HTC-15

24th - 26th January 2018
Cardiff, UK

15th International Symposium on

Hyphenated Techniques in Chromatography and Separation Technology

www.htc-conference.co.uk

kvcv
Royal Society of Chemistry
BMSS British Mass Spectrometry Society
Analytical Chemistry Trust Fund
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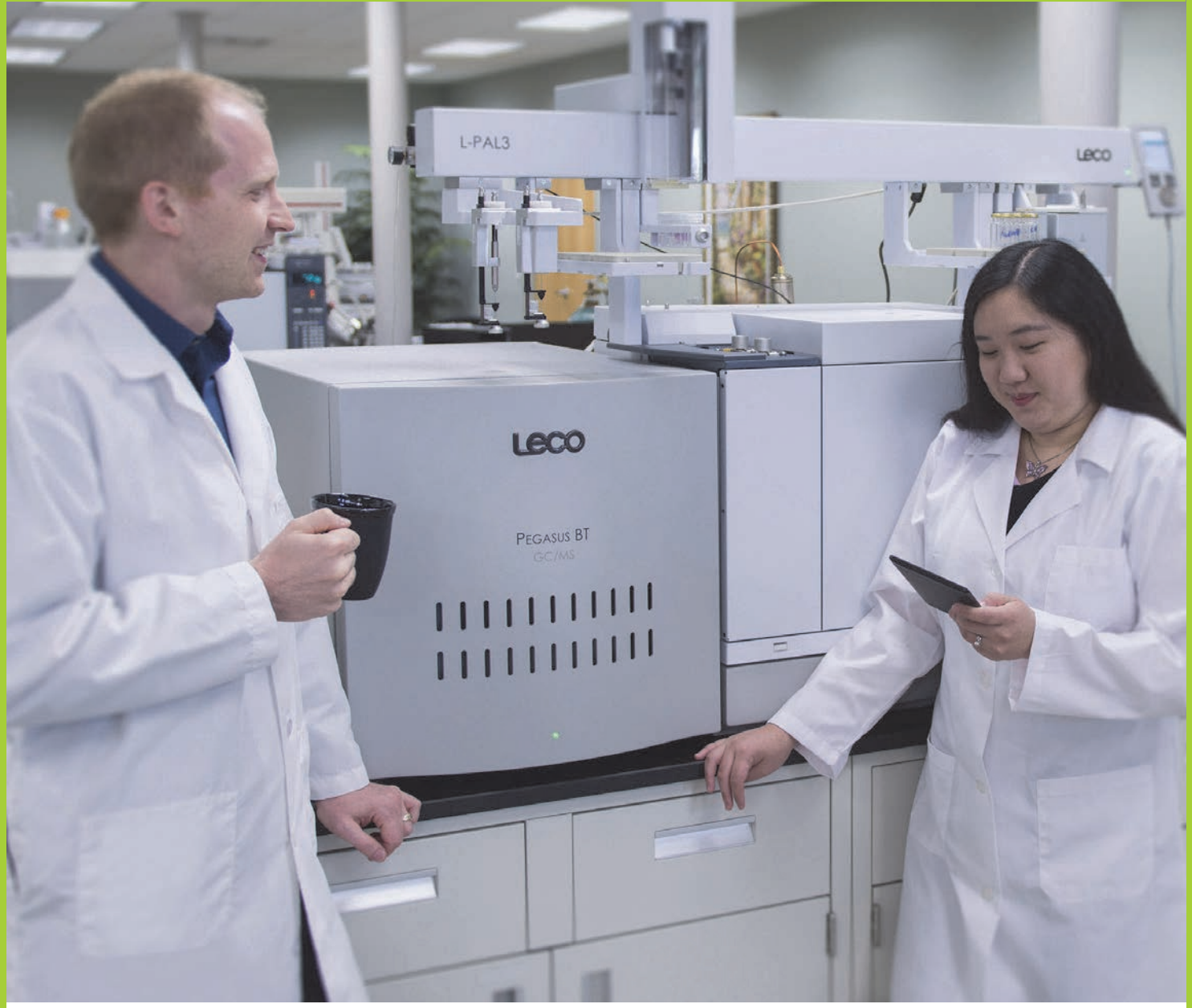
ILM Exhibitions
Chromatography Today
ChromSoc The Chromatographic Society - founded 1956



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Dear Delegate

Croeso i Gaerdydd. Croeso i HTC. - Welcome to Cardiff. Welcome to HTC.

We are very excited to bring HTC to the UK for the second time and to be hosting this meeting in the capital city of Wales. This year's Conference will be at Cardiff City Hall, a beautiful Edwardian City Hall that opened in 1904. So the venue opened within a year of Mikhail Twsett's lecture "On a New Category of Adsorption Phenomena and Their Application to Biochemical Analysis", the birth of chromatography. We are very grateful to the KVCV for trusting the Separation Science Group of the Royal Society of Chemistry to host the 15th HTC Conference in Wales.

This year's Conference will include 5 Plenary lectures, 16 Keynote lectures, 8 Tutorial lectures, 48 Oral presentations and 24 early career researcher (ECR) presentations plus over 70 posters. We are very pleased to have obtained significant support from our meeting sponsors who are recognised in this booklet and on the conference website. This has allowed us to achieve our aim to support all of our ECR speakers, and others, to attend and contribute to HTC. We were committed to creating opportunities for the next generation scientists to present within this meeting -after all they are the future of our science. Robert Smits, recognised as the father of HTC, was passionate that HTC should be a collaboration between industry and academia and HTC 15 continues with that tradition with over 39% of the oral programme from non-academic researchers.

We are also pleased to report that we have delegates form 21 countries, from all corners of the world, making HTC-15 a truly International event. Further, the gender balance of the meeting is ~60:40 male:female and this is matched within the presenters at HTC, meeting another important criterion for the organising committee.

An integral part of the meeting will be a full trade show with 24 companies exhibiting. There is time set over Wednesday, Thursday and Friday and Tuesday lunchtimes visit the exhibition and posters. Tea and coffee will also be served in the Exhibition area mid-morning, mid-afternoon and at lunchtimes so please take the opportunity to support the poster presenters and exhibitors.

We hope you will find this style attractive and informative and we look forwards to your feedback on this and all aspects of the Conference.

HTC-15 will be preceded by three short courses, building on the successful SFC short course at HTC-14 in Ghent. This year the short course topics are 'An introduction to Biopharma', 'SFC – from theory to application' and 'Statistical analysis of chromatographic data'. All hot topics and drawing a lot of attention.

An important part of HTC has always been the social networking where all delegates mingle and share experiences and meet friends and colleagues, old and new. Keeping with tradition there will be a Beer Degustation evening on the Wednesday evening at The Yard, Brains Brewery's main outlet, situated in the old Brewery quarter. The highlight of the social programme will be the Conference Gala Dinner at the Museum of Wales, next door to City Hall. The Dinner will be preceded by a drinks reception in the viewing galleries so don't lose your ticket.

This meeting would not take place without the input of the organising and scientific committees, with particular mentions to Tom Lynch and Ruth Godfrey here, without their time and dedication to the cause we would not have the fantastic meeting to attend. Similarly the excellent exhibition would not be what it is today without the efforts of David Hellyer and his team. We are also very pleased with our choice of PCO, working with ILM has been a pleasure and made the whole process viable.

It is just left for me to say 'diolch yn fawr' to you all for attending this meeting and supporting HTC. I hope you enjoy all aspects of the meeting and the city of Cardiff too.

Professor John Langley

HTC Chair

Free wifi is available in the hall, please use **'Cardiff Free Wifi'**

HTC Opening Times

Wednesday 24th Jan - **8.00am – 6.00pm**
Thursday 25th Jan - **8.00am – 5.30pm**
Friday 26th Jan - **8.30am – 4.30pm**

Exhibition Times

Wednesday 24th Jan - **9.45am – 6.30pm**
Thursday 25th Jan - **9.45am – 4.30pm**
Friday 26th Jan - **9.45am – 1.00pm**

HTC-15 Sponsors

The HTC Committee would like to thank it's sponsors for supporting this event

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Lunch Seminars

SCIEX Lunch Seminar: Mapping Protein Modifications Using Novel MS Strategies

Wednesday 24th January at 12.00 - 14:00pm - Council Chamber

Visit stand 8 to register to attend our Lunch Seminar "Mapping Protein Modifications Using Novel MS Strategies", presented by Dr Steve Lock and Neil Walsh (SCIEX, Warrington, UK), and Prof. Claire Eyers (University of Liverpool, UK).

In this workshop, we will start by showing how SWATH Acquisition is being used to help proteomics researchers answer questions. We will then discuss how capillary electrophoresis coupled to mass spectrometry (CESI-MS) is helping scientists detect post translation modifications which may have been missed by LC-MS separation and look at how this technique can be used to drive quantitation limits lower using injection techniques only open to CE. Finally, we will finish by a look into the future on how CESI-MS is now starting to be used to separate variants and identify proteoforms at the intact level eliminating the need for digestion.

The format of this Lunch Seminar is 40 minutes, and lunch will be provided.

Sandwiches, Cookies and Drinks included.

VRS Recruitment Lunch Seminar: Securing YOUR Perfect Job!

Wednesday 24th January at 12.30pm - Room D

Workshop to include:- where to find scientific jobs, how to apply, CV and cover letter writing, Interview preparation, and tips to negotiate improved offers!

About VRS:- For the last 15 years VRS has been the UK's market leading Analytical Chemistry recruitment consultancy. In addition to specialising in jobs within the field of mass spectrometry and chromatography, VRS has now expanded their recruitment services. VRS are now proud to offer recruitment solutions covering Life Sciences, Engineering and Sales!

Who should attend? Delegates looking for a new job and/or exhibitors looking to hire!

Ellutia Lunch Seminar: MAX a New Concept in GC-FTIR

Thursday 25th January at 12.30pm - Room A

GC-FTIR has not been prevalent in the analytical laboratory mostly due to its lack of sensitivity. MAX GC-FTIR is a new concept that uses signal averaging and peak integration to improve the sensitivity by up to 50 times compared to traditional GC-FTIR. The improved sensitivity combined with qualitative, quantitative and structural information of FTIR makes the MAX a unique alternative to GCxGC and GC-MS. This seminar will cover the technology theory and some application areas of this exciting new detector.

Free Scones, Cakes and Refreshments included.

Shimadzu Lunch Seminar: Smart & Green Hyphenated Solutions

Thursday 25th January at 12.30pm - Room B

Presentation 1: Title: SFC-MS vs LC-MS: The power of Selectivity and the Strength to choose
Presenter Name: Gesa SCHAD
Company/Organisation: Shimadzu Europa GmbH

Presentation 2: Title: Solutions for allergens analysis in fragrances
Presenter Name: Mariosimone Zoccali
Company/Organisation: University of Messina

Attendees will receive a free gift.

Agenda

TUESDAY 23RD JANUARY

Short Course Registration open from 9.30am

Short Courses

10am – 4pm – Ferrier Hall
Introduction To Biopharmaceutical Analysis

10am – 4pm – Room L
Supercritical Fluid Chromatography: From Theory To (Industrial) Application

10am – 4pm – Council Chamber
Statistical analysis of chromatographic data: a practical guide

2pm – General Registration open

We have purposely left the Tuesday evening free of organised events to allow delegates to explore the bars, restaurants and the beautiful city of Cardiff and Cardiff Bay (Home of the Welsh Assembly and Doctor Who!). There will be an informal gathering at The Urban Tap, 25 Westgate St, www.tinyrebel.co.uk/bars/cardiff/, from 20.00 for those who may want to catch-up with friends, old and new."

WEDNESDAY 24TH JANUARY

8am – 8.45am – Registration Open

8.45am – 9.00am
Opening Ceremony – Lower Hall

9.00am – 9.50am
Plenary: "Why Do We Still Use Silica?" KNOX Award Lecture by Prof Peter Myers, University of Liverpool – Lower Hall

9.50 – 10.30am
Coffee Break, Exhibition and Posters – Marble Hall and Assembly Room

10.30 – 12pm
Conference sessions in Lower Hall Syndicate Room D and Ferrier Hall

12pm – 2pm
Lunch, Vendor Seminars, Exhibitions and Posters - Marble Hall and Assembly Room

12.30pm – 1.10 pm
VRS Recruitment Lunch Seminar "Securing YOUR Perfect Job!" – Room D

12.30pm – 1.30 pm
SCIEX Lunch Seminar "Mapping Protein Modifications Using Novel MS Strategies" – Council Chamber

2pm – 3.30pm
Conference sessions in Lower Hall Syndicate Room D and Ferrier Hall

3.30 – 4pm
Coffee Break, Exhibition and Posters – Marble Hall and Assembly Room

4pm-5.30pm
Conference sessions in Lower Hall , Syndicate Room D and Ferrier Hall

5.30pm
Mixer and Exhibition – Join the exhibitors for beer, wine and soft drinks. Assembly Room and Marble Hall.

Sponsored By Leco

7.30pm
Beer Degustation evening.
Please bring your tickets and vouchers with you. This is being held at the Jam jar which is located on the first floor of the Yard Bar and Kitchen. 42 - 43 St Mary's Street, Cardiff, CF10 1AD

THURSDAY 25TH JANUARY

Registration open from 8am

8am – 8.45am
Breakfast workshop: Professional development and careers [Dayna Mason (RSC CPD & outreach in Cardiff) & Deirdre Cabooter – Room A

8.55am-9am
HTC15 Announcements – Lower Hall

9am-9.50am
Plenary: Eric Little (Osthus) Transforming Big Data Into Big Analysis: The Power of Finding Use-Value In Your Data – Lower Hall

9.50 – 10.30am
Coffee Break, Exhibition and Posters – Marble Hall and Assembly Room

10.30 – 12pm
Conference sessions in Lower Hall Syndicate Room D and Ferrier Hall

12pm – 2pm
Lunch, Vendor Seminars, Exhibitions and Posters - Marble Hall and Assembly Room

12.30pm – 1.30pm
Ellutia Lunch Seminar "MAX a new concept in GC-FTIR" Includes Free Cakes and Scones for attendees Room A

12.30pm – 1.30pm
Shimadzu Lunch Seminar "Smart & Green Hyphenated Solutions" – Room B

2pm – 3.30pm
Conference sessions in Lower Hall Syndicate Room D and Ferrier Hall

3.30 – 4pm
Coffee Break, Exhibition and Posters – Marble Hall and Assembly Room

4pm-5.30pm
Conference sessions in Lower Hall Syndicate Room D and Ferrier Hall

7.15pm
Gala Dinner at Cardiff Museum
Please bring your dinner ticket with you and note that we are using the side entrance as directed on the tickets. Cardiff Museum Cathays Park, Cardiff, CF10 3NP.

FRIDAY 26TH JANUARY

Registration open from 8.30am

8.55am-9am
HTC15 Announcements – Lower Hall

9am-9.50am
Plenary: Tuulia Hyotylainen - Hyphenated Techniques for comprehensive analysis of all metabolites in biological systems to describe metabolic changes caused by disease, environmental, nutritional, or genetic factors

9.50 – 10.30am
Coffee Break, Exhibition and Posters – Marble Hall and Assembly Room

10.30 – 12pm
Conference sessions in Lower Hall Syndicate Room D and Ferrier Hall

12pm – 12.40pm
Lunch, Exhibitions and Posters - Marble Hall and Assembly Room

1pm – Exhibition Close

12.40pm – 2.10pm
Conference sessions in Lower Hall Syndicate Room D and Ferrier Hall

2.10pm – 2.30pm
Flash Poster Presentations - Lower Hall

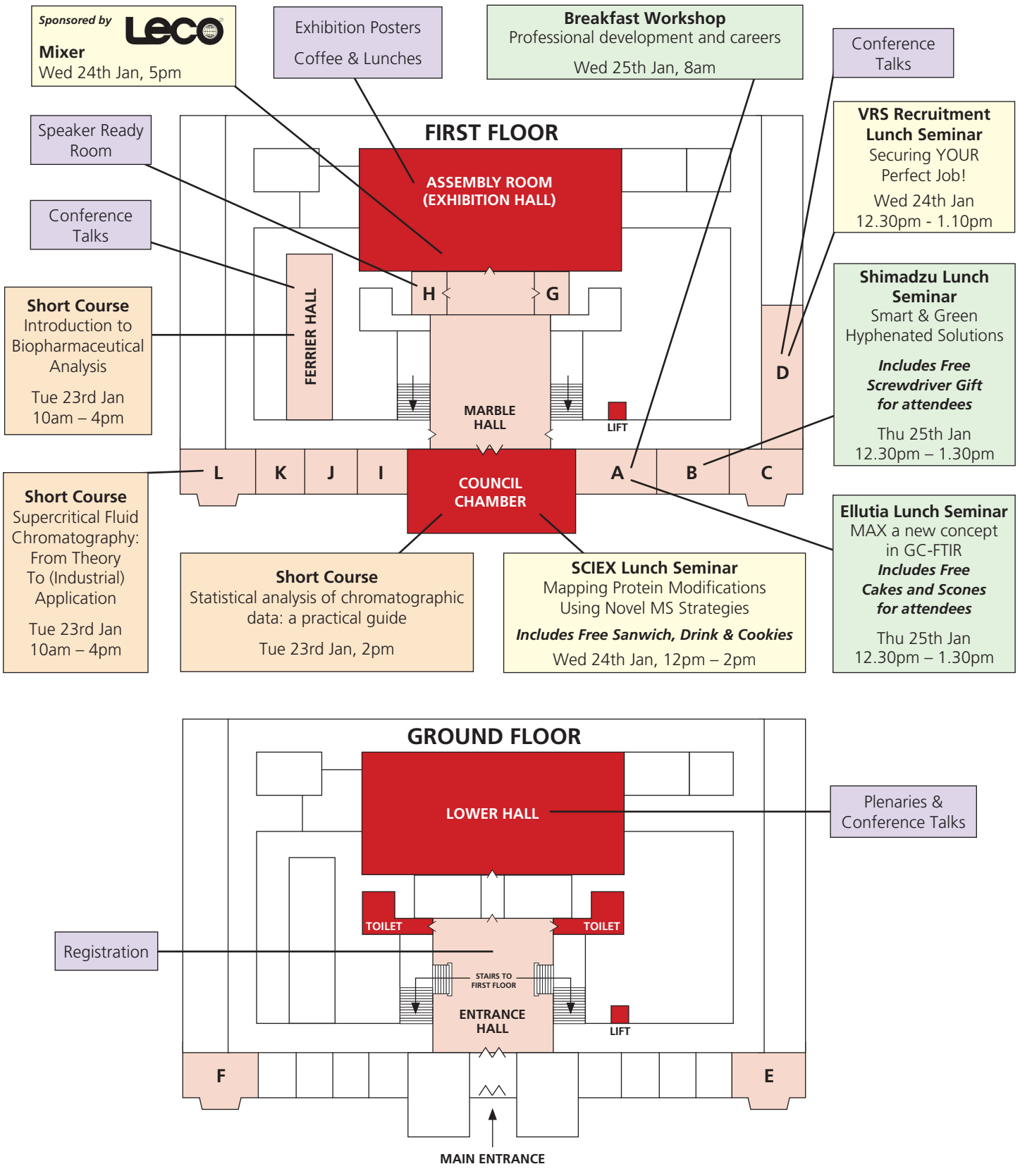
2.30pm – 3.20pm
Plenary: Of mice, sex and mass spectrometry - Rob Beynon – Lower Hall

3.20pm – 4.10pm
Plenary: Multi-dimensional liquid chromatography of complex mixtures Plenary - Peter Schoenmakers Lower Hall

4.10pm – 4.30pm
Awards - Lower Hall

4.30pm
Farewell - Lower Hall

Cardiff City Hall Floorplan



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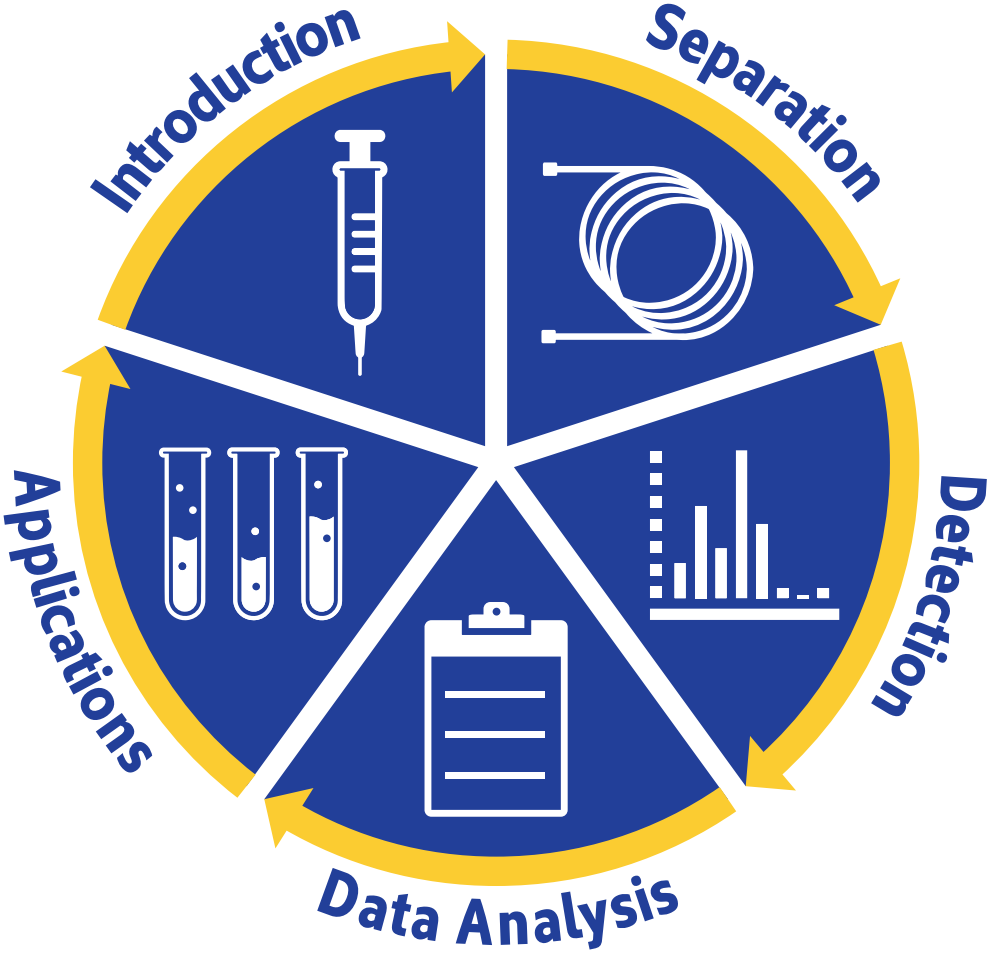

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Floorplan & Exhibitors

Agilent Technologies	19	ESSLAB	28	JMP, SAS Institute	9	ROMIL Ltd	12
Anthias Consulting & DAS	22	EST Analytical (Represented)	4	JSB UK and Ireland Ltd	18	Sartorius (Represented)	28
ATG Scientific	25	GL Sciences	27	KRSS Ltd	17	SCIEX	8
Chiron (Represented)	28	Hamilton (Represented)	28	LCGC	10	Sepsolve Analytical Ltd	4
Chromatography Direct (Represented)	17	Hichrom	31	Leco Instruments	30	Shimadzu Europa GmbH	3
Chromatography Today	29	IMSPEX -GAS (Represented)	20	Markes International	6	Spex (Represented)	28
Crawford Scientific	16	IMSPEX Diagnostics	20	MS Noise (Represented)	25	Thermo Fisher Scientific	26
CTC Analytics (Represented)	4	Inorganic Ventures (Represented)	28	Postnova Analytics UK	11	VUV Analytics (Represented)	4
Diagenode (Represented)	25	IonBench (Represented)	25	Reichert (Represented)	25	Waters European Headquarters	1
Ellutia	7	JEOL EUROPE	21	Restek Corporation	15	YMC Europe (Represented)	16

