HPLC Analytical Method Development and Validation

22 & 23 June 2020, London UK

9 & 10 November 2020, London UK

Good HPLC methods must satisfy both technical requirements (sensitivity, specificity, linearity, accuracy and precision) as well as business needs (reliability in routine use and a run time appropriate to the number of samples to be tested). These requirements are equally important in both a development and routine QC context: decisions during drug development must be based on reliable data, and routine QC testing, including stability studies, must control risks to product quality and patient safety.

This course presents a logical, step-wise approach to the development of HPLC methods (Day 1) and then explains how to validate chromatographic methods in line with regulatory expectations and best practice (Day 2).

The course is intended for analytical scientists who have experience of operating HPLC instrumentation.

Speaker:

Dr Mark Powell

Mark is a Fellow of the Royal Society of Chemistry with over twenty years' experience as an analytical chemist. His PhD project involved the characterisation of bitumen by chromatographic, spectroscopic and thermal methods, providing a good grounding in a wide range of analytical techniques.

He then worked for five years in the environmental industry, with responsibility for the development of analytical methods capable of quantifying very low levels of pollutants in drinking water and a variety of other sample types.

Having joined Liverpool John Moores University's School of Pharmacy and Chemistry in 1997 as a Senior Lecturer, Mark was responsible for the University's MSc programme in analytical chemistry, and was also active in research and consultancy.

In 2003, he joined the newly-formed Quay Pharmaceuticals, a contract research and manufacturing organisation specialising in early-stage drug development, where he was responsible for analytical development. Since 2010, as Scientific Manager, Mark was involved more generally with drug development programmes and also established collaborations with a number of UK universities and instrument manufacturers. His work at Quay has resulted in a number of published papers and presentations at scientific conferences.

HPLC Analytical Method Development and Validation

Course Programme

Day One

Revision of chromatographic theory
- Separation modes
- Factors affecting resolution
- Peak symmetry
- Band broadening
- The effect of particle size and extra-column volume on efficiency

Important chemical concepts
- Factors affecting analyte/stationary phase interactions (polarity, hydrogen bonding and pKa)
- Stationary phase endcapping

Analyte properties affecting pKa, solubility and detectability
Workshop: reading solute structures
Matrix properties
- Effect on extraction
- Chemical interference
- Selectivity and detector wavelength
- Injection solvent strength

Method performance requirements
- Defining method performance requirements
- Measurement uncertainty vs. specification limits

Sample preparation
- Selective sample preparation
- Choice of filter membrane
- Chemical and physical stability of samples

Developing the separation
- Starting conditions for different separation types
    * Neutral/ionisable organic molecules
    * Special cases
- Separation modes: isocratic, gradient, ion pairing/suppression, HILIC, aqueous normal phase, normal phase, ion exchange and size exclusion
- Retention mechanisms
- Choice of stationary phase (including silanol activity considerations)
- Mobile phase pH and solute pKa
- Choice of pH buffer
- Temperature effects
- Core-shell and UHPLC columns
- Detector selection

Gradient elution
- When to use gradient elution
- Significance of gradient delay volume
- Retention and resolution models in gradient separations
- Gradient profile optimisation
- The effect of column dimensions and temperature
- Step-wise gradient method development strategy

Case studies
Workshop: selecting starting conditions for method development

Day 2 – HPLC Method Validation

Regulatory guidance (ICH, US, EU, WHO)
Validation terminology
Setting meaningful acceptance criteria
- Acceptance criteria based on specification limits
- Measurement uncertainty and sources of error
- Typical acceptance criteria
- Lifecycle approach to method validation: Analytical Target Profile (ATP) and analytical control strategy

Experimental approaches to method validation
- Specificity: with and without impurity standards
- Linearity: best practice
- Use of spiking experiments
- Options for evaluating sensitivity
- Recommended robustness experiments

Phase-appropriate method validation

Workshop – setting method validation acceptance criteria
Writing effective analytical methods, validation protocols and reports
- Pre-validation check-list
- Contents of method and validation documentation
- Mistake-proofing analytical methods

Dealing with validation failures
Setting system suitability criteria
- Regulatory guidance
- Statistically-based methods

Verifying compendial procedures
- Regulatory guidance
- Approaches for different method types

Workshop – planning a method validation exercise

PharmaTraining

HPLC Troubleshooting
24 June 2020, London UK

Overview

The ability to identify and correct problems with HPLC or UHPLC separations is an important skill for practising chromatographers. In addition to the mechanical or electronic problems that can compromise the performance of instrument modules, changes in the chemical or physical characteristics of the column and/or unintentional changes in mobile phase chemistry can alter the quality of the separation.

