Short Course on Supercritical Fluid Chromatography

October 17, 2018, 10:00 to 17:30 PM

Course Description

This course will focus on fundamentals and advances in packed-column supercritical fluid chromatography (pcSFC) employing carbon dioxide-based mobile phases. Particular emphasis will be directed toward modern instrumentation and method development in both analytical-scale and preparative-scale SFC, including chiral separations. The advantages of SFC will be illustrated with applications in pharmaceuticals and other fields where SFC currently plays, or can play, a critical role such as metabolomics, specialty chemicals, biodiesel, foods, natural products, and fragrances. The course will also touch on recent advances in supercritical fluid extraction (SFE). The course is taught by three scientists (1 academic, 2 industrial) who have spent the majorities of their careers working in SFC and SFE.

Course Outcomes

Course attendees will learn the following:

- Advantages of SFC over HPLC-based separations and purifications
- How to develop analytical SFC separations and preparative SFC purifications
- How carbon dioxide-based mobile phases improve efficiency and throughput while reducing costs of analysis and purification
- How to choose stationary phases for achiral and chiral SFC separations
- How to use SFC to increase the environmental friendliness of separations
- Which molecules are suitable for SFC, and which molecules are not suitable
- How to modify HPLC processes to exploit the advantages of SFC
- The role of carbon dioxide, modifiers, and additives in SFC separations
- Options for detection in SFC, with special emphasis on SFC/MS
- The latest developments in SFE
- The future of SFC and SFE

Who Should Take This Course

The course is for researchers and managers interested in increasing the efficiency of analyses and separations while reducing costs.

- Academic and industrial separation scientists
- Process chemists
- Laboratory and R&D managers working in the following areas:
    - Pharmaceuticals
    - Specialty chemicals
    - Advanced materials
    - Food technology
    - Metabolomics

- Biodiesel technology
- Natural products
- Fragrances
- Medical research

Course Prerequisite

Knowledge of chromatographic principles is desirable. Actual experience or knowledge of SFC is not required.

Course Agenda

(see below for detailed agenda)

10:00 – Introduction of Instructors and Course Orientation

10:10 – Introduction to Supercritical Fluids and SFC – Taylor

11:10 - Instrumentation in Analytical-Scale SFC - Pinkston

11:40 – Method Development for Chiral Analytical SFC – Miller

12:30 - Lunch

13:10 – Method Development for Achiral SFC – Pinkston

14:10 – Preparative SFC - Miller

15:10 – Break

15:25 – Detection in Packed Column SFC - Pinkston

16:15 – Recent Advances in Supercritical Fluid Extraction - Taylor

17:15 – Discussion, Questions & Answers

17:30 – Adjourn

Detailed Course Agenda

10:00 – Introduction of Instructors and Course Orientation

10:10 – Introduction to Supercritical Fluids and SFC – Taylor

Properties of supercritical fluids, phase behavior, packed columns vs. open tubular SFC, effect of diffusivity and viscosity on efficiency and speed of separation, CO_2 vs. other mobile phases, column pressure drop, retention, subcritical separations, safety considerations

11:10 - Instrumentation in Analytical-Scale SFC – Pinkston

Differences between instrumentation in SFC and HPLC, flow vs. pressure control, fluid delivery and pumping, injection in SFC, tubing and connections, column & mobile phase thermostating and detection, backpressure regulation, waste disposal, safety considerations

11:40 – Method Development for Chiral Analytical SFC – Miller

Introduction to chirality, chiral recognition, advantages of SFC vs. HPLC, stationary phase and mobile phase screening, method development strategies

12:30 - Lunch

13:10 – Method Development for Achiral SFC – Pinkston

Intermolecular interactions, retention vs. selectivity, choices in modifiers & additives, polar and apolar stationary phases, generic SFC methods, method development flow chart for all users

14:10 – Preparative SFC – Miller

Introduction to small-molecule purification, HPLC vs. SFC; advantages and disadvantages, effect of solubility in preparative SFC, preparative sample injection options, chiral and achiral preparative SFC, mass direction purification, preparative SFC safety concerns

15:10 – Break

15:25 – Detection in Packed Column SFC - Pinkston

Detection pre- and post-decompression, UV/VIS & spectroscopic detection, non-specific detection such as light scattering, forms of interfacing to atmospheric pressure ionization mass spectrometry, limits of detection & linearity, comparisons with HPLC

16:15 – Recent Advances in Supercritical Fluid Extraction – Taylor

Basic principles of SFE, analytical applications including recent advances in polymeric materials and polymer additives, aqueous metal ions such as mercury, natural products like tobacco and cellulose, and advantages of on-line hyphenated processes such as SFE-SFC, SFE-GC, and SFE-HPLC.