Posters are displayed in clockwise numbering

Wednesday 24th January 2018

Conference Timetable

08:45 - 09:00	**HTC-15 OPENING CEREMONY (LOWER HALL)**		
09:00 - 09:50	**Plenary: Why Do We Still Use Silica KNOX Award Lecture by Prof Peter Myers, University of Liverpool (LOWER HALL)**		
09:50 - 10:30	Coffee Break & Exhibition (ASSEMBLY HALL)		
TIME	**ASSEMBLY HALL**	**FERRIER HALL**	**ROOM D**
	Session 1: Exploiting Separation Science (Chair: Tom Lynch)	**Session 2: (R)evolutions in Biopharmaceutical Analysis (KVCV) (Chair: Deirdre Cabooter)**	**Session 3: Fundamentals in Separation Science (Chair: Paul Ferguson)**
10:30 - 11:00	Keynote: Separation science and its use for generating data, information and understanding Presenter: Hans Gerd Janssen, Unileve Research and University of Amsterdam	Keynote: Recent advances in the analysis of protein biopharmaceuticals Presenter: Koen Sandra, Research Institute for Chromatography	Tutorial: A tutorial on the fundamentals of hydrophilic interaction chromatography (HILIC) for pharmaceutical analysis Presenter: James Heaton, Pfizer Ltd
11:00 - 11:20	Presentation: Novel ways to introduce the traditional salt based chromatography technique of Ion Exchange Chromatography of biopharmaceutical proteins into High Resolution Mass Spectrometry Presenter: Florian Füssl, Thermo Fisher Scientific	Presentation: From one to four comprehensive separation dimensions to characterize antibody drug conjugates Presenter: Davy Guillarme, University of Geneva	Presentation: Probing selectivity of mixed-mode reversed-phase / weak-anion-exchange columns for small-molecule separations in liquid chromatography Presenter: José Luís Dores-Sousa, Vrije Universiteit Brussel
11:20 - 11:40	Presentation: Hyphenated microdialysis and chromatography to monitoring protein free drug for pharmacokinetic study in rat Presenter: Tung-Hu Tsai, National Yang-Ming University	Presentation: Advancing the analytical toolbox using shotgun lipidomics for lipid modifying enzymes Presenter: Dr Erwin Kaal, DSM	Presentation: Understanding the possibilities of solvent-assisted post-column refocusing to enhance detection limits in 1-D and 2-D LC Presenter: Vincent Pepermans, Vrije Universiteit Brussel
11:40 - 12:00	Presentation: Expanding the Application of Thermal Desorber Devices towards Dynamic Headspace Gas Chromatography for the Determination of Residual Solvents Presenter: Erwin Adams, Pharmaceutical Analysis - KU Leuven	Presentation: Lipidomics by UHPL-CQTOF-MS/MS with Data-Independent Acquisition and Clinical Applications Presenter: Michael Laemmerhofer, University of Tuebingen	Presentation: Investigating the potential for improved temperature responsive separations in liquid chromatography Presenter: Mathijs Baert, Ghent University
12:00-14:00	Lunch Break, Vendor Seminars, Exhibition & Posters		

VRS Recruitment Lunch Seminar
Securing YOUR Perfect Job!
Wed 24th Jan, 12.30pm – 1.10pm
ROOM D

SCIEX Lunch Seminar
Mapping Protein Modifications Using Novel MS Strategies
Includes Free Sanwich, Drink & Cookies
Wed 24th Jan, 12pm – 2pm
COUNCIL CHAMBER

TIME	ASSEMBLY HALL	FERRIER HALL	ROOM D
	Session 4: Fundamentals in Separation Science (KVCV) (Chair: Sebastiaan Eeltink)	**Session 5: Advances in Clinical Analysis (Chair: Dave Perrett)**	**Session 6: Interfacing and Ionisation (Chair: Ruth Godfrey)**
14:00 - 14:30	Keynote:Recent Progress in the Development of Perfectly Ordered Separation Media Presenter: Gert Desmet, Vrije Universiteit Brussel	Keynote: UPLC-MS for Metabolic Phenotyping: Advantages, Assays and Applications Presenter: Elizabeth Want, Imperial College London	Tutorial: UHPSFC-MS of a Range of Steroidal Compounds Presenter: Julie Herniman, University of Southampton
14:30 - 14:50	Presentation: Considerations for the use of ultra-high pressures in liquid chromatography for 2.1mm inner diameter columns Presenter: Ken Broeckhoven, Vrije Universiteit Brussel	Presentation: Opportunities for ultra-rapid LC-MS/MS in high-throughput bioanalysis Presenter: Lewis Couchman, Analytical Services International (ASI) Ltd	Presentation: Characterization of complex polyether polyols using comprehensive two-dimensional liquid chromatography hyphenated with high resolution mass spectrometry (LCxLC-HRMS) Presenter: Gino Groeneveld, University of Amsterdam
14:50 - 15:10	Presentation: Matching 1st and 2nd dimension chemistries in the 2D-LC. Active Solvent Modulation Presenter: Konstantin Shoykhet, Agilent Technologies	Presentation: Apolipoproteins, non-polar lipids, polar lipids and lipoprotein particle numbers. Can we measure them all on large number of samples? Presenter: Zsuzsanna Kuklenyik, Centers for Disease Control and Prevention	Presentation: The comparison of Unispray and Electrospray for the ionization of neuropeptides Presenter: Yannick Van Wanseele, Vrije Universiteit Brussel
15:10 - 15:30	Presentation: Kinetics and mass transfer phenomena in modern chiral stationary phases Presenter: Alberto Cavazzini, University of Ferrara	Presentation: Detection and differentiation of botulinum neurotoxins for the diagnosis and prevention of botulism Presenter: John Barr, Centers for Disease Control and Prevention (CDC)	Presentation: Sequential Three-Dimensional Gas Chromatography with Accurate Mass Spectrometry: A Novel Tool for High-Resolution Characterization of Multicomponent Samples Presenter: Dandan Yan, Unversity of Tasmania
15:30 - 16:00	Coffee Break, Exhibition & Posters (ASSEMBLY HALL)		
	Session 7: High throughput versus High Efficiency Separations (CS) (Chair: Paul Ferguson)	Session 8: Exploiting Separation Science and Mass Spectrometry (Chair: Lewis Couchman)	Session 9: Microfluidics & Flow Process Technology (Chair: Sebastiaan Eeltink)
16:00 - 16:30	Keynote: High temperature chromatography: the winning solution allowing both throughput and efficiency? Presenter: Frederic Lynen, University of Ghent	Keynote: Supercritical Fluid Chromatography - Mass Spectrometry: Robust, Reliable and Required Presenter: John Langley, University of Southampton	Tutorial: Flow chemistry: A synthetic chemist's perspective Presenter: Anna Slater, University of Liverpool
16:30 - 16:50	Presentation: Strategies to Optimize Throughput in 2D-LC Presenter: Monika Dittmann, Agilent Technologies	Presentation: Possibilities of modern size-exclusion chromatography for therapeutic proteins; feasibility assessment for future mass spectrometry hyphenation Presenter: Szabolcs Fekete, University of Geneva	Presentation: Chemically Sensitive Online Detectors for SEC – current advances for SEC-MR-NMR and SEC-QCL-IR Presenter: Johannes Höpfner, Karlsruhe Institute of Technology (KIT)
16:50 - 17:10	Presentation: Fast, efficient and selective: separation science for modern organic synthetic chemistry Presenter: Tomas Leek, AstraZeneca	Presentation: Separation of isomeric metabolites of carbamazepine by liquid chromatography and high resolution accurate mass Presenter: Joanne Roberts, Glasgow Caledonian University	Presentation: Comprehensive Two-Dimensional Liquid Chromatography Coupled to Triple-Detection for Characterization of Branched Polymers Presenter: Nico Apel, Fraunhofer LBF
17:10 - 17:30	Presentation: Generic Reverse Phase Chromatography... Speed, performance... or both? Presenter: Bob Boughtflower, GSK	Presentation: Quantitative drug metabolite profiling without authentic standards or radiolabels using LC-ICP-MS Presenter: Filip Cuyckens, Janssen R&D	Presentation: Chromatographic strategies combining RPLC, mixed-mode HPLC and SFC coupled to MS for impurity profiling of drugs candidates Presenter: Elise Lemasson, Institut de Chimie Organique et Analytique(ICOA)
17:30-18:30	**MIXER & EXHIBITION (ASSEMBLY HALL)** *Sponsored by* LECO		
19:30	Beer Degustation Social at The Yard		

Thursday 25th January 2018

Conference Timetable

Time	ASSEMBLY HALL	FERRIER HALL	ROOM D
08:00 - 08:45	**Breakfast workshop: Professional development and careers [Dayna Mason (RSC CPD & outreach in Cardiff) & Deirdre Cabooter] (ROOM A)**		
08:55 - 09:00	HTC-15 Announcements (LOWER HALL)		
09:00 - 09:50	Plenary: Eric Little (Osthus) Transforming Big Data Into Big Analysis: The Power of Finding Use-Value In Your Data (LOWER HALL)		
09:50 - 10:30	Coffee Break & Exhibition (ASSEMBLY HALL)		
	Session 10: SSG session (Chair: John Langley)	**Session 11: Big Data - What do we do with it? (Chair: Arun Sen)**	**Session 12: Separations by shape: Instrumentation (Chair: Alison Ashcroft)**
10:30 - 11:00	Keynote: Advancing hydrophobic interaction chromatography methods to characterize biotechnology enzyme mixtures and to profile biotherapeutics Presenter: Sebastiaan Eeltink, Vrije Universiteit Brussel	Keynote: Clinical Lipidomic Quantitation Based on Mass Spectrometry: Case Study of Pancreatic Cancer Presenter: Michal Holcapek, University of Pardubice	Tutorial: Separation and characterisation by ion mobility-mass spectrometry Presenter: Antonio Calabrese, University of Leeds
11:00 - 11:20	Presentation: Methodologies to determine b-term coefficients revisited Presenter: Deirdre Cabooter, University in Leuven	Presentation: Replicates of Complex Mixtures in Ultra-High Resolution Mass Spectrometry Could Help Pave The Way to Big Data Presenter: Remy Gavard, University of Warwick	Presentation: FAIMS and fortune Presenter: Christianne Wicking, BP
11:20 - 11:40	Presentation: UHPLC quantitation and identity confirmation in drug development with a multi-detector approach Presenter: Frank Steiner, Thermo Fisher Scientific	Presentation: Big Data - When Less is More Presenter: Benjamin Woolford-Lim, GlaxoSmithKline	Presentation: Topology discrimination of saponin ions by Hyphenated Mass Spectrometry techniques and computational chemistry Presenter: Corentin Decroo, Umons
11:40 - 12:00	Presentation: LCGC innovation award 2018	Presentation: Looking Inside the Black Box of Machine Learning Methods: Applications in Analytical Chemistry Presenter: Phil Kay, JMP, SAS Institute	Presentation: Better Living Through (Flavor) Chemistry: Vacuum Ultraviolet Spectroscopy as a New Tool for GC Analysis of Terpenes in Flavors and Fragrances Presenter: Alex Hodgson, VUV Analytics
12:00- 14:00	Lunch Break, Vendor Seminars, Exhibition & Posters		

Ellutia Lunch Seminar
MAX a new concept in GC-FTIR
Includes Free Cakes and Scones for attendees
Thu 25th Jan, 12.30pm – 1.30pm
ROOM A

Shimadzu Lunch Seminar
Smart & Green Hyphenated Solutions
Includes Free Screwdriver Gift for attendees
Thu 25th Jan, 12.30pm – 1.30pm
ROOM B

Time	ASSEMBLY HALL	FERRIER HALL	ROOM D
	Session 13: Green Separations (Chair: Paul Ferguson)	**Session 14: Big Data Chemometrics and Method development (In-Silico) (KVCV) (Chair: Ken Broeckhoven)**	**Session 15: Approaches to maximising analytical data (Chair: Scott Fletcher)**
14:00 - 14:30	Keynote: 50 shades of Green SFC Presenter: Caroline West, University of Orleans	Keynote: A paradigm shift for (big) data analysis in chromatography: on the use of Bayesian statistics Presenter: Gabriel Vivo Truyols, Tecnometrix	Tutorial by Chris Hopley
14:30 - 14:50	Presentation: Can CE-MS improve the detection of peptides and intact proteins and in biological samples? Presenter: Stephen Lock, Sciex	Presentation: Chromatographic fingerprints: chemometrics and application in method development Presenter: Yvan Vanderheyden, Vrije Universiteit Brussel	Presentation: Data to decision: efficient processing of complex petroleomics data Presenter: Samuel Ellick, University of Bristol
14:50 - 15:10	Presentation: UHPSFC-MS of a Range of Steroidal Compounds Presenter: Julie Herniman, University of Southampton	Presentation: Use of different computer-aided method development software in late stages across global sites in pharmaceutical industry Presenter: Kai Chen, Janssen Pharmaceutical Companies of Johnson & Johnson	Presentation: Characterization of Small Heterogeneities in Polymers by Analysis of UPLC/ESI-MS Reconstructed Ion Chromatograms Presenter: Ruben Epping, Bundesanstalt für Materialforschung und -prüfung (BAM)
15:10 - 15:30	Presentation: Online extraction and determination of carotenoids from food sample by means of supercritical fluid extraction-supercritical fluid chromatography-mass spectrometry Presenter: Mariosimone Zoccali, University of Messina	Presentation: Structure driven prediction of retention :Improvement of accuracy Presenter: Roman Szucs, Pfizer	Presentation: Quantitative proteomics for molecular diagnostics of public health Presenter: Jack Rice, University of Bath
15:30 - 16:00	Coffee Break & Exhibition		
	Session 16: Ion Mobility - Mass Spectrometry (BMSS) (Chair: Alison Ashcroft)	**Session 17: Screening Enviroment Pollutants, what can the data tell us? (Chair: Graham Mills)**	**Session 18: Separations by shape: Further applications of chromatography (Chair: Frank David)**
16:00 - 16:30	Keynote: Ion mobility mass spectrometry - leveraging rich data on the gas-phase ion to separate and assign Presenter: Cris Lapthorn, GSK	Keynote: Urban water profiling to inform the state of the environment and public health Presenter: Barbara Kasprzyk-Hordern, University of Bath	Tutorial: Analysis of food products using advanced analytical techniques Presenter: Paola Dugo, University of Messina
16:30 - 16:50	Presentation: Understanding phosphorylation-mediated effects on NF- B interactions using IM-MS Presenter: Claire Eyers, University of Liverpool	Presentation: Suspect screening of aquatic environmental matrices using high resolution analysis and in silico tools for broad scope tentative contaminant identification Presenter: Leon Barron, King's College London	Presentation: On-line coupling of RPLC and chiral SFC for the analysis of pharmaceutical compounds Presenter: Marion Iguiniz, Université de Lyon
16:50 - 17:10	Presentation: An Advanced Cyclic Ion Mobility - Mass Spectrometry System Presenter: Kevin Giles, Waters Corporation	Presentation: Screening of environmental passive sampling extracts using LC Q-TOF-MS in data-independent acquisition mode Presenter: Anthony Gravell, Natural Resources Wales	Presentation: Enhanced resolution of stereoisomers through Stationary phase optimized selectivity liquid and supercritical chromatography (SOS-LC and SOS-SFC) Presenter: Ravindra Hegade, Ghent University
17:10 - 17:30	Presentation: FAIMS mass spectrometry for the analysis of peptides and proteins Presenter: Helen Cooper, University of Birmingham	Presentation: Combining high-capacity sorptive extraction with Thermal desorption pre-concentration for analysis of (S)VOCs in environmental samples Presenter: Lara Kelly, Markes International Ltd	Presentation: LC-MS/MS chiral ananlysis of chloramphencicol in the environment Presenter: Felicity Elder, University of Bath
19:15	Gala Dinner Event (CARDIFF MUSEUM)		

Friday 26th January 2018

Conference Timetable

08:55 - 09:00	HTC-15 Announcements (LOWER HALL)		
09:00 - 09:50	Plenary: Tuulia Hyotylainen - Hyphenated Techniques for comprehensive analysis of all metabolites in biological systems to describe metabolic changes caused by disease, environmental, nutritional, or genetic factors (LOWER HALL)		
09:50 - 10:30	Coffee Break & Exhibition (ASSEMBLY HALL)		
TIME	**ASSEMBLY HALL**	**FERRIER HALL**	**ROOM D**
	Session 19: Automating Complex Sample Workflows (Chair: Bob Boughtflower)	**Session 20: Comprehensive Chromatography - The State of the art (Chair: Hans Gerd Janssen)**	**Session 21: Energy & the Environment (Chair: Sam Whitmarsh)**
10:30 - 11:00	Keynote: Laboratory Automation: The Rise of the Machines Presenter: Scott Summerfield, GSK	Keynote: Flow Modulated Two-dimensional Gas Chromatography Coupled to Tandem Mass Spectrometry for "Comprehensive" characterization of Complex Samples Presenter: Luigi Mondello, University of Messina	Tutorial: Environmental health and well-being: diagnostics with hyphenated gas chromatography Presenter: John Dean, Northumbria University
11:00 - 11:20	Presentation: Automated sample preparation: the missing hyphen to hypernation Presenter: Camilla Liscio, Anatune	Presentation: Applicability of retention modelling in hydrophilic-interaction liquid chromatography for algorithmic optimization programs with gradient-scanning techniques Presenter: Bob Pirok, University of Amsterdam	Presentation: Fingerprint characterization of bio-oil from the pyrolysis of African palm and its esterification product Presenter: Diana Palacio, University of Warwick
11:20 - 11:40	Presentation: Solid Phase Micro-Extraction - Breaking Free from Total Concentration Analysis Presenter: Sheelan Ahmad, GlaxoSmithKline	Presentation: All Ion Differential Analysis in Product Control Applications using GC/MS and Comprehensive GCxGC/MS Presenter: Marco Ruijken, MsMetrix	Presentation: Mass Spectrometric Investigation of Compounds of Interest to the Chemical Investigation Programme (CIP) within Environmental Matrices: Homogenate Analysis Presenter: Rachel Townsend, Swansea University
11:40 - 12:00	Presentation: Analytical developments in sample preparation to reliably measure the emission of volatiles from materials in a high-throughput fashion Presenter: An Adams, Dow	Presentation: Detection of bacteriologically produced hydrogen sulphide using SHS-MCC-GC-IMS Presenter: John Dean, Northumbria University	Presentation: Petroleomic Depth Profiling of Staten Island Soil by GC and FT-ICR MS Presenter: Mary Thomas, University of Warwick
12:00 - 12:40	Lunch Break & Exhibition		
	Session 22: Analysis of Complex Energy Products (Chair: Tom Lynch)	**Session 23: Advanced Analysis of Food and Beverages (Chair: Lewis Jones)**	**Session 24: Life science & pharma (Chair: Bob Boughtflower)**
12:40 - 13:10	Keynote: Thermal Analysis in hyphenation with mass spectrometry as a versatile tool for the analysis of complex and high boiling petroleum products Presenter: Thomas Gröger, helmholtz Zentrum München GmbH	Keynote: Exploiting comprehensive two-dimensional liquid chromatography in food analysis Presenter: Paola Dugo, University of Messina	Tutorial: LC-MS small molecule quantitation: a short tutorial of best practice Ruth Godfrey, Swansea University
13:10 - 13:30	Presentation: Direct infusion MS - who needs hyphenation anyway? Presenter: Sam Whitmarsh, BP	Presentation: Can Gas Chromatography - Olfactometry Determine the Importance of Volatile Organic Chemical to Food and Beverage Odour? Presenter: Lewis Jones, Sensient Flavours	Presentation: From GC-MS to LC-MS/MS: Further Advances in Adrenal Cancer Diagnosis Presenter: Angela Taylor, University of Birmingham

TIME	ASSEMBLY HALL	FERRIER HALL	ROOM D
13:30 - 13:50	Presentation: Fast and efficient group-type analysis of hydrocarbons by GCxGC Presenter: Laura McGregor, SepSolve Analytical	Presentation: Rapid evaporative ionization mass spectrometry for high throughput screening in food analysis: the case of boar taint Presenter: Sara Stead, Waters Corporation	Presentation: Green Bioanalytical Analysis of Voriconazole and Tadalafil by HPLC Presenter: Aysegul Dogan, Hacettepe University
13:50 - 14:10	Presentation: Recent Advances in the Analysis of Petroleum-based Fuels using Gas Chromatography-Vacuum Ultraviolet Spectroscopy Presenter: James Diekmann, VUV Analytics	Presentation: Monitoring the effect of post-harvest storage on fruit quality by TD-GC×GC-TOF MS Presenter: Natasha/D. Spadafora, University of Calabria/Markes International	Presentation: Improving Untargeted Metabolomics with Ion Chromatography-Mass Spectrometry Presenter: John Walsby-Tickle, University of Oxford
14:10 - 14:30	Flash Poster Presentations (LOWER HALL)		
14:30 - 15:20	Of mice, sex and mass spectrometry Plenary by Rob Beynon (Chair: John Langley) (LOWER HALL)		
15:20 - 16:10	Multi-dimensional liquid chromatography of complex mixtures Plenary by Peter Schoenmakers (Chair: Frederic Lynen) (LOWER HALL)		
16:10 - 16:30	Awards (LOWER HALL)		
16:30	Farewell Event (LOWER HALL)		

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Posters

No	Poster Title	Presenter	Session	Country
1	An LC-MS/MS Method to Monitor Tamoxifen and its Metabolites in Rat for Pharmacokinetic Study	Yung-Yi Cheng, Institute of Traditional Medicine, National Yang-Ming University	(R)evolutions in Biopharmaceutical Analysis (KVCV)	Taiwan, Republic of China
2	Comparative Analysis in GCxGC/MS: Detection and Identification of Co-Eluting Unknowns	Marco Ruijken, MsMetrix	Advanced Analysis of Food and Beverages	Netherlands
3	Development and validation of screening method for pesticides residues analysis in vegetables and fruits	Aarif El-Mubarak, King Saud University	Advanced Analysis of Food and Beverages	Saudi Arabia
4	Quantitation of Dodecanoic Acid in Coconut Oil	John Moncur, SpectralWorks Limited	Advances in Clinical Analysis	UK
5	The analysis of steroid seizures from UK customs-Implications for Anti-Doping	Alan Brailsford, Kings College London	Advances in Clinical Analysis	UK
6	Application of gas chromatography-mass spectrometry (GC-MS) in the quantitative analysis of organic compounds generated in gasification/pyrolysis coupled Fischer-Tropsch (FT) reactor: syngas clean up and hydrocarbon production.	Geraint Sullivan, Swansea University	Analysis of Complex Energy Products	UK
7	Novel method for determination of acylcarnitines	Benjami Jenkins, University of Cambridge	Analysis of Complex Energy Products	UK
8	Automated combination of Purge & Trap with common GC-MS Injection techniques according to EPA Methods 524.2 and 8260 on the PAL automation platform.	Stefan Cretnik, CTC Analytics	Automating Complex Sample Workflows	Switzerland
9	Looking Inside the Black Box of Machine Learning Methods: Applications in Analytical Chemistry	Phil Kay, JMP, SAS Institute	Big Data Chemometrics and Method development (In-Silico) (KVCV)	UK
10	MsCompare: An Untargeted GC/MS Metabolomics Platform for Quality Control, Precise Deconvolution and Data Analysis	Marco Ruijken, MsMetrix	Big Data Chemometrics and Method Development(In-Silico) (KVCV)	Netherlands
11	Object orientated programming, a core skill for the modern analytical chemist?	Samuel Ellick, University of Bristol	Big Data Chemometrics and Method Development(In-Silico) (KVCV)	UK
12	Towards Multifactorial Method Development via Predictive Elution Window Stretching and Shifting	Gitte Coopmans, VUB	Big Data Chemometrics and Method Development(In-Silico) (KVCV)	Belgium
13	Oil analysis: an ensemble approach	Tom Hancock, BP	Big Data Chemometrics and Method Development(In-Silico) (KVCV)	UK
14	Characterisation of insulin analogues with SEC-MALS and complementary ultracentrifugation	Richard Gillis, University of Nottingham	BioPharma/Sample Prep & Automation	UK
15	Application of 2D-LC for the traceable quantification of human growth hormone in serum	Sophie Inman, LGC	Challenges in Quantitative Analysis	UK
16	GCxGC–HRTOFMS analysis of a complex lipid profile in human sebum	Masahiro Hashimoto, JEOL(Europe)SAS	Comprehensive Chromatography - The State of the Art	France
17	Assessing 2-Dimensional Comprehensive Flow-Modulated Gas Chromatography for fingerprinting archaeological bitumen	Thomas Van de Velde, Ghent University	Comprehensive Chromatography - The State of the Art	Belgium
18	Exploring the possibilities of temperature responsive columns in comprehensive two-dimensional liquid chromatography.	Mathijs Baert, Ghent University	Comprehensive Chromatography - The State of the Art	Belgium
19	An informatics based approach to developing stability indicating methods	Peter Russell, Advanced Chemistry Development, Inc. (ACD/Labs)	Comprehensive Chromatography - The State of the Art	UK