This one-day course is designed to equip analytical scientists with the knowledge and tools required to correctly diagnose mechanical or chemical problems in HPLC or UHPLC separations. The course includes details of simple tests that can be performed to help identify the cause of the problem as well as simple precautions that can be taken to prevent faults occurring in the first place.

- Review of HPLC theory as it applies to troubleshooting and instrument maintenance
- Operating principles of each module in a HPLC/UHPLC system
- Performance qualification (PQ) is so important
- Techniques for systematic problem solving and instrument maintenance
- Preventing common hardware problems and method failures

Course Programme

Chromatographic Theory and Troubleshooting Principles
- Causes of adsorptive interactions in HPLC
- Chromatographic behaviour of acids and bases
- Revision of chromatographic theory
- Record-keeping
- Tips for effective troubleshooting
- Using information from system suitability tests
- Tubing and extra-column dispersion

Pump Problems
- Different pump types
- Common problems with HPLC pumps
- Flow rate and leak tests

Detector Problems
- Common detector types and characteristics
- Noise and drift tests
- Mobile phase absorbance in UV detection
- MS and evaporative detector-compatible mobile phases
- Effect of flow-cell volume
- Selecting appropriate data system settings

Mobile Phase and Gradient Mixing
- Mobile phase filtration – when and how?
- Deaeration
- Buffer selection
- Mixing solvents – miscibility and other considerations
- Mobile phase stability
- Gradient mixing and delay volume
- Gradient performance qualification tests

Autosampler and Column Thermostat Problems
- Autosampler types
- Common autosampler problems
- Causes of poor precision
- Curing carryover
- Column temperature effects

Column Problems
- Typical problems/symptoms
- Sample matrix effects
- Injection solvent composition and volume injected
- Column ageing
- Causes of peak asymmetry and retention time shifts
- Causes of high back pressure; sample filtration
- Sample stability and analyte adsorption
- Tests to troubleshoot problems with separation chemistry

Quantitation
- Peak integration and smoothing
- Advantages of second derivative plots
- Common integration errors

Workshop: Troubleshooting Examples

Who Should Attend?

Scientists working with HPLC who need to improve their understanding of HPLC instrumentation and troubleshooting techniques.
HPLC validation and maintenance staff.

Venue:

DoubleTree Hilton Hotel Islington, 60 Pentonville Road, London, N1 9LA
Website: www.http://doubletree3.hilton.com/ Close to Kings Cross/St Pancras Stations

Please note accommodation is not included in course fee.

Accommodation and travel directions are available on our website

For 5 or more staff requiring training it may be beneficial to run a course in-house.

The benefits of running a course in-house:

- Save on travel or accommodation costs
- Customised content to meet your requirements
- Big print savings on course material - especially with larger groups
- Courses arranged for large groups up to 24 staff
- Tutorials available for small groups of 2 or 3 staff
- Meet course speakers in advance to discuss design and content

Contact **Judy Callanan at any time to discuss**
Ph: 0044 (0)20 8133 2605, **Email:** judy@pharma-training-courses.com

Course Programme 2020

Hands-on Tablet Development including the principles of pre-formulation, formulation and process development
1, 2 & 3 April *and* 2, 3 & 4 December 2020, **Croydon Greater London**
***

Pharmaceutical Dissolution Testing – a Hands-on Course
12, 13, 14 & 15 October 2020, **London**
***

QbD and Lifecycle Management of Analytical Methods
21 & 22 May 2020, **London**
***

Stability Testing in Pharmaceutical Development and Manufacture
8 & 9 June 2020 **London**
***

HPLC Analytical Method Development and Validation
22 & 23 June 2020 **London**
***

Tablet Compaction Analysis and how to improve your products
26 June 2020 and 19 November 2020 **London**
***

HPLC Troubleshooting 24 June 2020 **London**
***

Pharmaceutical Packaging – an introductory course
25 & 26 June *and* 30 November & 1 December 2020 **London**
***

Introduction to the Formulation and Stabilisation of Protein and Peptide Drugs
14 & 15 September 2020 **London**
***

Latest Advances in the Formulation & Stabilisation of Protein and Peptide Drugs
16 & 17 September 2020 **London**
***

