compounds, the automation and remote access/operation of laboratory instrumentation, and the development of virtual laboratories for remote measurements of analytes and teaching purposes.

Suggested Scheduling

- A. Monday
 1. First Session (80 min., lectures)
 2. Morning Break (15-20 min.)
 3. Second Session (80 min., lectures)
 4. Lunch (60 min.)
 5. Third Session (80 min., lectures)
 6. Afternoon Break (15-20 min.)
 7. Fourth Session (40 min., lectures)
 8. Fifth Session (40 min., open discussions)
- B. Tuesday
 1. First Session (80 min., lectures)
 2. Morning Break (15-20 min.)
 3. Second Session (80 min.)
 4. Lunch (60 min.)
 5. Third Session (80 min.)
 6. Afternoon Break (15-20 min.)
 7. Fourth Session (40 min.)
 8. Fifth Session (40 min., open discussions)
- C. Wednesday



Gas Chromatography/Mass Spectrometry

(4 Day On-Site Short Course)

1. First Session (80 min.)
2. Morning Break (15-20 min.)
3. Second Session (80 min.)
4. Lunch (60 min.)
5. Third Session (80 min.)
6. Afternoon Break (15-20 min.)
7. Fourth Session (40 min.)
8. Fifth Session (40 min., open discussions)

- 4 Break (7-10 min.)
- 5 Third Session (60 min., lectures)
6. Lunch (60 min.)
7. Forth Session (60 min., lectures)
8. Break (7-10 min.)
9. Fifth Session (40 min., lectures)
10. Sixth Session (40 min., open discussions)

D. Thursday

1. First Session (80 min.)
2. Morning Break (15-20 min.)
3. Second Session (80 min.)
4. Lunch (60 min.)
5. Third Session (80 min.)
6. Afternoon Break (15-20 min.)
7. Fourth Session (40 min.)
8. Fifth Session (40 min., open discussions)

D. Thursday

1. First Session (60 min., lectures)
2. Break (7-10 min.)
3. Second Session (60 min., lectures)
- 4 Break (7-10 min.)
- 5 Third Session (60 min., lectures)
6. Lunch (60 min.)
7. Forth Session (60 min., lectures)
8. Break (7-10 min.)
9. Fifth Session (40 min., lectures)
10. Sixth Session (40 min., open discussions)

or

A. Monday

1. First Session (60 min., lectures)
2. Break (7-10 min.)
3. Second Session (60 min., lectures)
- 4 Break (7-10 min.)
- 5 Third Session (60 min., lectures)
6. Lunch (60 min.)
7. Forth Session (60 min., lectures)
8. Break (7-10 min.)
9. Fifth Session (40 min., lectures)
10. Sixth Session (40 min., open discussions)

B. Tuesday

1. First Session (60 min., lectures)
2. Break (7-10 min.)
3. Second Session (60 min., lectures)
- 4 Break (7-10 min.)
- 5 Third Session (60 min., lectures)
6. Lunch (60 min.)
7. Forth Session (60 min., lectures)
8. Break (7-10 min.)
9. Fifth Session (40 min., lectures)
10. Sixth Session (40 min., open discussions)

C. Wednesday

1. First Session (60 min., lectures)
2. Break (7-10 min.)
3. Second Session (60 min., lectures)

Logistics/Site Preparation

All local site preparation and associated site costs are the responsibility of the course purchaser. The course instructors will provide their own computer/digital projection equipment. However, the purchaser will need to arrange for a projection screen or usable white or nearly white wall surface and if necessary audio equipment (the latter typically is not required in a small room setting). Prior to arrival the course material (i.e., copies of the power point slides) will be shipped in electronic printable format. Unless otherwise specified, it is the responsibility of the purchaser to prepare hardcopies of the electronic materials and distribute them to their employees. However, if bound hardcopy manuals are preferred, these can be supplied at a cost of \$24.95/copy plus shipping.