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Posters

20	Combined separation strategy for chiral method development of acidic and non-acidic pharmaceutical compounds in capillary electrochromatography (CEC) separation technique	Dima Albals, Faculty of pharmacy, Department of Pharmaceutical Science, Yarmouk University	Comprehensive Chromatography - The State of the Art	Jordan
21	Simultaneous determination of lovastatin and its metabolite in rat plasma by liquid chromatography tandem mass spectrometry	Wen-Ya Peng, National Yang-Ming University	Comprehensive Chromatography - The State of the Art	Taiwan, Republic of China
22	Finding a needle in a haystack: analysis of GC x GC Data	Alexandra Harvey, Defence Science and Technology Lab	Comprehensive Chromatography - The State of the Art	UK
23	Comprehensive Analysis of Complex Environmental Samples using Comprehensive Two-Dimensional GC with Ultra High Resolution Time-of-Flight Mass Spectrometry (GCxGC-HRMS)	Alan Griffiths, LECO UK	Comprehensive Chromatography - The State of the Art	UK
24	Enhancing detection limits in chromatography using solvent-assisted post-column refocusing	Vincent Pepermans, Vrije Universiteit Brussel (VUB)	Comprehensive Chromatography - The State of the Art	Belgium
25	Molecule, particle, vesicle? - The new challenge for analytical separation technologies in polymer and protein characterisation	Paul Clarke, Postnova Analytics	Comprehensive Chromatography - The State of the Art	UK
26	Microwave-assisted hydrolysis of Alginic Acid	Tiffani Bouanati, UMons	Energy & the Environment	Belgium
27	Thallium bioaccumulation and cytosolic distribution among biomolecules of different molecular masses in the liver of brown trout (Salmo trutta Linnaeus, 1758) from the Croatian river Krka	Nesrete Krasnici, Rudjer Boskovic Institute	Energy & the Environment	Croatia
28	High-throughput GC×GC-FID for routine environmental analyses	Laura McGregor, SepSolve Analytical	Energy & the Environment	UK
29	Chiral Stationary Phase Optimized Selectivity Supercritical Fluid Chromatography (SOS-SFC): a novel approach for optimizing the separation of enantiomers	Ravindra Hegade, Ghent University	Exploiting Separation Science	Belgium
30	Characterization of Mesoporous Silica Used for Drug Delivery by Sorptive Interaction - Multiple Headspace Extraction - Gas Chromatography	Juan Aspromonte, Pharmaceutical Analysis - KU Leuven	Exploiting Separation Science	Belgium
31	The power of selectivity and the strength to choose - Chiral Screening using an SFC / LC Switching System	Gesa Schad, Shimadzu Europa GmbH	Exploiting Separation Science	Germany
32	Identification of Aspergillus spores by preparative IEF and MALDI-TOF MS	Jiri Salplachta, Czech Academy of Sciences, Institute of Analytical Chemistry	Exploiting Separation Science	Czech Republic
33	Stepwise HPLC Extraction and Purification of Endogenous Urinary Metabolites for Their Annotation and Identification in Large Scale Phenotyping Studies	Luke Whiley, MRC-NIHR National Phenome Centre, Imperial College London	Exploiting Separation Science	UK
34	Quantification of the extended list of suspected allergens in fragrance materials by GCxGC-TOFMS	Sebastiano Panto, LECO European Application & Technology Center	Exploiting Separation Science	Germany
35	Underivatized LC-MS/MS determination of ethyl carbamate in wines	João Micael Leça, Faculty of Exact Sciences and Engineering - University of Madeira	Food/Drink	Portugal
36	SPE-GC-MS screening of the volatiles from musts collected at different Madeira winemakers	Andreia Miranda, University of Madeira	Food/Drink	Portugal
37	Development of a new method of headspace analysis on flavored mineral waters	Emilie Descours, ISIPCA	Food/Drink	France
38	Application of tandem UV/Charged aerosol detection in analysis of bioactive substances in food supplements	Jakub Fibigr, Charles University, Faculty of Pharmacy	Food/Drink	Czech Republic

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Posters

39	On-line solid phase extraction coupled to liquid chromatography using column-switching system for mycotoxin analysis in beer	Ivona Lhotská, Charles University, Faculty of Pharmacy in Hradec Králové, Department of Analytical Chemistry	Food/Drink	Czech Republic
40	Polar pesticide analysis by CESI-MS	Stephen Lock, Sciex	Food/Drink	UK
41	Flavour profiling of milk using high-capacity sorptive extraction and TD–GC×GC–TOF MS	Laura McGregor, SepSolve Analytical	Food/Drink	UK
42	Dioxin, PCB, PBDE and PFAA concentrations in Baltic salmon during the 2000's	Jani Koponen, National Institute for Health and Welfare (THL)	Food/Drink	Finland
43	Sample Preparation Options for determination of Volatile compounds in Food	Camilla Liscio, Anatune Ltd	Food/Drink	UK
44	Separation of amino acids and aromatic carboxylic acids on silica monolithic capillary columns	Dana Moravcova, Czech Academy of Sciences, Institute of Analytical Chemistry	Fundamentals in Separation Science & Sample Prep	Czech Republic
45	Selectivity and mixed-mode retention mechanism of small-molecules in liquid chromatography using a reversed-phase / weak-anion-exchange stationary phase	José Luís Dores-Sousa, Department of Chemical Engineering, Vrije Universiteit Brussel	Fundamentals in Separation Science & Sample Prep	Belgium
46	Synthetic polymeric resins in downstream processing for food, fine chemicals and pharmaceuticals	Benjamin Summers, Purolite Ltd.	Fundamentals in Separation Science & Sample Prep	UK
47	SFC-MS compared to LC-MS in the analysis of small molecules	Gesa Schad, Shimadzu Europa GmbH	Fundamentals in Separation Science (KVCV)	Germany
48	Gas Chromatographic Computer Modeling Software for Optimized Method Development and Education	Hansjoerg Majer, Restek Corp	Fundamentals in Separation Science (KVCV)	United States
49	Analysis of Aromatic Substituted Quaternary Ammonium Salts using a Headspace Gas Chromatography Based Methodology	Erwin Adams, Pharmaceutical Analysis - KU Leuven	Hyphenated Techniques for Comprehensive Analysis	Belgium
50	Application of an Atmospheric Micro Hollow Cathode Discharge Set-up in a Novel GC-detector	Kris Wolfs, Pharmaceutical Analysis - KU Leuven	Interfacing and Ionisation	Belgium
51	Primary and secondary structures of peptoids as probed by Ion Mobility Mass Spectrometry	Emilie Halin, UMONS	Ion Mobility - Mass Spectrometry (BMSS)	Belgium
52	From clear liquids to clear solutions!	Alex Ttofi, Bp	Ion Mobility - Mass Spectrometry (BMSS)	UK
53	Understanding the dynamics of ion suppression caused by column bleed	Liz Bevan, Agilent Technologies	Ion Mobility - Mass Spectrometry (BMSS)	UK
54	The Analysis of Extractable and Leachable Components from Common Packaging Material using Ion-Mobility-Mass Spectrometry : Monitoring System Performance	Jane Cooper, Waters Corporation	Ion Mobility - Mass Spectrometry (BMSS)	UK
55	Application of Headspace – Solid-Phase Microextraction Coupled with Gas Chromatography/Mass Spectrometry for the Characterization of Polymeric Materials	Peter Kusch, Bonn-Rhein-Sieg University of Applied Sciences	Microfluidics & flow process technology	Germany
56	Direct Coupling of Solid Phase Microextraction to an Active Capillary Plasma Ionization Source for High-Throughput Trace Analysis in Solid and Liquid Matrices	Stefan Cretnik, ETH Zürich	Other	Switzerland
57	The Analysis of Complex Methylene Diphenyl Diisocyanate (MDI) Oligomers using UHPSFC-APPI+ MS	Dovile Lingaityte, University of Southampton	Other	UK
58	Extending Thermal Stability and Column Lifetime of WAX GC Columns	Laura Provoost, Agilent Technologies	Other	Netherlands
59	Characterisation and biological evaluation of Chilean quinoa saponin	Emmanuel Colson, UMONS	Quantitation, Pharma & Forensics	Belgium
60	Novel method for simultaneous determination of perfluoroalkyl acids, parabens and cotinine from a low volume human serum sample	Jani Koponen, National Institute for Health and Welfare (THL)	Screening Enviroment Pollutants, what can the data tell us?	Finland

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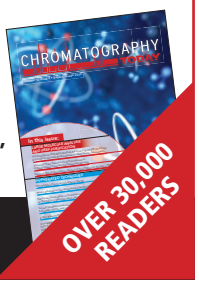

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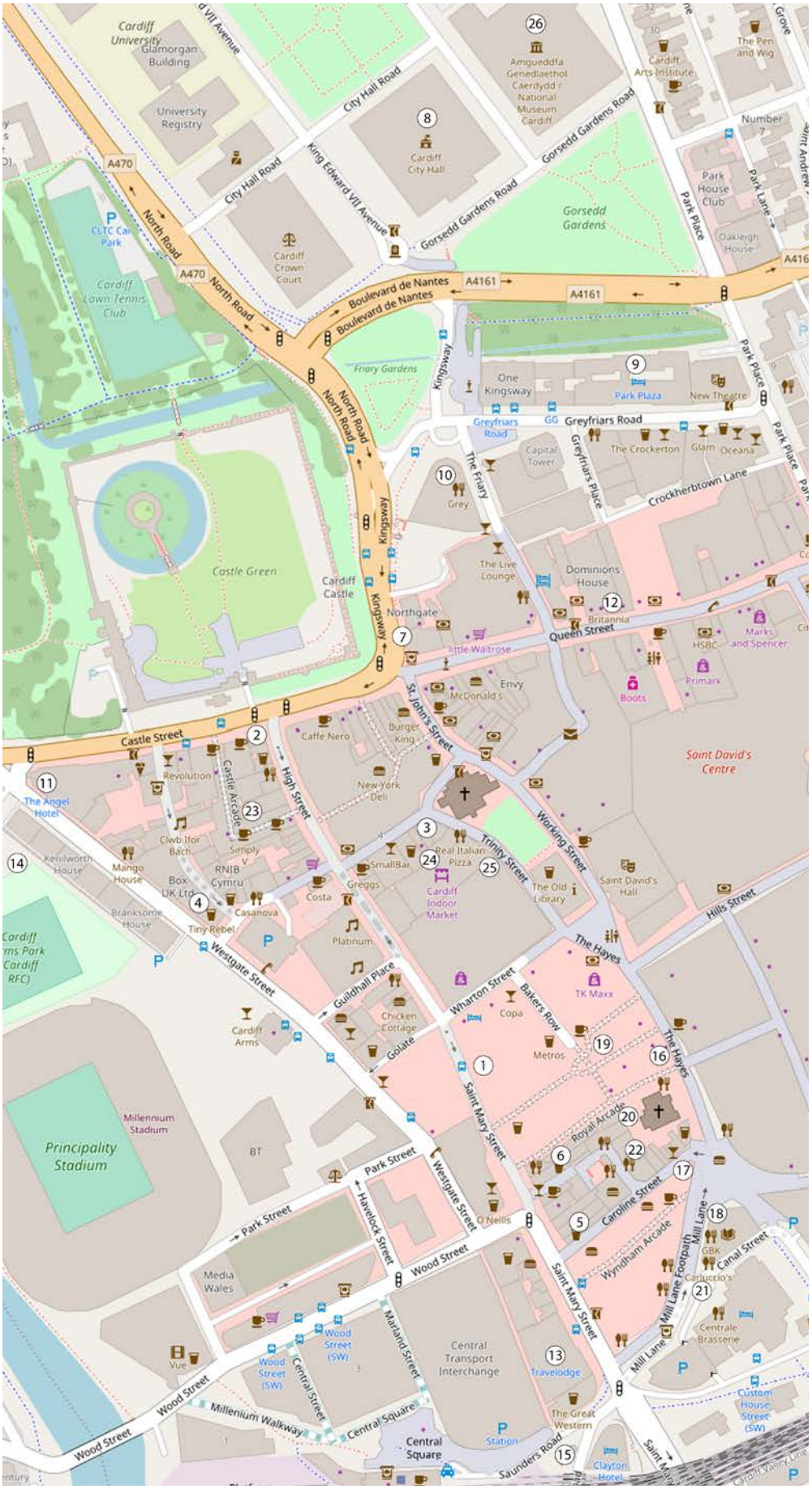
Cardiff City Map

1. The Cottage
2. Goat Major
3. Old Arcade
4. Tiny Rebel Cardiff
5. The Cambrian Tap
6. Yard Bar & Kitchen
7. Hopbunker
8. Cardiff City Hall
9. Park Plaza Hotel Cardiff
10. Hilton Cardiff
11. The Angel Hotel - part of The Cairn Collection
12. Hotel Indigo Cardiff
13. Travelodge Cardiff Central Queen Street
14. Holiday Inn Cardiff City Centre
15. Sleeperz Hotel Cardiff
16. Giovanni's Restaurant
17. Bill's Cardiff Central Restaurant
18. Wagamama Cardiff Library
19. The Plan
20. Spice Quarter
21. Carluccio's
22. Dorothy's Fish Bar
23. The Potted Pig
24. Cafe Citta
25. The Real Italian Pizza Co
26. National Museum Cardiff



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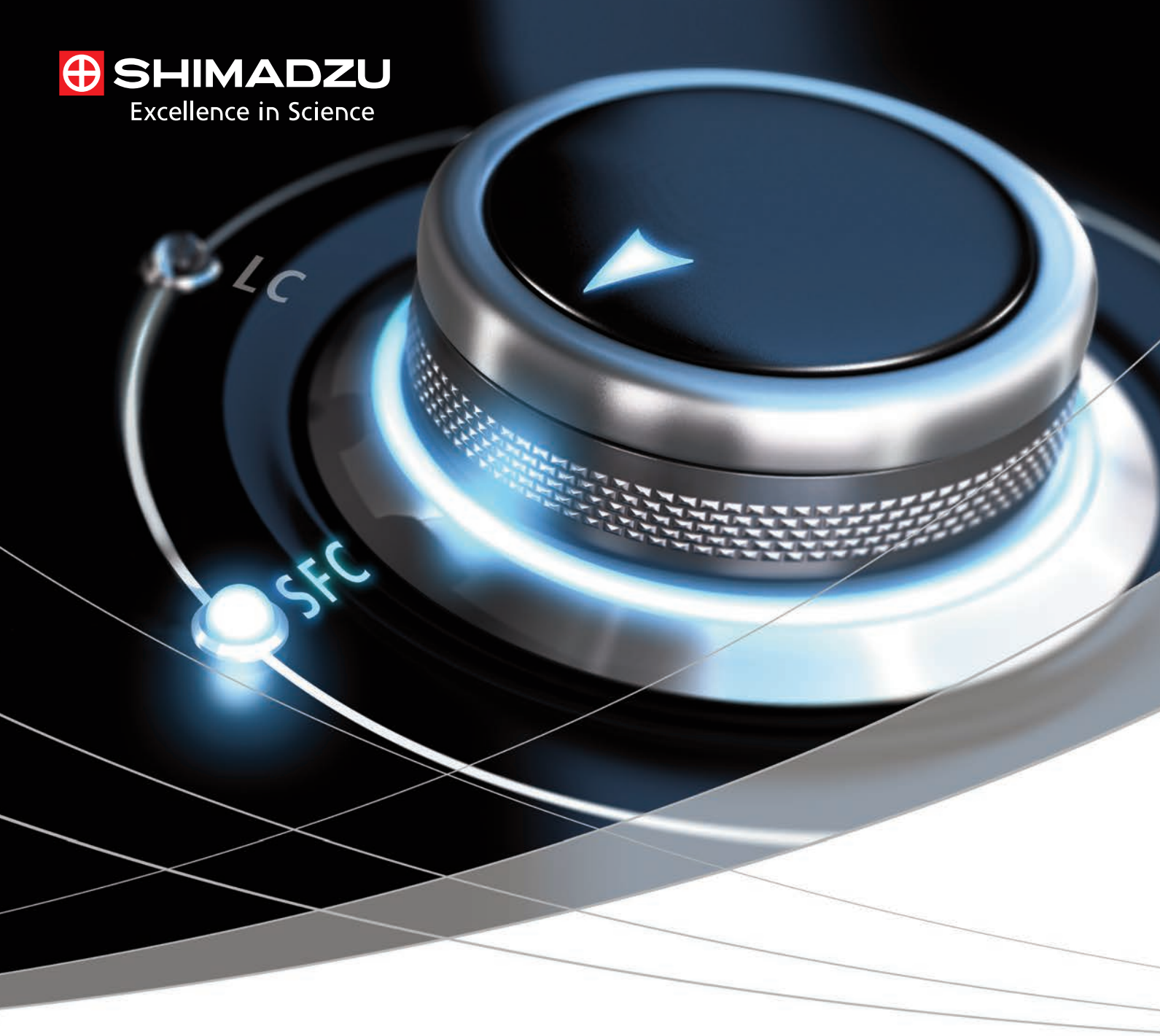

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HTC-15 Abstracts ordered by presenter surname A-Z

Expanding the Application of Thermal Desorber Devices towards Dynamic Headspace Gas Chromatography for the Determination of Residual Solvents (Seminar)

Author: Erwin Adams, Pharmaceutical Analysis - KU Leuven
Country: Belgium
Co-Authors: Adissu Alemayehu Asfaw, Ann Van Schepdael, Kris Wolfs

Session: Quantitation, Pharma & Forensics
Day & Time: Wednesday 24th January 2018, 11:40 - 12:00
Room: Lower Hall

Mesoporous silica (MPSi) and gelatin are frequently used in drug delivery systems. The process usually involves loading of the drug by immersion of the carrier in a drug solution followed by removal of the solvent. Unfortunately, it is practically not possible to remove the solvent completely, which leads to the presence of residual solvents (RS).

Determination of RS is commonly performed using static headspace - gas chromatography (sHS-GC), which is based on the distribution of analytes at equilibrium between the HS and the sample. Although sHS-GC is mainly developed for liquids or solutions of the sample, solids can in principle be processed as well. However, adsorption phenomena like encountered with MPSi make that the equilibrium is no longer purely partition driven. As a result, the relationship between the original concentration of analyte in the sample and the equilibrium concentration in the HS gas phase is highly nonlinear and thus unsuitable for quantitative work. The use of the total or full evaporation approach under HS conditions is also not an option since complete evaporation of the RS from the MPSi can not be guaranteed.

A similar problem arises when RS in drug loaded gelatin samples have to be determined. Indeed, when heated above 50°C in an aqueous environment, gelatin undergoes a physicochemical transformation and loses its solubility in most solvents. So, also here one can not be sure about the complete transfer of the analyte to the vapor phase.

In this work, thermal desorption - gas chromatography - flame ionization detection (TD-GC-FID) is adapted to enable the determination of RS in MPSi and gelatin used for drug delivery. The major modification is related to the sample introduction into the TD tube. Parameters were optimized and validation was performed in terms of sensitivity, linearity, repeatability and recovery. The approach was applied to MPSi loaded with itraconazole using dichloromethane and to gelatin loaded with carbamazepine using DMSO.

To verify the results, alternative methods were developed. For MPSi, this was based on complete dissolution of MPSi in hydrofluoric acid followed by full evaporation HS-GC. For gelatin, the sample was first subjected to enzymatic degradation. Results obtained with the HS-GC methods were found to be similar to those obtained with the TD-GC method, but seen its simplicity and user friendliness, the novel approach is considered to be superior.

Analytical Developments in Sample Preparation to Reliably Measure the Emission of Volatiles from Materials in a High-throughput Fashion (Seminar)

Author: An Adams, Dow
Country: Netherlands
Co-Authors: Francois Huby, Peilin Yang, Jian Zou

Session: Other
Day & Time: Friday 26th January 2018, 11:40 - 12:00
Room: Lower Hall

The growing global concern for Indoor Air Quality results in an increased demand for low VOC (Volatile Organic Compounds) emitting materials when these are applied in building interiors or passenger cars. Regulatory and equipment manufacturers requirements for emission specifications are built on a scattered variety of reference test procedures, often based on large-scale emission chambers (≥ 1 m3). These analytical procedures are not suitable for testing large amounts of samples and for screening the emission potential of newly developed materials. As a result, within Dow Analytical Sciences effort has been devoted to the development of analytical capabilities for the reliable and sensitive measurement of the emission of (semi-) volatile organic compounds from a wide variety of materials in a relatively high-throughput fashion.

Whereas GC-MS/FID is the method of choice for separation, identification and quantification of the volatiles, the sample introduction step is critical for a reliable assessment of the volatiles emitted from materials under realistic conditions. For this purpose, the use of custom-built chambers of varying size, commercial microchambers or gas bags has been evaluated. The influence of different test parameters on the precision and accuracy of the measurements has been investigated.

To improve the sample throughput, different direct-injection soft-ionization mass spectrometric solutions for real-time quantitative analysis of low level volatiles have been evaluated as well, in particular SIFT-MS (Selected-Ion Flow Tube Mass Spectrometry). The direct injection feature avoids trapping of volatile analytes on adsorbent media, and thus decreases the chances for compound modification or discrimination. In addition, the analysis time can be significantly reduced. These type of techniques can be very valuable for the analysis of reactive compounds, such as formaldehyde, which otherwise requires an additional derivatization step.

Solid Phase Micro-Extraction - Breaking Free from Total Concentration Analysis (Seminar)

Author: Sheelan Ahmad, GlaxoSmithKline
Country: United Kingdom
Co-Authors:

Session: Other
Day & Time: Friday 26th January 2018, 11:20 - 11:40
Room: Lower Hall

Drug molecules are either bound to plasma proteins (plasma protein binding) or are free (unbound) and can diffuse through biological membranes or bind to receptors. It is generally believed that the free concentration is the fraction which is responsible for causing a pharmacological response, only free drug molecules can interact with the therapeutic target and are available for distribution, elimination and other PK and PD actions. Hence, accurate determination of this parameter is essential for therapeutic drug monitoring, specifically for drugs with a narrow therapeutic window.

In this study the utility of BioSPME fibres for measuring free drug concentration from biological matrices was demonstrated both in vitro and in vivo. An in vitro experiment was conducted to compare the amount of drug extracted from protein free matrix (PBS) with the amount of drug determined from plasma (high protein content matrix). Three analytes were studied (metoprolol, propranolol and diclofenac) at three different concentrations (10, 100 and 500 ng/mL). The percentage of bound drug concentration extracted was then calculated and compared with values obtained using a conventional technique, rapid equilibrium dialysis (RED) device, which is routinely utilised for establishing plasma drug protein binding. An in vivo study was also performed to determine the free concentration of metoprolol in conscious rats.

This presentation will give an overview of both studies and the potential of using SPME as a tool to measure free drug concentrations.

Comprehensive Two-Dimensional Liquid Chromatography Coupled to Triple-Detection for Characterization of Branched Polymers (Seminar)

Author: Nico Apel, Fraunhofer LBF
Country: Germany
Co-Authors: Robert Bruell, Tibor Macko, Stephan Moyses, Elena Uliyanchenko, Christian Wold

Session: Comprehensive Chromatography - The State of the Art
Day & Time: Wednesday 24th January 2018, 16:50 - 17:10
Room: Room D

Polymer processing properties and their performance in final applications strongly depend on their composition. Branching, in particular, affects the rheological properties of the polymer melt and, thus, influences the processing conditions, enabling new forming processes and, consequently, new applications.

However, in addition to branching, branched polymers are distributed with regard to molecu-lar weight, end-group moiety and sometimes chemical composition. Because of this high structural complexity, their characterization is fundamentally challenging. There is no univer-sal analytical method or technique that is able to provide detailed information on branching distribution alongside with other molecular characteristics. Size-exclusion chromatography (SEC) in combination with triple-detection (TD) is the most common technique for characteri-zation of branched macromolecules. In SEC strong eluent is applied and the separation is based on hydrodynamic volume and driven by entropy effects. Therefore, some coelution of linear and branched structures with the same hydrodynamic volume can occur throughout the chromatogram. This leads to inaccurate branching information determined by TD, since only average degrees of branching are measured at each elution slice.