Powder Technology for Pharmaceutical Development and Manufacturing
23, 24 & 25 September 2020 **London**
***

Pharmaceutical Dissolution Testing – a Hands-on Course
13, 14, 15 & 16 October 2020 **London**
***

Parenteral Products
tba 2020 **London**
***

Pharmaceutical Granulation and Compression
tba 2020 **London**
***

GMP Auditor Training for Quality Systems
tba November 2020 **London**
***

HPLC Analytical Method Development and Validation
9 & 10 November 2020 **London**
***

Development of Stability-Indicating HPLC Methods
11 November 2020 **London**
***

Pharmaceutical Aerosols, Dry Powder Inhalation Systems and Nasal Delivery Devices
tba 2020 **London**
***

Pharmacokinetics in Drug Development - an integrated approach
23 & 24 November 2020 **London**

PharmaCourses Ltd
Suite 1327 Kemp House
152 City Road
London
UK

Tel: ++44 (0)20 8133 2605

Email: info@pharma-training-courses.com

Keep up to date with industry requirements

REGISTRATION DETAILS:

HPLC Troubleshooting, 24 June 2020 *London*

Early-bird fee: 1 day course £540.00 (+ VAT if applicable, see notes on VAT)
For registering and paying by 11 May 2020
Full Fee: 2 day course £600.00 (+ VAT £240.00 if applicable, see notes on VAT)

HPLC Analytical Method Development and Validation, 22 & 23 June 2020 *London*
Early-bird fee: 2 day course £1080.00 (+ VAT £216.00 if applicable, see notes on VAT)
For registering and paying by 11 May 2020
Full Fee: 2 day course £1200.00 (+ VAT £240.00 if applicable, see notes on VAT) if registering after 11 May 2020

HPLC Analytical Method Development and Validation, 22 & 23 June 2020 *and* **HPLC Troubleshooting, 24 June 2020 *London***

BOOK both course for a reduced rate -
Early-bird fee: 3 day course £1440.00 (+ VAT if applicable, see notes on VAT)
For registering and paying by 11 May 2020
Full Fee: 3 day course £1620.00 (+ VAT if applicable, see notes on VAT)
if registering for both courses after 11 May 2020

UK: Under UK law all UK-based applications are subject to VAT at the prevailing rate however most UK VAT registered companies/organisations can reclaim this tax.

EU: With effect from 1 January 2011 applications from delegates whose companies are based in EU countries will not be subject to VAT **PROVIDED THAT** valid VAT ID details are provided at the time of booking, otherwise VAT will be charged.

OTHER: With effect from 1 January 2011 applications from delegates whose companies are based outside of the UK/EU will be outside the scope of VAT, ie no VAT is charged or payable.

Methods of Payment available:

- Cheque (**Please make payable to "PharmaCourses Ltd")**
- Bank transfer
- Credit/Debit Card (If paying by Credit Card please register online)

Online Registration is available on our website:
www.pharma-training-courses.com

Data Protection
PharmaCourses Ltd gathers personal data in accordance with the UK Data Protection Act 1998 and we may use this to contact you by telephone, fax, post or email to tell you about other products and services. If you have any queries or want to update any of the data that we hold then please contact us.

Terms and Conditions: Delegate fees: Fees for this programme are shown overleaf. Delegate fees are inclusive of course documentation, refreshments and lunch. Payment of the registration fee must be paid within 14 days of commencement of the course. Upon receipt of payment, a proof of payment will be sent to you. **Cancellation Policy:** Full refunds less a handling fee of £100 will be made for cancellations received in writing within 28 days of the commencement of the course. Refunds of 50% will be made for cancellations received in writing between 28 and 7 days prior to the commencement of the course. Regrettably no refunds will be made after 7 days prior to commencement of the course. Substitutions can be made at any time.
Liability: PharmaCourses Ltd reserves the right to change the programme, speakers, date or venue without notice or cancel the event. If cancellation occurs delegates will be notified as soon as possible and will receive full refund of fees paid. PharmaCourses Ltd will not be responsible for any airfare, accommodation or other travel costs incurred.
PharmaCourses Ltd is Registered in England and Wales No. 08509096

PharmaCourses Ltd
Suite 1327 Kemp House
152 City Road
London
UK

Tel: ++44 (0)20 8133 2605

Email: info@pharma-training-courses.com