17:15 – Discussion, Questions & Answers

17:30 – Adjourn

Course Instructors

Larry Miller

Larry Miller is a Principal Scientist in the Discovery Attributes Sciences group at Amgen in Cambridge, MA, USA. He has over 30 years of experience performing small-molecule, achiral and chiral purifications at the milligram to multi-kilogram scale, using preparative HPLC, SFC, steady-state recycle (SSR), and simulated moving-bed (SMB) chromatography. During his career he spent twenty years at Searle/Pharmacia and has spent the last fourteen years at Amgen. At Amgen he manages a center of excellence at two R&D sites that is responsible for all small molecule purification support for Amgen's discovery and development departments.

His early purification experiences used HPLC, but over the past fifteen years he has moved heavily into SFC and serves as a strong ambassador for the expansion of SFC into new industries and areas of separation science. Larry has more than 30 peer reviewed publications and over 35 presentations at scientific meetings and also serves as co-instructor for SFC short courses in the US, Europe and Asia. From 2011 to 2016 he served as president of the Green Chemistry Group and is currently Co-Chair of the Scientific Committee as well as a Board Member of the Green Chemistry Group.

J. David Pinkston

J. David Pinkston is a Principal Scientist in Kellogg's Global Chemistry organization. His research interests include the development and application of various forms of pressurized fluid chromatography and the coupling of these separation methods to intelligent detectors, such as mass spectrometry. Most recently, his interests have focused on flavor/aroma chemistry; trace-level contaminants; and oil/fat chemistry, degradation, and preservation.

David received his Ph.D. in 1985 from Michigan State University. He worked on separations and mass spectrometry under the direction of J. Throck Watson and John Allison.

He was Chair of the American Chemical Society (ACS) Division of Analytical Chemistry (ANYL) in 2002–2003, was ANYL's Program Chair for the Fall 2002 and Spring 2003 National ACS Meetings, and has served on the Executive Committee of ANYL Subdivision of Chromatography & Separations Chemistry. David is now on the Board of Directors of the Green Chemistry Group, the organizer of the annual SFC meeting.

David has authored or co-authored over 55 publications and presented over 130 lectures, the majority of which are about SFC and SFC/MS. He has taught various versions of the SFC short course in the USA, Europe, and Asia since the early 1990s. David loves downhill skiing and is an avid cyclist.

Larry T. Taylor

Larry T. Taylor is Emeritus Professor of Chemistry, Virginia Tech (Blacksburg, VA) and President of Applied Analytical, Inc. He is a member of the editorial boards for the Journal of Chromatographic Science, Chromatographia, and the Journal of Supercritical Fluids. He was a member of the Organizing Committee for the 4th through the 10th International Symposia on SFC/SFE during the 1980`s. More recently he has served as the Co-Chair with Larry Miller (Amgen) of the Scientific Committee for SFC 2008 (Zurich), SFC 2009 (Philadelphia), SFC 2010 (Stockholm), SFC 2011 (New York City), SFC 2012 (Brussels), SFC 2013 (Boston), SFC 2014 (Basel), SFC 2015 (Philadelphia), SFC 2016 (Vienna), SFC 2017 (Rockville, MD), and SFC 2018 (Strasbourg). Larry is the author or co-author of approximately 400 peer-reviewed publications, one book entitled “Analytical Supercritical Fluid Extraction”, and 12 patents. He presently serves as co-teacher of short courses addressed to SFC and SFE. Recent industrial partnerships have included: Waters Corp., Pfizer, Eli Lilly, Princeton Chromatography, ES Industries, Amgen, Applied Separations, Procter & Gamble, Merck, Wythe, and Abbott Labs.