Depending on the experimental conditions (stationary phase, eluent strength), polymers may be separated by other chromatography modes according to different molecular metrics. Ap-plying weak eluents separation occurs in adsorption mode (liquid adsorption chromatog-raphy, LAC) and is mainly determined by the chemical composition of the polymer. At the transition between LAC and SEC the macromolecules elute independently of their molar mass for a given repeating unit (so-called liquid chromatography at critical conditions, LCCC). LCCC can be used to separate macromolecules according to end-groups.

In the present case, a new chromatographic method, namely solvent gradient at near-critical conditions, was developed applying a mobile phase gradient in a very narrow range around the critical point and allowing for a separation according to end-group moiety of the analyzed polymer. As branched polymers often vary with respect to their number of end-groups a sep-aration of linear and branched polymer structures could be attained (upper chart in Fig. 1). The separation was further augmented by hyphenating the developed method with SEC in a two-dimensional (2D) setup allowing detailed characterization of branched chains simultane-ously with molecular weight. Finally, the 2D-LC method was coupled with TD, comprising a concentration, a light scattering and a viscosity detector. That allowed for analyzing the degree of branching of individual branched structures as a function of their absolute molar masses without the coelution problem encountered in one-dimensional analysis. For the first time a 2D contour plot of the degree of branching could be presented highlighting the high potential of 2D-LC hyphenated with TD.

Investigating the Potential for Improved Temperature Responsive Separations in Liquid Chromatography (Seminar)

Author: Mathijs Baert, Ghent University
Country: Belgium
Co-Authors: Filip Du Prez, Frederic Lynen, Steven Martens

Session: Green Separations
Day & Time: Wednesday 24th January 2018, 11:40 - 12:00
Room: Room D

The use of polymer derived stationary phases in liquid chromatography, either as a replacement for or as a hybrid silica based stationary phase, has been expanding in last decades. An interesting discovery in this field has been the development of temperature responsive stationary phases, wherein a temperature responsive polymer is used to achieve separation.1 This type of polymer is an "intelligent material", as it is able to respond to a small variation in its surroundings with a sharp change in its physical properties. In the case of temperature responsive polymers, they possess a unique characteristic that allows them to change their water solubility based on changes in the ambient temperature. More specifically they show a decrease in polarity with increasing temperature. Implementing this polymer into a bonded-phase for liquid chromatography, allows for the control of the column polarity through control of the column temperature. This introduces the possibility to perform reversed phase like liquid chromatography in pure water, whereby the polarity is controlled through temperature, therefore eliminating the need for any organic modifiers.

Although successful implementation of this technique has already been demonstrated for several applications, the technique is still in its developmental stage. As a result, this strategy has not yet reached its full potential and is still being plagued by several shortcomings. Examples of this are, the often too low polymer coupling efficiencies resulting in low carbon loading, the questionable stability of the silica base at elevated temperature in fully aqueous conditions, or the less than optimal peak capacities which have been reached thus far.

In this contribution strategies are described to increase overall performance of temperature responsive columns in purely aqueous HPLC. This includes the development of improved coupling reactions between the polymers and the silica support, the study of alternative supporting materials and the development of temperature responsive stationary phases allowing for higher column efficiencies. Next to this, the applicability and potential of this promising separation mode will be illustrated through a number of (1D and 2D) applications.

Detection and Differentiation of Botulinum Neurotoxins for the Diagnosis and Prevention of Botulism (Seminar)

Author: John Barr, Centers for Disease Control and Prevention (CDC)
Country: United States
Co-Authors: Suzanne Kalb

Session: Clinical Hyphenations
Day & Time: Wednesday 24th January 2018, 15:10 - 15:30
Room: Ferrier Hall

Background/Aim
Botulinum neurotoxins (BoNTs) are the most poisonous substances known to mankind and cause the disease botulism. The analysis of trace amounts of protein toxins in highly complex matrices is an analytical challenge. We have developed and implemented a sensitive method that uses the enzymatic amplification of products with mass spectrometry to allow detection of BoNT and determine its serotype (A-G) in low attomole/mL. Furthermore, we have performed detailed proteomics analysis to aid epidemiologic investigations discovering sources of intoxication and commonality/differences between concurrent botulism outbreaks.

Methods
This method has three levels of selectivity including immunomagnetic extraction with monoclonal antibodies, enzymatic activity of the toxin on a peptide substrate that mimics the toxins natural, in vivo target, and specific detection of product peptides with MALDI TOF MS. The subtype and toxin variant are then identified by digesting the bead mixture with trypsin/chymotrypsin followed by nLC-MS/MS on a high resolution hybrid mass spectrometer.

Results
The mass spectrometry-based BoNT analysis can detect and differentiate all seven serotypes. The detection limits are less than 0.1 mouse LD50 (low attomole/mL) in stool, serum, and food and this method is much more rapid than the traditional mouse bioassay. The method has been used to investigate many botulism outbreaks and has confirmed botulism for foodborne, infant, adult colonization, and wound botulism. The addition of subtyping the toxins has added a new layer of information that has been used in several outbreaks across the United States.

Conclusions
The new BoNT method has allowed for more sensitive and rapid conformation of botulism. Serum now is routinely tested and we are now able to routinely detect BoNT in wound botulism cases. The subtyping has provided rapid information for botulism investigations and has provided a deeper understanding to the diversity of subtypes that are causing human botulism in the United States.

Suspect Screening of Aquatic Environmental Matrices Using High Resolution Analysis and in Silico Tools for Broad Scope Tentative Contaminant Identification (Seminar)

Author: Leon Barron, Analytical & Environmental Sciences Division, King's College London
Country: United Kingdom
Co-Authors:

Session: Other
Day & Time: Thursday 25th January 2018, 16:30 - 16:50
Room: Ferrier Hall

Characterising the breadth of emerging environmental contaminants is a dynamic challenge. Recent attention has focussed on liquid chromatography-high resolution mass spectrometry (LC-HRMS) for large and retrospectively mineable dataset capture. For preliminary identification of new/additional compounds, most works rely on HRMS data interpretation alone. Chromatographic data is often limited due to the unavailability and/or cost of reference standards for comparison. Prediction of retention has proved problematic especially for ionisable compounds under gradient conditions. Herein, a selection of analytical approaches developed for suspect screening of river water and wastewater in London is presented for compounds of both forensic and environmental interest. The work focuses on using both full-scan high resolution mass spectrometry data mining and machine learning tools for gradient chromatographic retention time prediction to tentatively identify new contaminants more rapidly. Data screening using these combined tools applied to river and waste water from the London area showed that the number of new compounds identified through in silico data processing halved in comparison to the use of HRMS alone. Approximately >30 additional compounds tentatively identified on any one day in influent/effluent wastewater and in receiving river water are discussed. The generalised performance of the approach was investigated using retention data for >1,100 compounds present in several complex matrices and across ten gradient reversed-phase LC-HRMS methods from different laboratories. Blind test predictions yielded an absolute accuracy of 1.02 ±0.54 min across all methods. Optimised and replicated network dependency on molecular descriptor data is also presented. Finally, recent advances using this approach to predict passive sampler uptake rate constants and combined retention time-collisional cross section predictions for methods using ion mobility - high resolution mass spectrometry (LC-IM-HRMS) are introduced.

Of mice, sex and mass spectrometry (Plenary)

Author: Robert Beynon, University of Liverpool
Country: United Kingdom
Co-Authors:

Session: Other
Day & Time: Friday 26th January 2018, 14:30 - 15:20
Room:

In my talk, I will explore how successful collaborations between protein chemists and evolutionary biologists can yield new insights in this area. The biologists bring the big ideas, while the protein chemists find sneaky and novel ways to test their hypotheses, developing a truly interdisciplinary team approach - a model of modern biological research. In my presentation, I will address the considerable role that different mass spectrometric modalities (GC-MS, LC-MS, LC-MS/MS, REIMS-MS) have played in understanding the chemistry and biology of the system.

Most of evolutionary biology and animal behaviour is about sex! Sex is, in part, mediated through proteins and their interactions with other proteins. Thus, the study of proteins involved in topics such as sexual reproduction, sperm competition, mate selection and inbreeding avoidance could provide valuable new insights into the drivers of speciation and evolution. These proteins evolve rapidly and bring problems when working with species for which there is no reliable annotated genome.

To illustrate, mouse urine is unlike normal human urine, particularly in protein content. The obligate proteinuria is considerable (it can exceed 50mg/mL), almost exclusively composed of Major Urinary Proteins (MUPs), 19 kDa beta-barrel lipocalins that are involved in chemical communication between individuals. They play multiple roles, including coding of owner identity and transport/slow release of bound volatile pheromones. The accessibility of the MUPs, the ease with which recombinant MUPs can be made and the ability to ‘close the loop’ with behavioural studies has greatly increased our understanding of these proteins, driven by detailed mass spectrometric characterisation.

Generic Reverse Phase Chromatography… Speed, Performance….or Both? (Seminar)

Author: Bob Boughtflower, GSK
Country: United Kingdom
Co-Authors:

Session: High Throughput versus High Efficiency Separations (CS)
Day & Time: Wednesday 24th January 2018, 17:10 - 17:30
Room: Lower Hall

The adoption of reverse phase generic gradient (U)HPLC methods of analysis has been widespread throughout many parts of the analytical measurement industry. However, it is commonplace to see practise that does not take advantage of the separation speed and performance that is available from modern materials and instrumentation. This presentation will attempt to demonstrate the performance that is easily accessible and what this means for choice of instrument and column formats. Examples will be shown of ultra-fast separations and conclusions drawn about where these methods can routinely be applied.

Considerations for the use of Ultra-High Pressures in Liquid Chromatography for 2.1mm Inner Diameter Columns (Seminar)

Author: Ken Broeckhoven, Vrije Universiteit Brussel
Country: Belgium
Co-Authors: Gert Desmet

Session: Fundamentals in Separation Science (KVCV)
Day & Time: Wednesday 24th January 2018, 14:30 - 14:50
Room: Lower Hall

The reduction of particle size is a time-proven method to decrease analysis time and improve the quality of chromatographic separations. Sufficiently long columns packed with small particles (sub-2µm) can however only be operated at their optimal velocity if sufficiently high operating pressures are available. This requires to consider the thermal effects that result from pumping a liquid through a porous medium. This so-called viscous heating or viscous dissipation of the mechanical energy increases the temperature of the mobile phase, column bed and hardware (wall, fittings, frits). The heat can either be removed at the column outlet (leading to an axial gradient in mobile phase temperature) or through the column walls (leading to a radial temperature gradient). This contribution gives an overview of the effects of viscous heat dissipation in chromatographic columns (with an emphasis on so-called narrow bore columns with an inner diameter of 2.1mm) using numerical simulations of the temperature and velocity profiles and the resulting band broadening, for the first time at operating pressures up to 2000 bar, i.e. the expected operating pressure of LC instruments of the future. When operating columns under well-thermostatted conditions to maintain a constant temperature of the mobile phase, a dramatic increase in plate heights can be observed that voids any advantage one could expect from the possibility to use smaller particles offered by the increased pressure limit. In practice, most columns are placed in thermostatted column compartment (still-air oven), where, due to natural convection, a significant loss of heat will take place, resulting in local radial temperature and velocity gradients in the column. Although these gradients have an opposite sign at the front and the back of the column, these effects do not entirely compensate each other, especially when the bulky column endfittings are taken into account. Thus even when the column is not actively temperature controlled a significant loss in performance under standard operating conditions can be expected for operating pressure above 1250 bar, which increases with increasing temperature dependency of the retention factor. In addition, unprecedented experimental measurements of the temperature effects at an operating pressure up to 2600 bar were performed on a 10cm long, 2.1mm ID column showing a dramatic temperature increase up to 60°C relative to the inlet temperature when using methanol as a mobile phase. These results make it clear that drastic changes in column or instrument hardware are required before a further increase in operating pressure up to 2000 bar and beyond can become possible when using single, short (5-15cm) columns, such as an even further reduction in column ID (down to and below 1mm) with a concomitant decrease in instrument dispersion, the development of pressure capable column hardware with a very low wall conductivity or the availability of a cheap and user friendly vacuum housing for columns.

Methodologies to Determine b-term Coefficients Revisited (Seminar)

Author: Deirdre Cabooter, University in Leuven
Country: Belgium
Co-Authors: Gert Desmet, Donatela Sadriaj, Huiying Song

Session: Challenges in Quantitative Analysis
Day & Time: Thursday 25th January 2018, 11:00 - 11:20
Room: Lower Hall

The accuracy of the longitudinal diffusion term (b-term) plays a vital role in the study of mass transfer mechanisms in high performance liquid chromatography (HPLC). In this presentation, three commonly used methodologies (peak parking; fitting of an experimental plate height curve; and the so-called dynamic method) for the determination of the b-term constant will be discussed in detail. The three methods will be compared based on their mutual agreement, the intra- and inter-day variation of the obtained values and the time required to measure them. It will be demonstrated that whereas the dynamic method is often plagued by impractically long waiting times and concomitant baseline variations compromising accurate measurements of the band broadening, the two other methods lead to very similar b-values, i.e., well within the 1% RSD inter-day variation typically marking both methods in the present study. It will moreover be shown that the best way to study the agreement of the peak parking and plate height fitting method is in a plot of h.v versus v, providing a much better zoom on the b-term region of the van Deemter curve than the customarily employed h versus v-curve and hence allowing to identify any anomalous measurement values (usually related to measurements with a long experimentation time). Finally, the implications of a certain variation in b-term value for the further determination of column performance parameters will be discussed.

Separation and Characterisation by Ion Mobility-Mass Spectrometry (Tutorial)

Author: Antonio Calabrese, University of Leeds
Country: United Kingdom
Co-Authors:

Session: Separations by Ion Mobility
Day & Time: Thursday 25th January 2018, 10:30 - 11:00
Room: Room D

Ion mobility spectrometry involves the separation of ions in the gas phase based on their mass, collision cross-section (size, shape) and charge. Whilst ion mobility has found use as a standalone technique (for example in the detection of explosives and narcotics), it is often combined with mass spectrometry and high-performance liquid chromatography. Ion mobility can be used to separate analytes from the sample background, separate charge states and resolve isobaric ions. Moreover, collision cross-sections can be used to validate analyte identifications, or provide structural information for both small and large molecules and complexes. In particular, ion mobility-mass spectrometry has emerged as an invaluable tool in the structural biology toolkit. Several ion mobility devices (e.g. drift tube, travelling wave, FAIMS and TIMS) have been developed and commercialized. In this presentation, the theory of ion mobility, commercially available devices, and several applications will be highlighted, demonstrating the utility of ion mobility-mass spectrometry in a range of Omics fields.

Kinetics and Mass Transfer Phenomena in Modern Chiral Stationary Phases (Seminar)

Author: Alberto Cavazzini, University of Ferrara
Country: Italy
Co-Authors: Martina Catani, Francesco Gasparrini, Omar H Ismail

Session: Fundamentals in Separation Science (KVCV)
Day & Time: Wednesday 24th January 2018, 15:10 - 15:30
Room: Lower Hall

In the last decades, several examples of chiral stationary phases (CSPs) prepared on particles (both fully porous and pellicular) suitable for high efficient, ultrafast enantioseparations have been presented. These phases have been successfully employed to perform sub-second separations of several classes of enantiomers.

Contrary to what happened in achiral reversed phase separations especially on C18 columns, however, the fundamentals of mass transfer phenomena on modern CSPs have been barely studied. In this work, state-of-art techniques have been employed to study mass transfer in modern CSPs. To this scope, different kinds of CSPs have been considered including teicoplanin-based and Whelk O1 CSPs. Teicoplanin-based SPPs of 2.6 and 2.0 µm particle diameter and Whelk-O1 FPPs of 2.5 and 1.9 µm were employed in our study.

The results of this investigation reveal some unexpected facts. In particular, they oppose the idea of the supposed superiority of chiral SPPs over their fully porous counterparts to achieve high efficient, ultrafast enantioseparations.

Use of Different Computer-aided Method Development Software in Late Stages Across Global Sites in Pharmaceutical Industry (Seminar)

Author: Kai Chen, Janssen Pharmaceutical Companies of Johnson & Johnson
Country: Belgium
Co-Authors: Jean-Paul Boon, Peter Van Broeck, Mario Hellings

Session: High Throughput versus High Efficiency Separations (CS)
Day & Time: Thursday 25th January 2018, 14:50 - 15:10
Room: Ferrier Hall

In contrast to early development, method development in late development (LD) in pharmaceutical industries is aimed to provide a robust method for right-first-time method transfer and lifecycle management. Systematic, scientific, risk-based, multivariate and proactive approaches are therefore needed to be already applied in the method development stage.

By considering the current practice of liquid chromatographic method development, certain gaps are identified towards these goals. Many methods from earlier phases are developed on the screening platform and optimized from time to time mostly dependent on analyst's personal experience and preference. The trial-and-error and one-factor-at-a-time (OFAT) methodology enhances chance of missing the optimal conditions and brings larger variance in method output. Furthermore, although most global sites are equipped with computer-assisted LC method development software, the use of different stationary and mobile phases in screening, along with the application of different algorithms/strategies in optimization often results in different optimal method outputs from these softwares. In addition, robustness estimation is lacking so that the performance of the developed method cannot be foreseen for follow-up method validation, transfer and lifecycle management.

In this presentation, the current practices of using computer-assisted LC method development software in different sites are compared. The advantages and disadvantages of each practice with software like AutoChrom, ChromSword, DryLab and Fusion are concluded. A harmonized work flow is proposed aiming to obtain same/similar method developed from different sites, by minimizing the impact of the native differences in instrumentation and software and avoiding overcomplicated experiment design or method development. A pilot testing is performed to evaluate the proposed work flow with a real project sample. By this, sources of variability that may lead to development deviation or poor method robustness are identified for further improvement.

FAIMS Mass Spectrometry for the Analysis of Peptides and Proteins (Seminar)

Author: Helen Cooper, University of Birmingham
Country: United Kingdom
Co-Authors:

Session: Ion Mobility - Mass Spectrometry (BMSS)
Day & Time: Thursday 25th January 2018, 17:10 - 17:30
Room: Lower Hall

High field asymmetric waveform ion mobility spectrometry (FAIMS), also known as differential ion mobility spectrometry, is emerging as a powerful technique in biomolecular analysis. FAIMS relies on differences in ion mobility in high and low electric fields to achieve gas phase separation of ions at atmospheric pressure, offering advantages including reduced interference from ions of similar mass-to-charge (m/z), and separation of isomers and positional variants.

Here, we demonstrate the application of FAIMS in proteomics analyses and in ambient surface analyses. The inclusion of FAIMS in large-scale phosphoproteomics analysis of the fibroblast growth factor signalling pathway resulted in an increase in the relative proportion of pThr and pTyr and increase in the identification of multiply-phosphorylated peptides. The application of FAIMS to the study of O-glycosylation in flagellin from Campylobacter jejuni 11168 revealed multiply- and differentially glycosylated peptides. We also show that by coupling liquid extraction surface analysis (LESA) with FAIMS significant improvements in the direct analysis of complex biological samples are achieved. The inclusion of FAIMS results in improved signal to noise ratios, shorter acquisition times, and separation of molecular classes, e.g., proteins and lipids. The benefits afforded by FAIMS suggest that the technique could find applications in LESA mass spectrometry imaging protocols. We show LESA FAIMS mass spectrometry imaging of proteins in sections of mouse brain and liver tissue, and compared those results with LESA mass spectrometry images obtained in the absence of FAIMS.

Opportunities for Ultra-Rapid LC-MS/MS in High-Throughput Bioanalysis (Seminar)

Author: Lewis Couchman, Analytical Services International (ASI) Ltd.
Country: United Kingdom
Co-Authors:

Session: Advances in Clinical Analysis
Day & Time: Wednesday 24th January 2018, 14:30 - 14:50
Room: Ferrier Hall

Typically, targetted quantitative LC-MS/MS analyses using gradient elution are carried out at the rate of a few minutes per injection. In this paper, an approach for ultra-rapid LC-MS/MS analysis will be described in which complete injection-to-injection cycle-times are just 30 seconds, equating to at least one 96-well plate per hour. These cycle times are more akin to those of direct injection methods such as flow-injection analysis, but despite this include efficient chromatographic separation of target analytes. This enables

(i) the removal of interference from isobaric compounds (e.g. from other drugs and/or metabolites) and also

(ii) the reduction of ionization effects caused by co-eluting matrix components. In this presentation, application of the approach will be demonstrated using examples from therapeutic drug monitoring and clinical and forensic toxicology. The practical considerations for implementation of the approach to dramatically speed up other analyses will be discussed.

Quantitative Drug Metabolite Profiling Without Authentic Standards or Radiolabels using LC-ICP-MS (Seminar)

Author: Filip Cuyckens, Janssen R&D
Country: Belgium
Co-Authors:

Session: Other
Day & Time: Wednesday 24th January 2018, 17:10 - 17:30
Room: Ferrier Hall

Radiotracer technology (14C or 3H) still is the method of choice to study the in vivo disposition of a new drug. Due to its structure independent response and selectivity it enables the quantitative detection of the parent drug and all of its metabolites in verbonden metcomplex matrices and in the absence of authentic standards. There can, however, be ethical reasons or cost concerns that hinder the use of radiotracers, e.g., no radiolabels are usually available in discovery or used in first-in-human studies.

Inductively coupled plasma - mass spectrometry (ICP-MS) allows the detection of elements that are often encountered in drug molecules. Since the intensity of the signal obtained with ICP-MS is also independent of the chemical structure analysed it can be a valuable alternative for radioactive detection.

LC-ICP-MS methods were developed for the accurate quantification of bromine, chlorine and sulphur containing drug molecules and their metabolites. Online isotope dilution, reverse online isotope dilution or a mathematical correction5 were applied to correct for the change in instrumental response inherent to the changing eluent composition during the LC gradient.

The quantitative results obtained by LC-ICP-MS and radio-LC analysis on the same in vivo samples were in very good accordance. The sensitivity of the LC-ICP-MS detection showed to be comparable or better than with radioactive detection. Bromine quantification with LC-ICP-MS on patient samples was used, for the first time ever, as an alternative for a traditional radioactive human ADME study.

Finally, simple derivatization strategies were developed introducing ICP-MS detectable hetero-elements in compounds containing an amine, phenol and/or carboxylic acid moiety, making the analysis and quantification possible of compounds normally not detectable with ICP-MS.

Detection of Bacteriologically Produced Hydrogen Sulphide using SHS-MCC-GC-IMS (Seminar)

Author: John Dean, Northumbria University
Country: United Kingdom
Co-Authors: John Perry, Stephen Stanforth, Ryan Thompson

Session: Clinical Hyphenations
Day & Time: Friday 26th January 2018, 11:40 - 12:00
Room: Ferrier Hall

Hydrogen sulphide (H2S) is a volatile airborne compound with the characteristic rotten egg odour at low concentrations, and is commonly associated with bacterial contamination of food and water sources. Current tests employed for the detection of bacterial H2S rely on specialised growth mediums that are rich in sulphur, usually in the form of sodium thiosulphate, cysteine, or cystine, to induce significant production of H2S. These mediums are also combined with a visible colour change following incubation; usually facilitated via ferric ammonium citrate or lead acetate, both of which form a black precipitate upon contact with H2S.

One of the primary drawbacks of current methods are the subjective nature of the results interpretation, which essentially ask the question 'how black is black?' We propose a new method for rapid and sensitive detection of H2S using static headspace - multi capillary column - gas chromatography - ion mobility spectrometry (SHS-MCC-GC-IMS). Our new method allows for sensitive detection and quantification of H2S evolved into the gaseous headspace above a bacterial suspension. Fresh colonies taken from day old Tryptone Soy Agar plates were suspended in nitrate broth and adjusted to an inoculum of 105 CFU/ml, which were then sealed in headspace vials and incubated overnight at 37°C. Following incubation, 2.5 mL of the gaseous headspace above the suspension was extracted and analysed via SHS-MCC-GC-IMS. Nitrate broth (potassium nitrate 1 g/L, peptone 5 g/L, meat extract 2.4 g/L, pH 7.4)3 was the chosen medium due to its non-selective nature and inexpensive constituents, thereby removing the need for expensive specialised growth media.

Hydrogen sulphide detection via MCC-SHS-GC-IMS was found to be extremely sensitive, allowing for quantification as low as 5 ppb. A diverse range of bacteria were tested, many of which the H2S production status was undefined by current methods, such as B. cereus and E. cloacae. Interestingly, this method also detected consistent H2S production in 2 of 6 tested E. coli strains, a species which is largely classified as H2S negative according to standard testing procedures4.This method shows great promise for a rapid and extremely sensitive analytical technique for the detection and quantification of H2S. Future applications could include detection of bacterial contamination in food and water sources, and bacteria in various human clinical samples e.g. blood from suspected sepsis patients.

Topology discrimination of saponin ions by Hyphenated Mass Spectrometry techniques and computational chemistry (Seminar)

Author: Corentin Decroo, Umons
Country: Belgium
Co-Authors: Jerome Cornil, Patrick Flammang, Pascal Gerbaux

Session: Ion Mobility - Mass Spectrometry (BMSS)
Day & Time: Thursday 25th January 2018, 11:20 - 11:40
Room: Room D

Because of their huge biological and pharmacological activities, saponins arouse nowadays an increasing interest. When deciphering the biological activities or the potential applications of natural products, the structural characterization of the targeted molecules must be performed with great many details. Generally, the structural characterization of biomolecules, like saponins, is realized based on NMR measurements on purified and isolated molecules. Nowadays, mass spectrometry coupled with liquid chromatography appears to be an inescapable tool for the determination of molecular structures since no extreme purification nor isolation is mandatory to identify molecules. The structure of saponin is often derived from tandem mass spectrometry experiments, with a special interest in collision-induced dissociation experiments. Unfortunately, for saponin ions, MSMS appears to be not sufficient because of similar decomposition pathways between isomers and even between congeners. In the present work, we consider ion mobility spectrometry (IMS) as an orthogonal tool for the gas phase separation of saponin isomers. Indeed, this technique allows for the separation of gas-phase ions based on their mobilities in an electric field when submitted to collision against a countercurrent inert gas. IMS can go far beyond ion separation when considering the measurement of the collision cross sections (CCS) of the saponin ions. In first approximation, the CCS data is highly related to the 3D structure of the gaseous ions and is then intrinsically related to the molecular structures of the ionized molecules. Nevertheless, CCSexp measurements only become relevant from a structure analysis point of view upon the direct comparison with the CCS calculated for candidate ion structures generated upon theoretical calculations - CCSth. In the present study, we submitted to the IMS/theoretical chemistry combination selected saponins presenting model structures. The different molecules are extracted from different plants such as soy (Glycine max) and quinoa (Chenopodium quinoa) but also from marine animals like sea cucumbers, Holothuria forskali. By this selection, we get access to different structures such as monodesmosidic and polydesmositic saponins. We here present our experimental and theoretical results revealing the potentialities and the weaknesses of IMS for the structural characterization of saponins.

Recent Progress in the Development of Perfectly Ordered Separation Media (Keynote)

Author: Gert Desmet, Vrije Universiteit Brussel
Country: Belgium
Co-Authors:

Session: Fundamentals in Separation Science (KVCV)
Day & Time: Wednesday 24th January 2018, 14:00 - 14:30
Room: Lower Hall

The present contribution aims at illustrating and demonstrating how micro-machining technology can boost High Performance Liquid Chromatography (HPLC). Currently, HPLC is routinely used in nearly every chemical analysis lab. Despite its high degree of maturity, the technique still does not deliver the required separation power needed to unravel the complex samples encountered in the state-of-the-art research in biology and drug development (e.g., proteomics and metabolomics), or in contemporary food and environmental analysis, etc.

One of the reasons for the performance limitations of packed bed HPLC columns is that they are packed randomly. This randomness forces the liquid to follow different paths with different path lengths, which in turn broadens the individual sample component bands. To solve this packing disorder problem, we have used advanced photolithographic etching techniques such as the Bosch-process to produce perfectly ordered porous support columns with optimized hydrodynamic shape and optimized external porosity.

Using this approach, we have been able to realize sub-micrometer plate heights using radially elongated diamond-shaped pillars that are up to 15 times wider than their axial dimension (5 µm). The use of such a high-aspect ratio pillars allowed for a 5-fold reduction of the minimal plate height compared to beds filled with pillars with a similar inter-pillar distance (2.5 µm) but with an aspect ratio around unity (cylinders, diamonds). This increase in performance can be largely attributed to a decrease of the B-term band broadening, which is about a factor of about 25 smaller in the large-aspect ratio columns compared to the cylindrical pillar columns. In addition, the columns also generate only a minimal C-term band broadening, as the space formed between the high-aspect ratio pillars is very uniform and basically resembles the through-pore space one would have in a parallel array of flat plates. The concept also enables a drastic reduction of the footprint of pillar array columns, allowing to fabricate columns offering very high efficiency on the surface of a single silicon wafer.

Recent Advances in the Analysis of Petroleum-based Fuels using Gas Chromatography-Vacuum Ultraviolet Spectroscopy (Seminar)

Author: James Diekmann, VUV Analytics
Country: United States
Co-Authors: Jack Cochran, Philp Walsh, Dan Wispinski

Session: Analysis of Complex Energy Products
Day & Time: Friday 26th January 2018, 13:50 - 14:10
Room: Lower Hall

Gasoline is the primary transportation fuel in our society today and plays a pivotal role in the global economy. One of the many ways refineries across the world analyze their finished fuel products is by gas chromatography - flame ionization detector (GC-FID). Typically, "detailed hydrocarbon analysis" (DHA) or a multidimensional GC (MDGC) approach is used for determining hydrocarbons in these complex samples. DHA is a 3-hour method that requires a pre-column that must be "tuned" connected to a 100m x 0.25mm x 1.00µm GC column to separate and speciate as many compounds as possible. The MDGC analysis is a 1-hour method that uses a complicated series of valves and columns and traps to separate and group by compound classes and carbon numbers.

Recently, VUV Analytics developed an alternative method for gasoline hydrocarbon analysis using GC - Vacuum Ultraviolet (VUV) absorbance spectroscopy for finished gasoline and gasoline-range product streams. Unlike DHA and MDGC, GC-VUV simplifies the setup and speeds up the analysis by using a single 30m x 0.25mm x 0.25µm polydimethylsiloxane GC column. This 30-minute method employs rapid spectroscopic deconvolution to produce hydrocarbon class and carbon number breakdown for samples, while still providing some compound speciation (e.g., iso-octane, benzene, toluene, ethylbenzene, xylenes, naphthalene, methylnaphthalenes, and select oxygenates). The method has been propagated as ASTM D8071-17, Standard Test Method for Determination of Hydrocarbon Group Types and Select Hydrocarbon and Oxygenate Compounds in Automotive Spark-Ignition Engine Fuel Using Gas Chromatography with Vacuum Ultraviolet Absorption Spectroscopy Detection (GC-VUV).

In this presentation, we present the recent advances in the characterization of petroleum-based fuels using GC-VUV, as an alternative to current techniques that are time-consuming, cumbersome, and/or complicated. We investigate the possibility of reducing the GC-VUV runtime by half with modifications to simple GC parameters such as flow rate and oven ramp. We also highlight the feasibility of speciating compounds outside the scope of ASTM D8071.

Strategies to Optimize Throughput in 2D-LC (Seminar)

Author: Monika Dittmann, Agilent Technologies
Country: Germany
Co-Authors: Stephan Buckenmaier, Konstantin Shoykhet

Session: High Throughput versus High Efficiency Separations (CS)
Day & Time: Wednesday 24th January 2018, 16:30 - 16:50
Room: Lower Hall

Two-dimensional liquid chromatography (2D-LC) is becoming a routine technology as more and more instrument solutions are becoming commercially available from instrument vendors.

Although 2D-LC is inherently a high-resolution technique, it is still worthwhile to consider possibilities to optimize the analysis time required to achieve a given separation goal. This optimization should mostly aim at increasing the throughput of the 2nd dimension analysis as this determines the total separation time.

The main operation modes in 2D-LC are full-comprehensive 2D-LC for very complex samples, heart cutting and multiple heart cutting (selective comprehensive) for samples of low to medium complexity.

The optimization strategy for full-comprehensive operation aims mostly at achieving a desired peak capacity in the shortest time possible. In this context, the gradient kinetic plot model (KPM) can be employed to find the best column dimensions and operating conditions for the 2nd dimension (particle size, column length and ID, flow rate, temperature etc.). The model takes limiting factors such as maximum operating pressure or flow rate into account. An extended version of the KPM also includes the impact of external band-broadening that limits the use of very short, narrow columns.

Another means to improve peak capacity per times the use of shifted gradients, i.e. adjustment of 2nd D gradient start and end conditions depending on the sampling point within the 1stD gradient.

If the sample is only of limited complexity or if only certain parts of the 1D chromatogram are of interest, selective comprehensive 2D-LC can be used to achieve the desired separation in a shorter time by analyzing only the areas of interest. In this case the optimization might aim more at achieving certain selectivities (through selection of stationary phases, 2D eluent, gradient range and slope) than aiming exclusively at optimizing peak capacity. Here a combination of kinetic optimization and retention modeling can be useful.

The authors will discuss the different optimization strategies and the factors limiting speed in multidimensional analysis.

Green Bioanalytical Analysis of Voriconazole and Tadalafil by HPLC (Seminar)

Author: Aysegul Dogan, Hacettepe University Faculty of Pharmacy Department of Analytical Chemistry

Country: Turkey

Co-Authors: Nursabah E. Basci

Session: Green Separations
Day & Time: Friday 26th January 2018, 13:30 - 13:50
Room: Room D

Green chromatography techniques are a developing area in the scientific research studies due to their security for environment and human health. Greening procedures in HPLC techniques may be based on reduction of toxic solvent usage and/or the replacement of toxic solvents with less or non-toxic ones which were friendly to the environment. In this study; 30 drug molecules having different physicochemical properties analyzed using firstly with conventional organic modifiers, then with propylene carbonate included mobile phases and three of the molecules with the best chromatographic separation were selected for analyzing from plasma. Green HPLC methods of voriconazole, tadalafil and phenazopyridine (internal standart) was developed and applied in the light of model molecules, voriconazole and tadalafil, to the analysis from spiked plasma samples. For this purpose, non-toxic propylene carbonate and ethanol was used as solvents, and new generation low particle sized small column and microflow high performance liquid chromatography technique approaches was also tested. Shim-Pack XR-ODS (100 x 2.0 mm, 2.2 µm) analytical column was utilized for the analysis of voriconazole and tadalafil by using phenazoprydine as internal standart, and diode array dedector was set to 256 nm for voriconazole and 284 nm for tadalafil. Mobile phase composition was constracted as PC/(% 70 phosphate buffer (50 mM, pH: 3.0) + % 30 EtOH) (10:90, v/v) at a flow rate 0.3 mL min-1. Developed analysis methods were validated in terms of stability, linearity, accuracy, precision, specificity, ruggedness and robustness. Developed and optimized methods were tested for their applicability and relaibility plasma analysis by analytical/bioanalytical method validation strategies and according to the findings the methods were found to be accurate, precise, specific, sensitive, rugged, robust and reliabile in long term use.

Probing selectivity of mixed-mode reversed-phase / weak-anion-exchange columns for small-molecule separations in liquid chromatography (Seminar)

Author: Jose Luis Dores-Sousa, Department of Chemical Engineering, Vrije Universiteit Brussel
Country: Belgium
Co-Authors: Sebastiaan Eeltink

Session: Fundamentals in Separation Science (KVCV)
Day & Time: Wednesday 24th January 2018, 11:00 - 11:20
Room: Room D

Mixed-mode chromatography has been used in life science research for a long time, however a good understanding of the effects of mobile-phase composition on small molecules retention has still not been established. Due to presence of multimode retention mechanisms, method development is troublesome and time consuming. The current study comprises a systematic investigation to assess retention properties and selectivity of a mixed-mode reversed-phase / weak-anion-exchange (RP/WAX) stationary phase. Retention was investigated for different compound classes including aromatic hydrocarbons, halogenated aromatic hydrocarbons, phenols, and carboxylic acids, which vary in hydrophobicity, Van der Waals surface area, and charge. kw values of the linear solvent-strength model changed proportional to the log P value for non-charged analytes, when varying the ACN content in the mobile phase. Significantly lower S values (factor 2) were observed for phenols and aromatic acids compared to neutral analytes, suggesting the presence of a dual RP/WAX retention mechanism. The stoichiometric displacement net-charge model was applied to describe ion-exchange retention behavior when varying the salt concentration in the mobile phase. The IEX capacity of the mixed-mode column was also evaluated and at pH 6 anionic analytes exhibited maximum retention. Van't Hoff plots indicated that temperature significantly affect analyte retention in case of RP partitioning (aromatic hydrocarbons and halogenated aromatic hydrocarbons) and also in the case of a mixed-mode retention behavior (for aromatic acids).

Exploiting comprehensive two-dimensional liquid chromatography in food analysis (Keynote)

Author: Paola Dugo, University of Messina
Country: Italy
Co-Authors:

Session: Advanced Analysis of Food and Beverages
Day & Time: Friday 26th January 2018, 12:40 - 13:10
Room: Ferrier Hall

Food chemistry is continuously involved in the characterization of nutraceuticals viz. mole-cules with beneficial effects on human health. Selective and sensitive analytical methods should be capable to allow the determination of the main components occurring in food real-world-samples.

The power of multidimensional "comprehensive" liquid chromatography (LCxLC) methods, along with recent advances in mass spectrometry (MS), have enabled a much deeper insight into the true qualitative and quantitative composition of samples of food interest. The most striking beneficial aspect is the combination of two or more independent separation steps, in-creasing significantly the separation power of the corresponding one-dimensional LC counter-part.

Our research group have been actively engaged in the development and implementation of various LCxLC methodologies using different instrumental set-ups and column combination. In this contribution, food applications in the field of LCxLC will be presented, especially em-ploying reversed phase conditions in both separation dimensions (RP-LCxRP-LC).

Analysis of food products using advanced analytical techniques (Tutorial)

Author: Paola Dugo, University of Messina
Country: Italy
Co-Authors: Francesco Cacciola, Luigi Mondello, Francesca Rigano

Session: Other
Day & Time: Thursday 25th January 2018, 16:00 - 16:30
Room: Room D

The tutorial is focused on the use of different analytical techniques applied to the analysis of food products. The latter are very complex mixtures containing many nutrients of organic (li-pids, carbohydrates, proteins, vitamins) and inorganic (water, minerals, oxygen) nature but also xenobiotic substances that can come from technological processes, agrochemical treatments or packaging materials e.g. residues of pesticides, drugs, migrants from packaging, etc. Despite chromatographic techniques are of wide use, the great technological advances made in the mass spectrometry field, over the last decade, apparently diminished the need for a high-resolution chromatography step.

The benefits and disadvantages of each method will be discussed covering specific analytical needs in terms of characterization and fingerprinting of compounds of food interest.

Advancing hydrophobic interaction chromatography methods to characterize biotechnology enzyme mixtures and to profile biotherapeutics (Keynote)

Author: Sebastiaan Eeltink, Vrije Universiteit Brussel
Country: Belgium
Co-Authors:

Session: (R)evolutions in Biopharmaceutical Analysis (KVCV)
Day & Time: Thursday 25th January 2018, 10:30 - 11:00
Room: Lower Hall

The need for comprehensive characterization of protein-derived macromolecules applied in the biopharmaceutical and biotechnology industries is increasing rapidly. The biological activity and/or enzymatic reactivity of these biomolecules strongly depend on their chemical composition and three-dimensional structure. To obtain information on those characteristics it is important to maintain the original structural composition of bio-macromolecules during the analysis. Hydrophobic interaction chromatography (HIC) capitalizes the interaction between hydrophobic patches of proteins and weakly hydrophobic ligands attached to the stationary phase. HIC method development is a major bottleneck since effects of salt systems, buffers, and stationary-phase chemistry on protein retention are poorly understood.

The effects of kosmotropic/chaoptropic salt systems and the addition of mobile-phase additives (calcium and isopropanol) on retention have been assessed. The hydrophobicity index appeared to be a good indicator to predict the elution order of intact proteins in HIC mode. Furthermore, the effect of adding additives in the mobile phase, such as calcium chloride (stabilizing the 3D conformation of -lactalbumin) and isopropanol, on retention properties has been assessed. Results indicate that HIC retention is also governed by conformational changes in the proteins which affect the number of accessible hydrophobic moieties. Furthermore, method-development (MD) strategies will be discussed and the possibilities and limitations to develop methods for the separation of intact proteins in HIC mode, based on retention-time models in combinations gradient scouting runs. Finally, the potential of HIC has been assessed to profile biotechnology mixtures and biotherapeutics, including monoclonal antibodies and anti-body drug conjugates

LC-MS/MS chiral ananlysis of chloramphencicol in the environment (Seminar)

Author: Felicity Elder, University of Bath
Country: United Kingdom
Co-Authors: Erika Castrignano, Barbara Kasprzyk-Hordern

Session: Screening Enviroment Pollutants, what can the data tell us?
Day & Time: Thursday 25th January 2018, 17:10 - 17:30
Room: Room D

Antibiotic resistance has become a global health issue, with the World Health Organisation stating that 'AMR threatens the very core of modern medicine' and that the 'crisis must be managed with the upmost urgency'. In order to manage AMR not only do we need a thorough understanding of the different types of AMR but also the drivers behind their development and spread. What is becoming increasingly clear is that antibiotics are accumulating in our environment due to their extensive use in human and veterinary medicine, leading them to be classed as an emerging contaminant. This accumulation could be one of the possible drivers of antibiotic resistance that we need to understand to combat the threat of AMR.

Chloramphenicol is a broad spectrum bacteriostatic antibiotic. It was first isolated in 1947 and by 1949 due to its relatively simple chemical structure was the first antibiotic to be made solely synthetically in the enantiomerically pure form. In the UK chloramphenicol is almost solely used for the topical treatment of Otis externa or bacterial conjunctivitis, and rarely used systemically due to its toxic side effect. These toxic side effects also mean its use in food producing animals is banned. However, in 2005 the over the counter sale of topical chloramphenicol eye drops or ointment was approved in the UK leading to an increase in sales. This increase in the consumption of chloramphenicol eye drops and the uncertainty as to how they are being disposed of poses a possible driving factor for the development of chloramphenicol resistance within the population.

A chiral LC-MS/MS method has been developed and validated for the two chloramphenicol enantiomers R,R-(-)-chloramphenicol and S,S-(+)-chloramphenicol in wastewater and broth. This included the optimisation of a solid phase extraction method for sample clean up and concentration, followed by development of a LC-MS/MS method utilising an AGP chiral column coupled to a triple quadrupole mass spectrometer for the quantitative analysis of the two chloramphenicol enantiomers. This method has since been applied to quantify the two chloramphenicol isomers in wastewater and to study its degradation within laboratory microcosm studies to help further our understanding of the fate of chloramphenicol within the environment.

Data to decision: efficient processing of complex petroleomics data (Seminar)

Author: Samuel Ellick, University of Bristol
Country: United Kingdom
Co-Authors: Christopher Arthur, Paul Gates, Samuel Whitmarsh, Christianne Wickking

Session: Big Data Chemometrics and Method Development(In-Silico)(KVCV)
Day & Time: Thursday 25th January 2018, 14:30 - 14:50
Room: Room D

The information age is truly upon us and concepts like big data are becoming commonplace in both academic and industrial settings. In the analytical chemistry space the advancement of increasingly sophisticated instruments means more data from our samples, and with it, a larger requirement for effective data handling. This presentation discusses relevant data processing techniques for handling large complicated data sets demonstrated on one of the world's most complex samples: petroleum. High resolution mass spectrometry analysis of petroleum can yield in excess of 10,000 signals per spectra; this data is often of little value in its raw form, and so only hyphenating with advanced data handling and visualisation strategies can we generate insight and answer our difficult questions.

"Python" is a versatile and relatively easy to use object orientated programming language. Python scripts can interface with analysis software packages like KNIME and JMP, separating the user from the complexity of coding whilst automating file handling and visualisation. With this combined ensemble of tools, it is possible to, filter, collate, recalibrate and assign HRMS petroleomics data enabling enhanced visualisations and access to multivariate analysis/statistics.

From this presentation I will demonstrate how powerful these techniques can be and how accessible they are. The complexity of the questions we ask our data and speed at which we expect answers to be returned is ever increasing. Analytical chemists in the 21st century therefore need to be increasingly literate in such data processing tools. This presentation aims to highlight this need using practical, real-world examples.

Characterization of Small Heterogeneities in Polymers by Analysis of UPLC/ESI-MS Reconstructed Ion Chromatograms (Seminar)

Author: Ruben Epping, Bundesanstalt für Materialforschung und -prüfung (BAM)
Country: Germany
Co-Authors: Jana Falkenhagen

Session: Hyphenated Techniques for Comprehensive Analysis
Day & Time: Thursday 25th January 2018, 14:50 - 15:10
Room: Room D

From simple molar mass disperse homopolymers over copolymers to functionalized, 3-dimensional structures containing various distributions, the complexity of polymeric materials has become more and more sophisticated in recent years. With applications in medicine, pharmacy, smart materials or for the semiconductor industry the requirements for the characterization have risen with the complexity of the used polymers. For each additional distribution, an additional dimension in analysis is needed. Small, often isobaric heterogeneities in topology or microstructure can usually not be simply separated chromatographically or distinguished by any common detector. Instead of a complicated, time consuming and/or expensive 2d-chromatography or ion mobility spectrometry (IMS) method, that also has its limitations, here a simple approach using size exclusion chromatography (SEC) coupled with electrospray ionization mass spectrometry (ESI) is proposed. We used SEC for the separation because unlike other separation modes the separation in this mode solely should occur due to the hydrodynamic volume with no interference of other interactions. This simplifies the interpretation and the above mentioned heterogeneities should show a slight difference in hydrodynamic volume. ESI mass spectrometry can offer more than an access to mass dependent information like MMD, end group masses or CCD in polymer analysis. The online coupling to SEC allows the analysis of reconstructed ion chromatograms (RIC) of each degree of polymerization. While a complete separation often cannot be achieved, the derived retention times and peak widths lead to information on the existence and dispersity of heterogeneities in microstructure or topology, that are otherwise inaccessible or accessible only by time consuming or expensive methods. Because these heterogeneities might vary with the molar mass, analysis of the whole MMD-Peak (here the total ion current (TIC)) would not lead to the desired information. The broadening of the chromatographic peaks in this case does not origin from the already well known band broadening factors in chromatography from diffusion. This band broadening is attributed to the nature and composition of the analyte itself. Surprisingly there is very little investigation into the peak width or peak shape due to analyte structure itself found in literature. It is also shown, that with proper calibration even quantitative information could be obtained. This method is suitable to detect small differences in e. g. branching, topology, monomer sequence or tacticity and could potentially be used in production control of oligomeric products or other routinely done analyses to quickly indicate deviations from set parameters. Based on a variety of examples we demonstrate the possibilities and limitations of this approach.

Understanding phosphorylation-mediated effects on NF-_B interactions using IM-MS (Seminar)

Author: Claire Eyers, University of Liverpool
Country: United Kingdom
Co-Authors:

Session: Ion Mobility - Mass Spectrometry (BMSS)
Day & Time: Thursday 25th January 2018, 16:30 - 16:50
Room: Lower Hall

The NF-κB system is a critical signalling pathway mediating (amongst other things) cellular responses to inflammation and DNA damage. The five NF-κB family members are transcription factors that are extensively regulated by phosphorylation, which regulates its ability to dimerise and to bind DNA, although the precise regulatory mechanisms of many of these sites remain to be elucidated. Using a combination of ‘bottom-up’ phosphosite mapping and ‘native’ ion mobility mass spectrometry, we can start to delineate the roles of individual sites of phosphorylation on NF-κB interactions, stability and dynamics.

Possibilities of modern size-exclusion chromatography for therapeutic proteins; feasibility assessment for future mass spectrometry hyphenation (Seminar)

Author: Szabolcs Fekete, University of Geneva
Country: Switzerland
Co-Authors: Alexandre Goyon, Davy Guillarme, Jean-Luc Veuthey

Session: (R)evolutions in Biopharmaceutical Analysis (KVCV)
Day & Time: Wednesday 24th January 2018, 16:30 - 16:50
Room: Ferrier Hall

In the last few years, highly efficient UHP-SEC columns packed with sub-3 µm particles have been commercialized by several providers.

Besides the particle size reduction, the dimensions of modern SEC columns (150 x 4.6 mm) were also reduced, compared to regular SEC columns (300 x 6 or 300 x 8 mm) enabling to reduce analysis time. However, the contribution of the chromatographic system itself to peak variance can become a serious problem under UHP-SEC conditions and need to be evaluated.

The inertness of SEC stationary phases is another important feature as slight secondary (non-specific) interactions can drastically affect the observed amount of protein aggregates. The possible undesired interactions can be controlled by the addition of various mobile phase additives (salts, buffers, organic modifiers). To support the growth of mass spectrometry (MS) in the biopharmaceutical field and disclose the unambiguous identification of the separated species, volatile mobile phases have been recently suggested in SEC-MS for the analysis of reduced, denatured and intact monoclonal antibodies (mAbs). However by using such mobile phases, compromises have to be find between chromatographic efficiency and MS sensitivity.

The presentation discusses modern SEC column technology, the impact of stationary phase inertness and possibilities to perform direct UHP-SEC-MS experiments. The SEC chromatographic behavior of several commercial mAbs and mAb-related products covering a wide range of physico-chemical properties (molecular weights between 54 and 153 kDa, pI values comprised between 6.1 and 9.4) are illustrated. Possible MS hyphenation and multi-dimensional separations are discussed in details. Problems related to extra-column volume and band broadening in modern SEC are also shown. Finally, various applications and method development approach are presented.

Novel ways to introduce the traditional salt based chromatography technique of Ion Exchange Chromatography of biopharmaceutical proteins into High Resolution Mass Spectrometry (Seminar)

Author: Florian Fussl, Thermo Fisher Scientific
Country: United Kingdom
Co-Authors: Jonathan Bones, Ken Cook

Session: Hyphenated Techniques for Comprehensive Analysis
Day & Time: Wednesday 24th January 2018, 11:00 - 11:20
Room: Lower Hall

Thorough characterisation of Bio-therapeutic proteins is essential at all stages of development through to manufacture and final product quality control. Monoclonal Antibodies [Mab] are the fastest growing class of these new drugs due to their high specificity to the targets they are used against. Each Mab will have several different variant forms due to multiple post translational modifications that can occur during production, purification and storage. These modifications can often alter the charge distribution on the surface of the protein and so can be characterised by charge variant analysis using ion exchange chromatography. All modification variants on the Mab require characterisation and control to ensure product quality and reproducibility as they could have an impact on efficacy or safety. The charged variant profile specific to each biopharmaceutical product is also used as an indication of similarity in Biosimilar analysis. Identification of structural variants is a critical challenge and Mass Spectrometry [MS] is used as an essential tool in the characterisation and identification of the protein variants. However, the techniques of ion exchange and size exclusion of proteins both require high salt eluents in the chromatography which is incompatible with MS so the structural variants exposed by these techniques must be collected separately off-line, then desalted before further characterisation by MS. Here we describe novel direct on-line coupling of ion exchange to the MS instrument in the characterisation of Mab variants. The technique has a fast run time and greatly reduces analysis time and sample handling by avoiding fraction collection and separate desalting injections by reverse phase LCMS. The chromatographic resolution of MAb charged variants using pH gradient elution with a novel volatile buffer preparation compares favourably with traditional salt elution. The proteins enter the Orbitrap MS system in the native state with a reduced charge distribution and an elevated mass to charge ratio. Variants found with this direct on-line coupling include glycosylation, deamidation, oxidation and lysine truncation. However the final analysis results will also give a more accurate intact mass and a charged variant profile, elevating this novel methodology to a true multi attribute method with no sample preparation necessary.

Replicates of Complex Mixtures in Ultra-High Resolution Mass Spectrometry Could Help Pave The Way to Big Data (Seminar)

Author: Remy Gavard, University of Warwick
Country: United Kingdom
Co-Authors: Mark Barrow, David Rossell, Simon Spencer

Session: Big Data, The Last Hyphenation
Day & Time: Thursday 25th January 2018, 11:00 - 11:20
Room: Ferrier Hall

By using Fourier transform ion cyclotron resonance mass spectrometry (FTICR MS), scientists are able to determine an unprecedented number of components in crude oil. The statistical tools required to analyse the mass spectra struggle to keep pace with advancing instrument capabilities and increasing quantities of data. Today, we are facing "fat data" as we have lots of attributes but no "tall data" as there is a limited amount of exploitable training samples. This is because most ultrahigh resolution analyses for complex mixture samples are based on single, labour-intensive, experiments. As a result, it can be challenging to monitor repeatability and differentiate between noise and true signals. Another factor contributing to the low number of training samples available is that the data analysis is usually performed once for the purpose of a specific investigation but may not be stored for later use. In order to be able to develop methods to exploit greater numbers of samples, we need to ensure the consistency, the reliability and the organisation of MS data.

We present a new algorithm developed in R, named Themis, to jointly pre-process replicate measurements of a complex sample. False positive peaks with low intensity can arise throughout a single mass spectrum due to the presence of noise. The locations of these peaks are not consistent between replicate samples, due to the randomness of the noise. Researchers are typically faced with a trade-off; it is important to set peak picking thresholds low enough to avoid omission of genuine peaks, but setting the threshold sufficiently low can result in large numbers of noise peaks being included too. By combining information across datasets, we determine and reduce false positive peaks with a smaller margin for error. This enables true peaks of low intensity to be extracted from the background noise and improves consistency as a preliminary step to assigning chemical compositions and data analysis. Through the use of peak alignment and an adaptive mixture-model-based strategy, it is possible to distinguish true peaks from noise and obtain more reliable datasets for further use.

We applied Themis to a variety of crude oils and naphthenic acid samples. These results demonstrated a more effective removal of noise-related peaks and the preservation and improvement of the chemical composition profile. Themis enabled the isolation of peaks that would have otherwise been discarded using traditional peak picking (based upon signal-tonoise ratio alone) for a single spectrum, and therefore Themis ensures the inclusion of information that would typically be lost, while also reducing data set sizes.

Themis affords greater success with the assignment of chemical compositions to lowintensity peaks using petroleomic software. In addition, improved monitoring of data quality and handling of replicate datasets will allow researchers to process larger numbers of samples with greater confidence. This, in turn, will enable larger scale data analysis methods, which inform decision making.

An Advanced Cyclic Ion Mobility - Mass Spectrometry System (Seminar)

Author: Kevin Giles, Waters Corporation
Country: United Kingdom
Co-Authors: Martin Green, Keith Richardson, Nick Tomczyk, Jakub Ujma, Jason Wildgoose

Session: Ion Mobility - Mass Spectrometry (BMSS)
Day & Time: Thursday 25th January 2018, 16:50 - 17:10
Room: Lower Hall

The powerful combination of ion mobility (IM) with mass spectrometry (MS) has generated a significant amount of interest from researchers operating over a broad range of subject areas. Whilst there is a notable focus on bringing this technology into more routine use, the need to further advance the capabilities of this hyphenated approach is ever present. In more recent years there has been considerable progress in the separative capability of sub-ambient pressure IM with resolutions in the many hundreds being demonstrated. One particular system is a travelling wave-enabled cyclic IM (cIM) separator embedded in a quadrupole time-of-flight (Q-ToF) MS instrument, which not only provides high resolution but also the capability for sequential IM separations. What will be presented here are the design and performance characteristics of a second generation Q-cIM-ToF system.

The base instrument comprising the second generation system is a Waters Synapt G2-Si (Q-IM-ToF) mass spectrometer. Compared to the first generation cIM system there have been enhancements to the ion optics, mass analyser, detection system and instrument control. The new cIM cell has the same 100 cm path length as the original design but is constructed of quadrants as opposed to a single element design, which makes it easier to manufacture. This new construct is found to perform as well as the previous version, with mobility resolution in excess of 500 being realised. Modifications to both the instrument control software and firmware for the multi-function array at the heart of the cIM have been undertaken. These enable excision and storage of segments of mobility separated ions to be carried out, followed by activation and re-analysis in the cIM device. As such, IMn analyses are possible. A quadrupole ion guide with axial field replaces the standard transfer cell and is used to transport ions from the cIM to the ToF; this both maintains the mobility separation and conditions the ion beam for high transmission into the ToF analyser. The ToF itself is of increased length compared to a standard instrument and has a novel offset V/W geometry. A maximum m/z resolution of around 120k has been obtained in the offset W-mode. A new, dual gain ADC detection system has also been implemented, providing up to 60x increase in dynamic range over the standard instrument and work is underway to provide increased mobility record length to account for the longer separation times in the cIM device.

The performance of this system will be highlighted with a range of samples from small molecules to proteins and protein complexes.

LC-MS small molecule quantitation: a short tutorial of best practice (Tutorial)

Author: Ruth Godfrey, Swansea University
Country: United Kingdom
Co-Authors:

Session: Challenges in Quantitative Analysis
Day & Time: Friday 26th January 2018, 12:40 - 13:10
Room: Room D

Advancement of liquid chromatography-mass spectrometry (LC-MS) instrumentation, operating systems and software has enabled small molecule quantitation by this technique to become part of routine practice. However, many challenges remain in ensuring reliable and accurate quantitation, particularly when analysing complex samples and using soft ionization approaches, such as electrospray ionization. The solution(s) to these challenges is sometimes subtle and not easily accessible, often resulting in them being overlooked by non-expert users. A role of the Analytical Methods Committee (AMC) of the Royal Society of Chemistry (RSC) is to engage with both expert and non-expert users, providing educational and guidance material to enhance and support understanding for using analytical techniques. This tutorial is inspired by guidance prepared on behalf of AMC designed to help inform and support the use of LC-MS for small molecule quantitation.

Screening of environmental passive sampling extracts using LC Q-TOF-MS in data-independent acquisition mode (Seminar)

Author: Anthony Gravell, Natural Resources Wales
Country: United Kingdom
Co-Authors: Graham Mills

Session: Screening Enviroment Pollutants, what can the data tell us?
Day & Time: Thursday 25th January 2018, 16:50 - 17:10
Room: Ferrier Hall

Two different approaches are currently used for compound identification in environmental screening analysis in combination with LC-MS. These are targeted MS/MS and data-dependent MS/MS. These two approaches have significant disadvantages, namely: targeted MS/MS operation performs MS/MS only on what is included in the target list and never on unknown or unexpected targets and with data-dependent MS/MS desired precursors can be missed where multiple adducts are formed in highly complex sample extracts.

We have employed an alternative methodology based on data-independent MS/MS which overcomes the disadvantages of the other two approaches. With the data-independent MS/MS technique, high resolution accurate mass data is acquired using different collision energies, formed of a low value and one or more higher energy values. Target and non-targeted compounds are fragmented without precursor selection in very fast sequential steps and accurate mass precursor and fragment data are recorded for all collision energies. The result of alternating the collision energy is a data file with a low energy channel that contains predominantly precursor ions and one or several high-energy channels that contain precursor and fragment ions. Pharmaceuticals are ubiquitous in surface waters because of continuous discharges from municipal wastewater treatment plants and we still do not know which pharmaceuticals (including those not currently monitored) are reaching the environment, the size of the problem for exposed fauna, nor what the effects, if any, of that exposure may be. A searchable accurate mass compound database containing all relevant ion species, and specific qualifier ions, for over 250 of the most commonly prescribed pharmaceuticals in Wales was developed for the screening of pharmaceuticals in extracts of polar passive samplers using an LC Q-TOF-MS system operating in data-independent mode. Chemcatcher® polar passive samplers were deployed for a period of almost four weeks at three sewage treatment works in west Wales.

Following retrieval and subsequent processing of the passive samplers the extracts obtained were screened using the newly developed data-independent MS/MS technique and a total of 79 pharmaceuticals were identified with high confidence using the compound's protonated or sodiated molecular ion. Fifty of the 79 pharmaceuticals were unequivocally confirmed by evaluating five of the most specific compound fragment ions from the MS/MS spectral library.

Excellent mass accuracy was obtained from the analysis with 75% and 85% of compounds identified with errors of less than 3 and 5 ppm respectively. Applying data-independent MS/MS methods resulted in a highly efficient workflow for the analysis of the passive sampling extracts. The data- independent MS/MS acquisition method is fast and the acquired data was later successfully interrogated for pharmaceutical metabolites that were outside the initial scope of analysis. The results obtained established the value of the approach developed and should prove invaluable for investigative monitoring purposes under the remit of the EU Water Framework Directive.

Thermal Analysis in hyphenation with mass spectrometry as a versatile tool for the analysis of complex and high boiling petroleum products (Keynote)

Author: Thomas Groeger, Helmholtz Zentrum München GmbH
Country: Germany
Co-Authors:

Session: Energy & the Environment
Day & Time: Friday 26th January 2018, 12:40 - 13:10
Room: Lower Hall

Thermal analysis (TA) enables the analysis of high boiling and residual matrices derived from petroleum. TA is primarily of interest if the matrix cannot be analysed by methods like gas-chromatography and direct-injection mass spectrometry due limits in temperature range or ionisation effects. TA techniques can generally be divided into setups working under a defined atmosphere at pressures ranging from sub-ambient to increased pressure like thermogravimetry (TG) and methods working under highly reduced pressure such as direct insertion probe (DIP). However, TA only provides temperature resolved fundamental physical data like weight loss or heat consumption. For a more detailed chemical analysis, an evolved gas analysis (EGA) could be utilized by hyphenation to, e.g. mass spectrometry.

In this respect, an appropriate ionisation technique is essential for an in-depth chemical description. Electron impact (EIMS) and Photoionization mass spectrometry (PIMS) are particularly well suited for Evolved Gas Analysis (EGA). Both techniques could be used for a wide range of compound classes ("universal ionization") and are not very prone to produce ionisation artefacts. In particular, PIMS allows the determination of the molecular signatures (i.e. intact organic molecules) derived from desorption-, pyrolysis- and combustion-processes. If EI is applied, a pre-separation step is indicated to reduce the complexity of the spectral information. Therefore, a newly developed, ultra-fast cycling gas chromatography technique was implemented. This fast cycling GC technique is based on a rapidly IR-radiation heat-able (and quickly cool-able) gas chromatograph which is inserted between the TA and EI/PIMS device. Various petrochemical samples have been analysed in detail with this approach. Exemplarily, in the case of crude oils, the yield and chemical composition of the different distillation fractions were specified (50-400°C). At higher temperatures, cracking products of the non-volatile residue (e.g. resins, asphaltenes) are fingerprinted. From very heavy matrices and source rock samples the amount and composition of the thermally extractable volatile organic compounds were determined.

Aside from fast chromatographic pre-separation high-resolution mass analyzes is beneficial for TA based petrochemical description. For this reason, DIP could be directly hyphenated to high-resolution TOFMS (Pegasus HRT, LECO, USA) equipped with EI as well as PI for rapid screening and comparison of crude oil- and bitumen-samples. The achieved resolution (R ~ 30.000 - > 50.000) allows a partly temperature resolved Kendrick mass defect-based analysis of compound classes of the highly complex evolved gas mixtures. On the high-end side ultra-high mass resolution mass spectrometry (7T FT-ICR, Bruker, Germany, (R > 200.000) can be applied for TA-EGA with atmospheric pressure chemical and photo ionisation sources (APCI and APPI). For a more in-depth structural insight, collision-induced dissociation (CID) for tandem mass analysis (MS/MS) was applied. CID-MS/MS with moderate dissociation energies allows the analysis of aromatic core structures, e.g. from asphaltenes. The value of this concept was demonstrated exemplary by the comprehensive chemical characterisation of heavy petroleum and its fractions. Summarizing, the TA-PIMS-, DIP-PI-high-resolution-TOFMS-, TA-fast-GC-PIMS- and TA-FTICR-results suggest a broad applicability of the TA-MS approach for the analysis of crudes and petrochemical fractions as well as the simulation and optimization of industrial petrochemical processes.

Characterization of complex polyether polyols using comprehensive two-dimensional liquid chromatography hyphenated with high resolution mass spectrometry (LCxLC-HRMS) (Seminar)

Author: Gino Groeneveld, University of Amsterdam
Country: Netherlands
Co-Authors: Melissa Dunkle, Andrea Gargano, Edwin Mes, Ayako de Niet, Matthias Pursch, Marian Rinken, Peter Schoenmakers

Session: Comprehensive Chromatography - The State of the Art
Day & Time: Wednesday 24th January 2018, 14:30 - 14:50
Room: Room D

Polyether polyols are key components in the production of polyurethane products, such as coatings, adhesives, and sealants, as well as in many other applications. Polyols are formed by reacting the starter (e.g. monol, diol, triol, etc.) with organic oxides (e.g. ethylene oxide (EO) or propylene oxide (PO)) in the presence of a base catalyst. The resulting polyether polyols can have a high degree of complexity due to differences in the starter and the functionality type distribution (FTD: functional group present), the molar-mass distribution (MMD: the chain length) and the chemical composition distribution (CCD: the EO-PO content). The characterization of such chemical features is of great importance, as they define the properties of the polyurethane intermediates and, hence, the performance of final products.

Such full characterization can be a challenging task as it can be difficult to fully resolve all the present distributions with a single analysis technique. Thus far, LC-MS is often used for the characterization of polyether polyols. Although this technique is capable of resolving a great deal of the sample, a chromatographic system with more resolving power is required to address the sample dimensionality of the polyether polyols. Comprehensive two-dimensional liquid chromatography (LCxLC) has proven to be the method of choice for very complex samples.

In this presentation, LCxLC methods hyphenated with high resolution mass spectrometry (LCxLC-HRMS) are described to characterize a wide variety of industrial polyether polyols. Different orthogonal separation mechanisms were studied to allow characterization on the starting compounds, as well as the MMD as function of the type of polymer (homo- and co-polymers).

From one to four comprehensive separation dimensions to characterize antibody drug conjugates (Seminar)

Author: Davy GUILLARME, University of Geneva
Country: Switzerland
Co-Authors: Balazs Bobaly, Szabolcs Fekete, Alexandre Goyon, Jean-Luc Veuthey

Session: (R)evolutions in Biopharmaceutical Analysis (KVCV)
Day & Time: Wednesday 24th January 2018, 11:00 - 11:20
Room: Ferrier Hall

The characterization of antibody-drug conjugates (ADCs), combining the specificity of a mAb with a potent cytotoxic agent covalently bound via a linker to the antibody, is a tremendous challenge to state-of-the-art analytical technologies. Indeed, subtle changes in these large (> 150 kDa) molecules can have profound effects on efficacy and pharmacokinetic properties.

The aim of this work was to highlight the possibility offered by hydrophobic interaction chromatography (HIC) for the characterization of cysteine linked ADC. In this context, brentuximab vedotin (Adcetris®) has been taken as a case study and various separation dimensions were employed to obtain the most relevant information from the ADC sample: One dimension separation: HIC was successfully employed to determine the average drug-to-antibody (DAR) ratio of brentuximab vedotin. However, care should be taken when selecting the mobile phase conditions (addition of organic solvent or not), and the gradient profile (linear or logarithmic) for elution of the DAR species. Three dimensions separation: A comprehensive 2D-LC approach involving HIC in the first dimension and RPLC in the second dimension was employed in combination with high resolution mass spectrometry (HRMS) for the structural identification of the species observed on the HIC trace. A one-month and two-months stressed samples of brentuximab vedotin were also evaluated using this approach. Four dimensions separation: To avoid the denaturation of cysteine linked ADC under RPLC conditions, a comprehensive 2D setup was employed, including HIC and SEC in the first and second dimensions, respectively. Then, ion mobility spectrometry (IMS) was activated prior to HRMS detection to gain additional information. This non-denaturing strategy allows obtaining easy-to-interpret data on ADC.

A tutorial on the fundamentals of hydrophilic interaction chromatography (HILIC) for pharmaceutical analysis (Tutorial)

Author: James Heaton, Pfizer Ltd
Country: United Kingdom
Co-Authors:

Session: Fundamentals in Separation Science (KVCV)
Day & Time: Wednesday 24th January 2018, 10:30 - 11:00
Room: Room D

HILIC can be a useful separation tool for the co-analysis of polar as well as ionisable pharmaceutical and related compounds. The HILIC modality can be used as an alternative or complementary technique to traditional reversed-phase chromatography (RPLC) approaches. HILIC can be a particularly suitable tool for the analysis of very polar compounds such as those with negative logP/logD values. As in RPLC, it is important to examine the composition of physiochemical properties and structural features of compounds in a given sample. In this tutorial we will explore the role of these features in directing HILIC method development work. Pertinently, the role of mobile phase buffer components and pH in conjunction with stationary phase chemistry will be discussed. Finally, best practice for successfully developing HILIC methods with relation to understanding compound structural features and diluent compatibility will be discussed.

Enhanced resolution of stereoisomers through Stationary phase optimized selectivity liquid and supercritical chromatography (SOS-LC and SOS-SFC) (Seminar)

Author: Ravindra Hegade, Ghent University
Country: Belgium
Co-Authors: Frederic Lynen

Session: Exploiting Separation Science
Day & Time: Thursday 25th January 2018, 16:50 - 17:10
Room: Room D

The development of method(s) for the analysis of chiral molecules and recognition of its enantiomeric purity is crucial as the enantiomers have the same physical properties but differ in pharmacokinetic and pharmacological behavior which leads to discrimination and separation is a very difficult issue. About 56% of the drugs currently in use are chiral compounds, and 88% of these are used as racemates. Quantitative analysis of the active pharmaceutical ingredient (API) and of the corresponding chiral and achiral drug impurities, requires the use of chiral stationary phases.1 When complex samples (increasingly containing several APIs) are at hand, the availability of single chiral columns allowing the resolution of individual enantiomers proves, however, often insufficient. In this context the concept of Stationary Phase Optimized Selectivity Liquid Chromatography (SOS-LC), a novel tool for the separation of solutes in a predictable way on combined stationary phases, is particularly promising of the separation of mixtures of chiral solutes.

This approach allows now both isocratic and gradient analysis2, while it also proves applicable on the compressible phases such as used in supercritical fluid chromatography.3 Thus far the potential of Stationary Phase Optimized Selectivity Liquid Chromatography for the separation and purification of stereoisomers has not been fully investigated, although especially in the latter case both chiral SOS-LC and SFC could offer significant benefits to speed up the purification process or to obtain improved chiral screening of complex mixtures. Therefore in this work the possibilities offered by chiral SOS-LC and SOS-SFC for the separation of mixtures of pharmaceutical enantiomers are explored, whereby emphasis is also set on the combination of chiral and achiral columns. Optimization and separation of enantiomers in the isocratic mode was done using standard commercially available chiral columns and with the classical isocratic SOS-LC algorithm. Gradient predictions are accomplished by in-house developed Visual Basic algorithm. The methodology offers new potential for faster analytical and preparative separation of optical isomers for various applications.

UHPSFC-MS of a Range of Steroidal Compounds (Tutorial)

Author: Julie Herniman, University of Southampton
Country: United Kingdom
Co-Authors: John Langley

Session: Green Separations
Day & Time: Thursday 25th January 2018, 14:50 - 15:10
Room: Lower Hall

Steroids are a category of lipid molecules that include cholesterol, steroid hormones and bile salts. Traditionally gas chromatography mass spectrometry (GC-MS) has been used for analysis but often requires derivatisation to increase the volatility of the samples. Reversed phase HPLC-MS has been used for the analysis of many steroid compounds, however, some are over-retained and/or separation of mixtures is difficult to achieve. More recently high performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) has been utilised due to its improved specificity and selectivity. Similar to the GC approaches, derivatisation of the steroid is often required to aid ionisation.

There are varied research interests in lipids at Southampton and the application of UHPSFC-MS continues to grow in this field, and has recently been extended to a different steroidal compound classes. A Waters Acquity UPC2, coupled to a TQD mass spectrometer and different organic make-up solvents are used to promote ionisation for a range of different ionisation techniques, namely ESI, APCI and APPI. Separation was developed for mixtures of steroid compounds, such as cholanic acids/esters and steroid derivatives using different column phases and different modifier solvent gradients. The methods used for standard compounds have subsequently been embedded into an open access environment to facilitate the analysis of archaeological materials, biofuels and to monitor chemical synthesis.

Interfacing LC to MS (Tutorial)

Author: Julie Herniman, University of Southampton
Country: United Kingdom
Co-Authors:

Session: Other
Day & Time: Wednesday 24th January 2018, 14:00 - 14:30
Room: Room D

Whilst interfacing of liquid chromatography to mass spectrometry is now commonplace there are a number of important factors that need to be considered when using modern LC-MS. If approaching LC-MS from a chromatography background then compatibility of buffers used for reversed phase LC must be considered prior to the use of mass spectrometry as the detector.

Coupling of normal phase chromatography to mass spectrometry is inherently flawed, e.g. due to lack of protic media for electrospray ionisation; SFC-MS will be discussed as a surrogate for normal phase chromatography coupled to mass spectrometry.

This tutorial will explore the different approaches required to enable successful transfer of LC methods to LC-MS methods and also focus on different atmospheric pressure ionisation sources and optimisation thereof.

Better Living Through (Flavor) Chemistry: Vacuum Ultraviolet Spectroscopy as a New Tool for GC Analysis of Terpenes in Flavors and Fragrances (Seminar)

Author: Alex Hodgson, VUV Analytics
Country: United States
Co-Authors:

Session: Advanced Analysis of Food and Beverages
Day & Time: Thursday 25th January 2018, 11:40 - 12:00
Room: Room D

Terpenes contribute greatly to our senses of smell and taste and thus are integral to industries like herb and spice producers, essential oil manufacturers, cannabis growers/distributors, breweries, and distilleries, among countless others. Current terpene analysis is done via GC-FID and GC-MS; however, since many terpenes of interest are isomers, baseline separation is required for accurate quantification, which leads to relatively long run times. Vacuum ultraviolet (VUV) spectroscopy can spectrally distinguish isomers and quantitatively deconvolve co-eluting peaks, allowing for significant chromatographic compression.

In this presentation we measure 21 mono- and sesquiterpenes with a sub 9-minute run time in a variety of samples, including essential oils, dry herbs and spices, hops, and gins. Samples are injected using both headspace and solid phase microextraction (SPME) techniques (split 2.5:1) onto a Restek Rxi-624Sil MS column at a flow rate of 4 mL/min helium. Qualitative results confirm the presence (or absence) of certain target compounds in samples, and a more comprehensive flavor/odor profile can be seen in the chromatographic "fingerprint" Quantitative data is also provided for the 21 terpenes in the various sample matrices.

Clinical Lipidomic Quantitation Based on Mass Spectrometry: Case Study of Pancreatic Cancer (Keynote)

Author: Michal Holcapek, University of Pardubice
Country: Czech Republic
Co-Authors: Eva Cifkova, Tereza Hrnciarova, Roman Hrstka, Robert Jirasko, Ladislav Kuchar, Miroslav Lisa, Denise Wolrab

Session: Big Data Chemometrics and Method Development(In-Silico)(KVCV)
Day & Time: Thursday 25th January 2018, 10:30 - 11:00
Room: Ferrier Hall

A wide range of lipid molecules is present in all eukaryotic cells. They fulfill numerous important physiological functions, and their dysregulation is often related to serious human diseases. The widely accepted classification of lipids according to Lipid MAPS is based on 8 main lipid categories, and each category has numerous classes and subclasses. Furthermore, large number of lipid species can be detected in biological samples for each subclass due to various fatty acyl carbon lengths, double bond number and positions. This complexity results in the fact that multiple analytical methods may be needed to cover the broader range of lipidome. Mass spectrometry (MS) and its coupling with the liquid-phase separation techniques is strongly dominating in the lipidomic quantitation. Essential steps in the lipidomic quantitation include the following: the careful optimization of analytical conditions, the selection of appropriate internal standards (IS) not occurring in studied biological samples (at least one IS per each lipid subclass), the full validation of final methods using the addition of internal standards into pooled samples, the use of quality control samples (typically pooled sample) to control possible variability among batches. Other important aspects are the (semi)automation of the workflow and high-throughput, but these issues should not affect the analytical robustness. In our analytical workflow, we use mainly the following three MS based methods for the high-throughput clinical analysis: 1/ direct infusion tandem MS using characteristic neutral loss (NL) and precursor ion (PI) scans on triple quadrupole type mass spectrometers (shotgun MS), 2/ ultrahigh-performance supercritical fluid chromatography - mass spectrometry (UHPSFC/MS), and 3/ matrix-assisted laser desorption/ionization (MALDI) coupled to high-resolution Orbitrap mass analyzer. Shotgun MS and UHPSFC/MS techniques are applied mainly for glycerophospholipids, sphingolipids, and glycerolipids using positive-ion electrospray ionization (ESI), while MALDI is used in the negative-ion mode to obtain complementary information on sulfatides and other anionic lipid subclasses. All mentioned methods follow the basic rule of reliable lipidomic quantitation that IS should be coionized with analytes from the same lipid subclass. Our laboratory-made software LipidQuant is used for the semi-automated data processing, and then the absolute or relative concentrations of all quantified lipids in individual samples are statistically evaluated using multivariate data analysis (MDA) methods, such as nonsupervised principal component analysis (PCA), supervised orthogonal partial least square discriminant analysis (OPLS-DA), S-plots, box plots, hierarchical clustering, neuron networks, etc. The application of our approach to the analysis of serum of pancreatic cancer patients and healthy volunteers will be shown, which will illustrate the potential for the future early diagnosis screening based on the lipidomic analysis.

Chemically Sensitive Online Detectors for SEC - current advances for SEC-MR-NMR and SEC-QCL-IR (Seminar)

Author: Johannes Hopfner, Karlsruhe Institute of Technology (KIT)
Country: Germany
Co-Authors: Manfred Wilhelm

Session: Comprehensive Chromatography - The State of the Art
Day & Time: Wednesday 24th January 2018, 16:30 - 16:50
Room: Room D

Polymers have three important molecular characteristics: the molecular weight distribution (MWD), the chemical composition and the topology. The MWD is usually determined using size exclusion chromatography (SEC). SEC detectors commonly in use, such as refractive index detectors, light scattering or viscometers, do not provide information about the chemistry or topology. This information is normally gained in separate experiments. Especially current spectroscopic methods such as IR and NMR are very powerful in in obtaining detailed insights. However, when it comes to analyzing complex materials like copolymers, blends or unknown samples, the correlated measurement of size and chemical properties is of special interest but tedious with dedicated but separate instruments.

Online coupling of IR or NMR spectroscopy with SEC is a promising approach to gain this correlated information. The inherent challenges of this approach are the low signal-to-noise ratios due to low concentrations after separation on a column as well as the strong solvent signals overlapping regions of interest in the analytes spectrum. Therefore, carefully optimization of the sensitivity and solvent signal reduction are most important.

The first approach is the coupling of a table-top medium resolution (MR) NMR spectrometer to a SEC system. The commercial spectrometer consists of a permanent magnet with a magnetic field strength of 1 T (62 MHz for 1H-NMR). The sensitivity is optimized via different flow probes, chromatographic conditions and signal treatment. The setup is run on non-deuterated solvent and thus the solvent suppression relies on selective pulse sequences and mathematical solvent signal reduction. The best resulting LOD is in the order of 100 _g/ml for PMMA and is sufficient for first applications.

In a second approach, our group previously reported on a SEC-FTIR coupling with a limit of detection (LOD) for the carbonyl group in PMMA as low as 30µg. To gain an even higher sensitivity, different infrared light sources are needed. We present results from a SEC coupled with an IR spectrometer using a tunable Quantum Cascade Laser (QCL) light source, which has a higher light intensity, but a limited bandwidth. In this application, the SEC-QCL-IR has the best sensitivity when operated in single wavelength mode. This makes SEC-QCL-IR ideal for investigating specific features of interest, such as an end-groups, functional groups or branching points. Therefore, SEC-QCL-IR measurements complement, but do not replace SEC-FTIR results. The LOD for PMMA could consequently be reduced by a factor of 3.8 to 8µg.

The method development for SEC-QCL-IR and MR-NMR-SEC including the general setup, the sensitivity enhancement, the solvent suppression and the influence of chromatographic conditions will be presented. Examples illustrating the benefits of such hyphenated techniques will be shown as well.

Maximising analytical data by understanding the implications of uncertainty (Tutorial)

Author: Chris Hopley, LGC
Country: United Kingdom
Co-Authors:

Session: Principles of Metrology & Data Handling
Day & Time: Thursday 25th January 2018, 14:00 - 14:30
Room: Room D

All analytical data produced has a degree of variability, also known as error and uncertainty. Uncertainty is just the expression of a range or confidence interval in an analytical result, it is often misunderstood and made overly complex, however, it takes many forms and at its most basic level is a useful tool in an analytical scientists armoury. By exploring uncertainty in any analytical method, reproducibility, robustness and overall quality of the data produced will be improved, as identifying and reducing uncertainty will naturally lead to this outcome.

This tutorial will discuss the importance of reviewing data, both qualitative and quantitative, and the associated uncertainty, to be able to maximise the value and quality of analytical data being produced.

Hyphenated Techniques for comprehensive analysis of metabolites in biological systems to describe metabolic changes caused by disease, environmental, nutritional, or genetic factors (Plenary)

Author: Tuulia Hyotylainen, Örebro University
Country: Sweden
Co-Authors:

Session: Advanced Analysis of Food and Beverages
Day & Time: Friday 26th January 2018, 09:00 - 09:50
Room:

A major health care challenge is to understand the relationships and interactions between genetic variations and various environmental triggers of disease. The proportion of chronic disease explained by genetic variation is ca. 10-20 % while the rest is explained by various environmental factors, such as diet, lifestyle, gut microbial activity and exposure to environmental contaminants. Currently, it has been suggested that the exposure to toxic environmental chemicals, for example, exposome plays an important role for e.g. in the development of type 2 diabetes, autoimmune diseases, obesity, and in neurological diseases. Furthermore, recent findings have implicated that toxic exposure together with e.g. unhealthy diet may have a strong synergic effect on the disease development and progression. In health research, it is highly challenging to study the interplay of these various factors and for that, metabolomics combined with exposure studies, by utilization of comprehensive chemical characterization for both metabolites as well as environmental pollutants, is a very promising tool.

In our studies, we have been investigating the factors and metabolic pathways in non-alcoholic fatty liver disease (NAFLD), diabetes as well as in autoimmune diseases. By combining studies and data on metabolomics in both blood-based samples and tissues, exposure data, clinical variables, gut microbiota composition, genetics and transcriptomics, intervention studies as well as results from animal models, we have been able to gain novel insights of the factors leading to NAFLD and linked with the development of insulin resistance. Although NAFLD commonly coexists with obesity, insulin resistance and type 2 diabetes, common genetic variants have also shown to be associated with NAFLD. Interestingly, however, steatosis in patatin-like phospholipase domain-containing 3 (PNPLA3) associated NAFLD is not accompanied by features of metabolic syndrome while the MBOAT7 variant rs641738 increases severity of non-alcoholic fatty liver disease in humans. By using comprehensive metabolic characterization, we have been able to identify circulating metabolic markers that can not only predict the NAFLD but also differentiate the patients with the metabolic and the genotypic NAFLD. The differences are associated particularly with changes in lipid metabolism. We have also, by using genome-scale study, showed that the disease is related to reduced metabolic adaptability. Moreover, our studies indicate that the type of diet has a significant impact on the disease progression, and that exposure to environmental pollutants is associated with both the development of insulin resistance as well as the non-alcoholic fatty liver disease.

On-line coupling of RPLC and chiral SFC for the analysis of pharmaceutical compounds (Seminar)

Author: Marion Iguiniz, Institut des Sciences Analytiques, UMR5280, University de Lyon
Country: France
Co-Authors: Estelle Corbel, Sabine Heinisch, Nicolas Roques

Session: Comprehensive Chromatography - The State of the Art
Day & Time: Thursday 25th January 2018, 16:30 - 16:50
Room: Room D

For pharmaceutical industry, quality is of prime importance and has to be managed from the early stages of drug the second enantiomer should be considered in the same manner as for other impurities. It is therefore necessary to develop, at least, two independent separation methods. A RPLC method is generally used to assess the achiral purity while a chiral method is used to evaluate the enantiomeric purity.

In the recent decades two-dimensional (2D) chromatography has emerged as a valuable tool for pharmaceuticals, biomedical research, and other fields. In particular, 2D-LC has been successfully applied to the achiral_chiral analysis of pharmaceuticals since the end of the 1980's. However, most part of chiral methods were developed in normal phase liquid chromatography and that is a problem for the implementation of a 2D-LC system. Indeed, the incompatibility of RPLC and NPLC mobile phases makes this approach very challenging. We therefore investigated SFC instead of NPLC as second chiral dimension for the RPLC x SFC separation of drugs. In addition to being a greener technique, SFC is more versatile than NPLC and leads to much faster analysis times, suitable for a second dimension in comprehensive 2D-LC.

This study was carried out with a pharmaceutical sample in the early stages of development, provided by Oril Industrie. First part of the study was dedicated to the development of the chiral SFC second dimension, the first dimension of the 2D system being the method used for quality control of the pharmaceutical sample. We studied the effect of injecting polar sample solvent into a supercritical mobile phase, as well as experimental and instrumental aspects related to the interface of the system. It was very interesting to notice that the use of water as co-solvent additive allowed to increase the injected volume in second dimension, without peak distortion issues. The operating conditions were then optimized with a view to implement a 2D system in selective comprehensive mode (sRPLCxSFC), using chiral SFC in the second one. Finally the developed sLCxSFC system was successfully applied to the achiral-chiral analysis of the pharmaceutical sample.

These results allowed us to obtain both chemical and enantiomeric purity of an active principle ingredient in one single analysis.

Separation science and its use for generating data, information and understanding (Keynote)

Author: Hans-Gerd Janssen, Unilever Research and University of Amsterdam
Country: Netherlands
Co-Authors: Boudewijn Hollebrands

Session: Other
Day & Time: Wednesday 24th January 2018, 10:30 - 11:00
Room: Lower Hall

Separation methods generate data, or numbers. Sometimes these numbers are the aim of the analysis, but very often they are just the start of generating information, or ultimately knowledge and understanding. In the last two decades we have significantly improved our ability to obtain more data, more reliable data, cheaper and faster. Techniques that enable us to do so include comprehensive chromatography, hyphenated sample preparation methods, high resolution accurate mass MS etc. Due to these improvements the generation of data is hence no longer the bottleneck. The problem now is getting all relevant information out of the enormous amounts of data we generated. New data treatment and processing methods are needed. But we should also approach our problems in a cleverer manner. Not just try to generate as much data as possible, but try to obtain structured data, group separations, data related to effects, etc. In the presentation new methods of generating more data will be discussed. These include comprehensive GCxGC, LCxGC, high resolution MS, MS/MS etc. But specifically we will also show that sometimes 'less is more'. Selective derivatisation strategies will be used to monitor specific classes of compounds only, normal phase LC will be shown to provide selectivity rather than just huge peak numbers, and finally some simple yet clever data processing methods will be demonstrated.

HTC-15

Can Gas Chromatography - Olfactometry Determine the Importance of Volatile Organic Chemical to Food and Beverage Odour? (Seminar)

Author: Lewis Jones, Sensient Flavors
Country: United Kingdom
Co-Authors: James Addison, Richard Haydock, Kathleen Pinfold

Session: Food/Drink
Day & Time: Friday 26th January 2018, 13:10 - 13:30
Room: Ferrier Hall

Gas Chromatography - Olfactometry (GC-O) has been established for over 30 years. GC-O is used for the bioactivity guided identification of odor active volatile organic compound (odorants). However, there is little information of the techniques validity. Within this work the validity of GC-O has been investigated showing: (i) that humans are very imprecise GC detectors; (ii) that the methods used with the GC-O technique to prioritise odorants, such as Aroma Extract Dilution Analysis (AEDA), are very poor predictors of an odorants importance to a food or beverage. It is therefore recommended that when using GC-O multiple analyses should be carried out for odorant identification, and when using GC-O for prioritisation of odorants, other measures need to be taken into account.

To validate humans as GC detectors the probability of peak detection for two odorants, hexanal and 3-methylbutanal, over a range of concentration were studied. Using a probit model to transform the data, an estimate of the mean concentration for a 95% probability of odor detection was calculated with 95% confidence limits. For both odorants this "limit of detection" was unexpectedly high (95 and 28.6 mg / L respectively) with imprecision (upper 95% confidence limit 921 and 110 mg / L, lower 95% confidence limit 38.9 and 15.6 mg / L respectively).

To investigate the GC-O technique's ability to prioritise odorants, a meta-analysis of a selection of publications were carried out. For the meta-analysis, GC-O results for each odorant were used to predict an odorants odour-activity-value (OAV). The OAV is the ratio of the concentration of an odorant in the food and its odour detection threshold in a suitable matrix. OAV is assumed to relate to an odorants overall importance in a food. Using a simple linear model, results from the prediction were very poor with root mean square deviation (RSME) 1.14. However, by using additional data, such as an odorants vapour pressure and air/water partition coefficient, with a machine learning model, it was found that prediction improved with RMSE 0.58. Interestingly, within the model the best single predictor of OAV was linear retention index on a standard polar column. This shows that a combination of analytical measures is actually better at predicting an odorants importance to food than results from labour intensive GC-O methodologies.

Advancing the analytical toolbox using shotgun lipidomics for lipid modifying enzymes (Seminar)

Author: Erwin Kaal, DSM
Country: Netherlands
Co-Authors: Raymond Ramakers, Emilie Usureau, Rob van der Hoeven

Session: Advanced Analysis of Food and Beverages
Day & Time: Wednesday 24th January 2018, 11:20 - 11:40
Room: Ferrier Hall

Lipids are playing a significant role in food textural properties, flavor and health. Therefore, lipids are an important compound class for nutritional products. One way to improve food products are the use of enzymes, such as lipases, to convert lipids. To understand the role and the functionality of those enzymes in food, it is necessary to obtain quantitative information of all lipids present, including Regio-isomers, before, during and after enzyme treatment of food products. From an analytical perspective, this is a challenging task because of the large diversity of lipid classes present in food, different concentration ranges and large number of samples to be analyzed. Nowadays, many different analytical tools, such as LC/MS, GC/MS and NMR, are needed to obtain the complete lipid mass balance in food products including converted lipids. With the present way of working, a good overview of the lipids can be obtained. Nevertheless, the present workflow is time consuming, gives relatively large analytical errors and some details on the lipids cannot be obtained. Therefore, it is necessary to improve the workflow to have more valuable information on lipids in food. In this study, shotgun lipidomics including hyphenated ion-mobility mass spectrometry is evaluated for determining the complete lipid mass profile in food products. A comparison is made between LC/MS and the newly developed ion mobility-based shotgun lipidomics approach. The similarities and differences in the range of lipids detected and the congruency of their relative abundances as detected by each analytical platform are discussed.

Urban water profiling to inform the state of the environment and public health (Keynote)

Author: Barbara Kasprzyk-Hordern, University of Bath
Country: United Kingdom
Co-Authors:

Session: Screening Enviroment Pollutants, what can the data tell us?
Day & Time: Thursday 25th January 2018, 16:00 - 16:30
Room: Ferrier Hall

A new approach in public health epidemiology utilizing urban water fingerprinting with hyphenated mass spectrometry techniques has been recently pioneered to provide near real-time measurements of public health. Urban water fingerprinting provides anonymised but comprehensive and objective information on the health status of a population and surrounding environment in real time as urban water (sewerage system and receiving aqueous environment) pools the endo- and exogenous biomarkers of that population.

This cutting-edge approach of extracting epidemiological information from urban water emerged from Wastewater-Based Epidemiology (WBE). WBE was developed in a strong cross-sectoral and transdisciplinary collaborative ethos within SCORE (www.score-cost.eu) and SEWPROF teams (www.sewprof-itn.eu), and although still in its infancy, WBE is currently used to report on community-wide illicit drug use trends and feeds into the Europe-wide evidence based early warning system by European Agency for Drugs and Drug Addiction (http://www.emcdda.europa.eu/wastewater-analysis).

This talk will introduce the concept and its rapid advances. It will focus on pharmacologically active compounds in urban water and their stereochemistry in the context of environmental risk assessment. It will also explore new avenues in the utilization of urban water fingerprinting in the assessment of population health and health risk prediction.

Looking Inside the Black Box of Machine Learning Methods: Applications in Analytical Chemistry (Seminar)

Author: Phil Kay, JMP, SAS Institute
Country: United Kingdom
Co-Authors:

Session: Big Data Chemometrics and Method development (In-Silico) (KVCV)
Day & Time: Thursday 25th January 2018, 11:40 - 12:00
Room: Ferrier Hall

Developing robust and accurate analytical methods relies on collecting the best data and extracting maximum insight. The volume, diversity and complexity of data in analytical chemistry is increasing all the time. This means that analytical chemists often need the skills and tools of a data scientist to efficiently and effectively deal with these challenges. There is much promise around Machine Learning methods, in particular. However, many of the methods and their results can be appear to be somewhat of a black box.

In this presentation we will show the insight and efficiency that can be gained from applying modern data analytics to analytical chemistry data. You will also gain a greater understanding of the mechanisms behind methods including Neural Nets, Clustering and Decision Trees. And how you can make sense of the options to find the most useful solution for your analytical problem by visually interacting with the data and the models.

Combining high-capacity sorptive extraction with Thermal desorption pre-concentration for analysis of (S)VOCs in environmental samples (Seminar)

Author: Lara Kelly, Markes International Ltd
Country: United Kingdom
Co-Authors: Ilaria Ferrante, Massimo Santoro

Session: Other
Day & Time: Thursday 25th January 2018, 17:10 - 17:30
Room: Ferrier Hall

Thermal desorption (TD) combined with GC(MS) has long been used as a tool in the sampling and pre-concentration of (S)VOCs in vapour phase samples. Traditionally, TD has been employed extensively for environmental and workplace air monitoring, with other applications in materials testing, food profiling and forensic type applications becoming increasingly common. However, the ability to deal with liquid sampling as historically been somewhat limited to many users of TD technology. New developments in sampling technologies, including high-capacity sorptive extraction, has extended the applicability of TD to liquid and solid samples, with the capacity for both immersive and headspace sampling allowing the extraction of components from within the sample prior to pre-concentration and analysis.

When used in conjunction with Thermal desorption, high-capacity sorptive extraction offers a number of well-known advantages over traditional solvent-extraction methods for a wide range of VOCs and SVOCs, including greatly improved sensitivity due to the avoidance of dilution, high extraction efficiency, and efficient transfer/injection into the GC. Furthermore, high-capacity sorptive extraction offers an extension to SPE & SPME methods which is simple to employ and, offers a versatile, robust method for gaining complementary information to that contained using other sampling approaches.

This multi-facted sampling approach has been applied to a number of differerent sample types, including environmental matrices, foods, beverages and clinical samples, examples of which will be presented. The benefits of using TD sample introduction extend to the ability to re-collect sample for repeat analysis, assisting with method validation and eliminating the need to perform repeat extractions on limited sample quantities.

Apolipoproteins, non-polar lipids, polar lipids and lipoprotein particle numbers. Can we measure them all on large number of samples? (Seminar)

Author: Zsuzsanna Kuklenyik, Centers for Disease Control and Prevention
Country: United States
Co-Authors: John Barr, Toth Christopher, Michael Gardner, Feffery Jones, Bryan Parks

Session: Clinical Hyphenations
Day & Time: Wednesday 24th January 2018, 14:50 - 15:10
Room: Ferrier Hall

High and low density lipoproteins (HDL and LDL) are complex molecular assemblies that are key participants in extracellular lipid metabolism with important consequences in the formation of atherosclerotic lesions and the development of cardiovascular disease. A lipid/protein composition based conceptual framework of lipoproteins and the clinical measurement of an extended panel of lipids and proteins has been advocated for many years, however the development of high throughput multiplexed mass spectrometry (MS) based analytical techniques allowed effective lipoprotein composition analysis to become a reality only recently.

In this study we developed two simplified, high throughput "one-pot" extraction protocols in well-plate format, for polar lipids and non-polar lipids, and implemented an automated on-line protein digestion platform. These three high throughput sample preparation methods were coupled with liquid chromatography (UPLC) and multiplexed tandem mass spectrometry (MS/MS) detections, and quantified non-polar lipids (FC, CE, TG), polar lipids (PC, SM, LPC, PE and PI) and a panel of apolipoproteins (apos A-I, A-II, A-IV, B-100, C-I, C-II, C-III and E). We also applied a preparative size fractionation technique, asymmetric flow field-flow fractionation (AF4) to separate lipoproteins in 1-2 nm size increments, measuring hydrodynamic size by dynamic light scattering. Lipids and proteins were measured in each size fraction and in the whole serum, using a total of 0.1 ml of each serum sample (656 measurements per sample).

We demonstrate the applicability of this workflow to the analysis of 125 serum samples, from individuals with normal lipid profiles, hyperlipidemia, hypercholesterolemia, and hypertriglyceridemia. The hyphenation of methods provided an extensive amount of quantitative information for varying sizes of HDL and LDL (8 to 36 nm). The quantitative measurement of all major lipoprotein constituents and particle size allowed volumetric calculation of particle numbers and composition in number of molecules per particle. The comprehensive composition data provided clear evidence of sub-HDL and sub-LDL particle populations with distinctly different composition that can be interpreted by lipid-lipid and lipid-protein interactions. Statistical analysis of molar ratios, protein/protein (apoE/apoC-III, apoC-II/apoC-III, and apoA-I/apoA-II) and lipid/lipid (FC/PC, SM/PC, PE/PC, and PI/PC) allowed differentiation of dyslipidemic versus normolipidemic subjects. These differences can be linked to underlying irregularities in HDL and LDL particle functions, and clinically relevant metabolic conditions that causes accumulation of atherogenic lipoprotein particles.

For the amount of clinically relevant information, our hyphenation of multiplexed, high throughput LC-MS/MS methods is highly cost effective, especially for analysis of typically limited volumes of archived samples. In the past with traditional single-analyte approaches, elucidation of such depth of quantitative information required substantial amount of sample volume and combinations of sequential ultracentrifugation, immunofractionation, cross-linking, and gradient gel electrophoresis techniques. Our results demonstrate how combination of high throughput sample preparation and multiplexed LC-MS/MS based methods can reveal compositional characteristics of lipoprotein particles and underlying irregularities in lipoprotein functions in lipid metabolism pathways that lead to cardiovascular disease. The ability of these comprehensive measurements to predict risk of coronary artery disease will be the subject of planned future investigations.

Lipidomics by UHPL-CQTOF-MS/MS with Data-Independent Acquisition and Clinical Applications (Seminar)

Author: Michael Laemmerhofer, University of Tuebingen
Country: Germany
Co-Authors:

Session: Advances in Clinical Analysis
Day & Time: Wednesday 24th January 2018, 11:40 - 12:00
Room: Ferrier Hall

Lipidomics approaches are nowadays widely adopted to have a comprehensive view on lipid profiles in biological samples. Alterations in lipid profiles may provide a panel of biomarkers for diagnostic and prognostic purposes of various diseases. Untargeted lipidomics workflows may generate new hypothesis for support biological interpretation or complement other omics data. Targeted lipidomics approaches are often utilized to validate biomarkers found in a discovery phase or measure particular lipids associated with a certain disease or present in low abundance (e.g. lipid mediators in inflammation). In untargeted lipidomics, UHPLC-MS/MS with data-dependent acquisition (DDA) using Orbitrap or QTOF instruments is state of art. In DDA, subsequent to an MS full scan (survey scan) a certain number of MS/MS experiments are performed in which the most intensive precursor ions from the survey scan are selected for fragmentation to obtain MS/MS spectra for identification. This acquisition mode has some disadvantages including limited reproducibility of MS/MS experiments and incomplete coverage by MS/MS data. For this reason, we investigate the performance and advantages of UHPLC-ESI-QTOF-MS/MS with data independent acquisition (DIA) workflows using SWATH (sequential window acquisition of all theoretical fragment ion mass spectra). In SWATH, precursor ions for simultaneous fragmentation are selected with intermediate Q1 mass window width (e.g. typically 10-30 u) and the generated fragments readout by TOF analysis creating composite MS/MS spectra for each SWATH window. This leads to comprehensive MS and MS/MS data so that XICs can be generated both from precursors and fragments. This allows to select the most suitable signal (precursor or fragment ions) that shows the best sensitivity or better selectivity for generation of XICs and data processing, respectively. MS/MS spectra are available for identification over the entire chromatogram and all samples improving lipid coverage and identification percentage. On the other hand, DIA with SWATH can also provide better performance for quantitative analysis. In targeted lipidomics, triple-quadrupole instruments are the method of choice an data acquisition is done by SRM. This provides a good quantitation performance, yet only preselected targets are detected. On the other hand, if SWATH is used for quantitative analysis, besides the targets, the sensitivity and assay specificity of which can be improved by using narrow precursor isolation windows, also other lipids are simultaneously detected in the FULL MS scan and MS/MS data of other lipids are available from the other SWATH windows. This allows combination of targeted analysis with similar performance as QqQ with untargeted profiling. Possibilities, advantages and challenges of DIA with SWATH in untargeted and targeted lipidomics workflows will be discussed on clinical examples. In one study, lipid extracts of platelets of patients with stable angina pectoris and acute coronary syndrome have been profiled against controls and revealed significant alterations in lipid profiles of platelets which correlated with disease severity to some extent. In another study, the advantages of SWATH acquisition for targeted analysis of steroid hormones in plasma samples of patients treated with steroid hormone patches will be discussed.

Supercritical Fluid Chromatography - Mass Spectrometry: Robust, Reliable and Required (Keynote)

Author: John Langley, University of Southampton
Country: United Kingdom
Co-Authors:

Session: Exploiting Separation Science
Day & Time: Wednesday 24th January 2018, 16:00 - 16:30
Room: Ferrier Hall

SFC now delivers on the chromatographic promises with robust and reliable instrument platforms. The solvation power, selectivity and peak capacity of modern Ultrahigh Performance Supercritical Fluid Chromatography (UHPSFC) coupled with mass spectrometry affords the ideal analytical platform to address classes of compounds that were previously a challenge for GC-MS and RP-UHPLC-MS approaches.

UHPSFC, in the form of Ultra Performance Convergence Chromatography (UPC2) has been integral to Southampton Chemistry open access (walk-up and use) chromatography mass spectrometry facility since 2013 where it complements existing liquid chromatography and gas chromatography options (HPLC-MS (ESI/APCI), GC-MS (EI/CI) to afford a comprehensive, modern and fast separation science and mass spectrometry option to address the wide range of diverse and challenging chemistries.

The extended capability and complementarity of SFC delivers unique solutions across a broad range of application areas, e.g. synthetic organics, pharmaceuticals, petrochemistry, lipids, nucleotides and its impact in many other research areas will be discussed.

Ion mobility mass spectrometry - leveraging rich data on the gas-phase ion to separate and assign (Keynote)

Author: Cris Lapthorn, GSK
Country: United Kingdom
Co-Authors:

Session: Ion Mobility - Mass Spectrometry (BMSS)
Day & Time: Thursday 25th January 2018, 16:00 - 16:30
Room: Lower Hall

Ion mobility mass spectrometry is a separation technique that hyphenates to mass spectrometry and can separate, assist in assignment, and reveal properties of ions on a complementary timescale. Ions are separated based on their differential mobility based on factors including size, shape and charge distribution. Ion mobility has proven useful as a tool to separate components in complex mixtures and to differentiate ions where mass spectrometry alone proved challenging, and a deeper understanding of physicochemical properties that affect ion mobility is developing. From ion mobility drift-times collision cross-sections can either be calculated or derived based on calibration routines. Measuring collision cross-sections and recorded them in commercial and public databases is becoming commonplace with vendor offerings, public online databases and publications. Predicting collision cross-sections using molecular modelling and/or chemometrics can identify components that are not recorded in databases. .

Exciting developments in ion mobility include increasing complementarity of molecular modelling approaches focussed on modelling ensembles of molecular geometries and charge distributions with closer approximations to the true state of ions in the gas-phase under different ion mobility regimes e.g. linear vs. non-linear fields. Orthogonal activation and interrogative techniques such as UV photodissociation enable understanding of corresponding relative stabilities and collection of spectroscopic data useful to refine singular assignments and wider global prediction models.

The flexibility of ion mobility has allowed researchers to develop, and users to capitalise on, a variety of different ion mobility separation techniques but crucially the differences in their performance is a challenge to grapple with, and to understand the limitations and advantages. A combination of ion mobility, mass spectrometry, molecular modelling and orthogonal measurements offers an unprecedented opportunity to collect data on gas-phase ions and utilise these data to separate, and assign structures to, ions.

Fast, efficient and selective: separation science for modern organic synthetic chemistry (Seminar)

Author: Tomas Leek, AstraZeneca
Country: Sweden
Co-Authors: Werngard Czechtizky, Peter Sjo, Johan Soderlund

Session: High Throughput versus High Efficiency Separations (CS)
Day & Time: Wednesday 24th January 2018, 16:50 - 17:10
Room: Lower Hall

Within modern drug discovery there is a growing interest to explore compound classes that do not necessary fall within the traditional property space for orally absorbable drugs.

The extended property space and the increased molecular complexity presents additional challenges related to drug discovery including the demands on development of innovative drug formulations and delivery systems for non-oral routes of administration. From a synthetic chemistry perspective each of the individual compound classes possesses a significant challenge regarding synthesis, analysis and purification preceding biological testing.

Although the workhorse for small molecule separations has been reversed-phase (RP) separations with MS detection, a plethora of methods and separation techniques are required to cover the need of compound analysis in the extended property space. Further, the need of both high throughput and high efficiency separations are evident to meet the demands on analytical services during different phases of drug discovery.

With the recent development of analytical Supercritical Fluid Chromatography (SFC) a new route to fast and efficient analysis has emerged. Based on comprehensive method development, we now have a SFC methods designed to meet the analytical needs of a drug discovery project also beyond the traditional drug property space. The SFC methods are both providing orthogonal selectivity towards reversed phase separations and an extend application range. This utility leads to the possibility of replace reversed-phase, hydrophilic interaction chromatography (HILIC) and even some ion-pairing chromatography (IPC) LC systems with a single SFC/MS system.

This presentation will highlight the impact of not only fast and/or highly efficient separations but also the value of utilising orthogonal separation methods. From a performance perspective, the utilisation of sub-2 µ stationary phases and ultrahigh performance instrument in both RP and SFC gives the flexibility to utilise rapid and/or high efficiency separations. The impact of separation science related to organic synthetic chemistry will be illustrated with examples from a) high throughput examples (suitable for monitoring) and b) examples of closely related compounds where a combination of high efficiency and orthogonal selectivity is required for comprehensive chemical analysis prior to biological testing.

Chromatographic strategies combining RPLC, mixed-mode HPLC and SFC coupled to MS for impurity profiling of drugs candidates (Seminar)

Author: Elise Lemasson, Institut de Chimie Organique et Analytique (ICOA)
Country: France
Co-Authors: Sophie Bertin, Philippe Hennig, Eric Lesellier, Caroline West

Session: Fundamentals in Separation Science (KVCV)
Day & Time: Wednesday 24th January 2018, 17:10 - 17:30
Room: Room D

Impurity profiling of drug candidates is a significant concern of pharmaceutical industries. The identification and quantification of impurities must be strictly controlled to ensure the efficacy and limited toxicity of the active ingredient. It is therefore necessary to have efficient analytical methods to ensure that all impurities are identified. Today, reversed-phase HPLC with C18 stationary phase with UV and MS detection remains the method of choice for impurity profiling of drug candidates. However, this method sometimes fails, particularly when the active pharmaceutical ingredient is not sufficiently retained on the column. It is essential to turn to alternative and complementary analytical methods.

This work deals with the development of alternative analytical methods to reversed-phase HPLC on C18 phase for impurity profiling of pharmaceuticals. Reversed-phase HPLC on other stationary phases (e.g. pentafluorophenyl phases), mixed-mode HPLC (combining reversed-phase and ion-exchange mechanisms) as well as SFC were explored. The chromatographic and detection performances of each technique to analyze pharmaceutical compounds was evaluated with a set of 140 pharmaceutical compounds from Servier Research laboratories. These performances were compared with those of reversed-phase HPLC on a conventional C18 stationary phase. The comparison and the study of the different methods allowed proposing an overall strategy of analysis for any new active ingredient.

Automated sample preparation: the missing hyphen to hypernation (Seminar)

Author: Camilla Liscio, Anatune
Country: United Kingdom
Co-Authors:

Session: Fundamentals in Separation Science & Sample Prep
Day & Time: Friday 26th January 2018, 11:00 - 11:20
Room: Lower Hall

In 1980 the term “hyphenation” was firstly coined by Hirschfeld to denote the on-line combination of a chromatographic separation and one or more spectroscopic/spectrometric detection techniques. The marriage, to exploit the advantages of both, was driven by the constant need within the analytical community to push the boundaries of selectivity and sensitivity to tackle the continuously more challenging and demanding analytical applications.

Nearly 40 years down the line, hyphenated analytical techniques are now the favoured approach for complex qualitative and quantitative analytical problems. With hyphenated techniques such as GC-MS and LC-MS well-established techniques of choice, special attention is now devoted to systems in which multiple hyphenation, also known as hypernation, is an integral part of the whole setup. It’s within the perspective of hypernation that the on-line automation of sample preparation finds its perfect scope.

Indeed, sample preparation is an essential part of any analytical workflow and despite the excellent performances of the latest available hyphenated techniques, good quality data for complex matrices can only be achieved when counting on a robust and reproducible sample preparation. Nevertheless, the appeal of automated sample prep doesn’t lie only in very good method robustness and batch-to-batch reproducibility. The extremely accurate flow control (down to 0.1µL/s) in liquid handling and the ability to control timing accurately (e.g. incubation time for derivatisation purposes) open the doors to what could be considered “high performance” sample preparation.

SmartSPE is an emblematic example of the power of automation for hypernation. The automation of online SPE using ITSP (Instrument Top Sample Preparation) single use miniaturised cartridges allows precise flow control and the ability to achieve SPE chromatographic performance which is not accessible with a manual method. In fact, in contrast to manual larger volume SPE, smart SPE flow profiles follow the expected Van Deemter curves with clearly defined optima. As an outcome of the accurate flow control, absolute recoveries of >99% can be achieved along with a significant reduction in background matrix.

This talk will set the background for the hypernation of automated sample preparation to both GC-MS and LC-MS. It will encompass some relevant qualitative and quantitative applications developed in the Anatune demo lab where the hypernation of automated sample preparation excelled in the delivery of high quality analytical data.

Can CE-MS improve the detection of peptides and intact proteins and in biological samples? (Seminar)

Author: Stephen Lock, Sciex
Country: United Kingdom
Co-Authors:

Session: Challenges in Quantitative Analysis
Day & Time: Thursday 25th January 2018, 14:30 - 14:50
Room: Lower Hall

Capillary electrophoresis (CE) is an orthogonal technique to LC separating analytes based on their charge, and lends itself well to the analysis of peptides and proteins. The properties of CE enable the reduction and often elimination of carryover and wall absorption which effects peak resolution and sensitivity, as the CE capillary is not only the separation channel but also the autos-ampler and with no connections can be cleaned easily between analyses.

CE-MS especially techniques such as CESI (the integration of capillary electrophoresis (CE) and electrospray ionization (ESI) into a single process in a single device) are now enabling the easy connection of CE to a variety of mass spectrometers.

Some biologically important neuropeptides such as Vasoactive intestinal peptide (VIP) and Pituitary adenylate cyclase-activating polypeptide (PACAP) are very basic and are difficult to analyze by LC-MS methods as they bind to columns and have very poor chromatographic properties. In this work we will describe how a CESI-MS method was developed to detect intact neuropeptides of the size of PACAP (Molecular Weight of 4534.26 amu) and VIP in biological samples overcoming some of these challenges. The sensitivities from CESI-were compared with those from a high flow LC-MS method. We will discuss how sample preparation methods were developed to better enhance results after which the CE-MS method was tested on spiked samples to demonstrate how CE-MS can be used in bioanalysis. Finally we will discuss how CE-MS can then be used to quantitate even larger proteins.

High temperature chromatography: the winning solution allowing both throughput and efficiency? (Keynote)

Author: Frederic Lynen, Ghent University
Country: Belgium
Co-Authors:

Session: Other
Day & Time: Wednesday 24th January 2018, 16:00 - 16:30
Room: Lower Hall

Physical separation processes requiring the migration of solutes in liquids are inherently slower compared to methods taking place in a low viscosity medium like a gas. The fundamental performance of chromatographic techniques can therefore be increased by reducing diffusional distances, as is done in UHPLC, or by speeding up diffusion processes, as is done in SFC and in elevated temperature HPLC. The latter approaches also allow for the implementation of greener and more cost-effective chromatography. The various possibilities offered by exploiting the benefits of elevated, high and extreme temperatures in liquid chromatography are discussed in this presentation. In the range of elevated temperatures, temperature responsive liquid chromatography (TRLC), whereby columns are packed with particles on which polymers with tunable hydrophobicity are anchored, show significant promise. Such columns allow purely aqueous reversed phase HPLC, whereby retention is controlled through temperature within a mild range of room temperature to 60°C. The possibilities, drawbacks and recent incremental improvements of the technique are compared. Various solutions are thereby explored in order to allow higher throughput and higher peak capacities through TRLC. Alternatively the possibilities offered by the coupling of conventional reversed phase and HILIC columns at higher temperatures (˜60-80°C) are demonstrated. This includes the ability to generate much higher efficiencies (N=100.000) compared to conventional room temperature approaches within conventional analysis times. The potential and challenges presented by novel more hydrothermally stable organo-silica based materials is subsequently demonstrated through fast, high temperature applications allowing operation up to 150°C. Finally extremely stable hydrogenated synthetic micro-dispersed sintered detonation diamond particles are evaluated up to and above 200°C under reversed phase HPLC conditions up to 400°C and 1000 bar and under SFC conditions using only water as mobile phase; this to study the chromatographic behavior and potential under such harsh conditions allowing fast analyte diffusion.

Fast and efficient group-type analysis of hydrocarbons by GCxGC (Seminar)

Author: Laura McGregor, SepSolve Analytical
Country: United Kingdom
Co-Authors: Nick Bukowski, Matthew Edwards, Steve Smith

Session: Comprehensive Chromatography - The State of the Art
Day & Time: Friday 26th January 2018, 13:30 - 13:50
Room: Lower Hall

Precise characterization of petroleum-derived fuels is important for the oil industry and environmental monitoring alike. However, it is a tedious and difficult task to identify each of the thousands of individual components present in these complex samples.

Group-type analysis using comprehensive two-dimensional gas chromatography (GCxGC) offers significant advantages over conventional chromatography for such analyses, with its vastly expanded separation space and the added benefit of highly structured groupings of compounds for simple classification of hydrocarbons.

Despite this enhanced separation of GCxGC, overlap between classes may remain, making it imperative to be able to identify the class boundaries accurately. The use of parallel detection by mass spectrometry (MS) and flame ionisation detection (FID) enables confident classification and quantitation of hydrocarbon groupings respectively. Using a combination of EICs and filtering scripts, the MS data can be used to create accurate stencil regions which can then be applied to the FID data.

Here we demonstrate the use of reverse fill/flush flow modulation for robust, repeatable and affordable GCxGC, combined with simple, yet effective, data processing workflows for group-type analysis. A number of real-world samples will be explored, including quantitation of total petroleum hydrocarbons (TPH) in environmental soil samples.

Flow Modulated Two-dimensional Gas Chromatography Coupled to Tandem Mass Spectrometry for "Comprehensive" characterization of Complex Samples (Keynote)

Author: Luigi Mondello, University of Messina
Country: Italy
Co-Authors:

Session: Comprehensive Chromatography - The State of the Art
Day & Time: Friday 26th January 2018, 10:30 - 11:00
Room: Ferrier Hall

The search for more powerful and discriminating analytical techniques has grown exponentially over the last decade, to address the high complexity of many natural and synthetic samples, and the increased demands for specificity of information required. To achieve the desired level of accuracy and reliability of analytical data, the analysis of complex matrices requires the combination of both powerful separation techniques and sensitive detection.

A number of advantages are to be gained by coupling two-dimensional comprehensive chromatography (GCxGC) to Mass Spectrometry (MS): handle complex samples, reduce matrix complexity, detect trace or ultra-trace compounds, increase confidence in structure assignment and quantitative results. The hyphenation to triple quadrupole MS affords extremely high-resolution, as well as targeted and untargeted analysis; in the multiple reaction monitoring (MRM) mode selectivity is enhanced, sample consumption reduced, and also the need for tedious clean-up procedures.

The advantages of using a flow-modulation device in GCxGC will be also illustrated over the cryogenic one, consisting in the compatibility with virtually any detectors and flow rates, reduced costs and environmental risks.

A series of flow-modulated GCxGC-MS applications will be shown, relative to a variety of complex real-world samples.

Why Do We Still Use Silica (Plenary)

Author: Peter Myers, University of Liverpool
Country: United Kingdom
Co-Authors:

Session: Other
Day & Time: Wednesday 24th January 2018, 09:00 - 09:50
Room:

Silica was used in the first commercial LC columns dating back to the 1970's and today silica is still the most widely used support for HPLC and UPLC. So, after nearly 50 years why are we still using silica?

Is it that good there has been and still isn't a need for a replacement?

In this lecture I will describe the problems we had with silica in the 1980's and how very little progress has been made to date. In the 1980's we had 5micron spherical totally porous particles. Today we have 1.5micron particles. Is this a real development?

I will describe how bonded phase chemistries have had to develop to help conceal silica problems. In the 1980's the most popular phase was octadecyl. Today the most popular phase is still octadecyl. Is it that good?

Compare this to the computer revolution. In the 1980's we had Apple, Commodore, Atari, BBC Micro TRS-80, ZX81, ZX Spectrum, Commodore 128, and the Amstrad. Today we have i-pads, computers surfaces and smartphones all of which have hundreds or even thousands of times more processing power than the computers of the 1980's. Has chromatography moved on that fast?

But the main focus of the lecture will be to look into the future and offer some new alternatives to silica and also offer some new separation methods that do not rely on the classical theory of adsorption or partitioning and certainly do not rely on packed silica columns.

Fingerprint characterization of bio-oil from the pyrolysis of African palm and its esterification product (Seminar)

Author: Diana Palacio, University of Warwick
Country: United Kingdom
Co-Authors: Mark Barrow, Enrique Mejia-Ospino, Claudia Ramirez, Josear Sarmiento Chaparro

Session: Energy & the Environment
Day & Time: Friday 26th January 2018, 11:00 - 11:20
Room: Room D

Bio-oils are produced from renewable sources, such as biomass. The chemical composition of the bio-oil is an important feature for the optimization of the feedstock transformation processes. Pyrolysis of the biomass (thermal decomposition in the absence of oxygen) produces solid materials (chars), liquid (pyrolysis oil or bio-oil) and gas. The high oxygen content, poor volatility, high viscosity and corrosiveness of the resulting bio-oil obtained from pyrolysis limit its use as a fuel. For bio-oil to be refined into usable transportation fuel, post-production upgrading is required. Esterification is an important upgrading technique where and acid-catalysed reaction with excess of alcohol is used to reduce the acid content of the bio-oils. Bio-oils contain chemical compounds with varying volatility and polarity. Gas chromatography-mass spectrometry (GC-MS) is a useful technique that allows to identify lightweight compounds in bio-oil with high volatility and low polarity. Thus, polar organic compounds are difficult to analyse with GC-MS. To overcome this, highly polar compounds can be characterised by electrospray coupled to Fourier transform ion cyclotron resonance mass spectrometry (FT-ICR MS). This ultrahigh resolution mass spectrometry technique, affords the characterization of the samples including heteroatomic speciation (compounds with S, N, O, Ni, V and others), aromaticity and carbon number. FT-ICR MS offers the highest performance and it is the method of choice for fingerprint typification of crude oils. Thereby, the same technology and analytical approaches can be applied to the study of bio-oils.

For a comprehensive characterization of the pyrolysis of African palm bio-oil and its esterified product, GC-MS and FT-ICR MS techniques were employed. This allows to characterize high and low volatile compounds of the samples. The unique elemental composition obtained by ultrahigh resolution mass spectrometry allows the ability to assign around two thousands of compounds, which are dominated by Ox species. Higher Ox containing species (O5-O14) were identified by FT-ICR MS but could not be observed using GC-MS. Esterified bio-oil compounds contain lower Ox containing species than the crude bio-oil, showing the effectiveness of the esterification used as upgrading technique of this bio-oil. Thus, the relative abundance of compounds containing more than eight oxygen atoms was significantly reduced after esterification.

Understanding the possibilities of solvent-assisted post-column refocusing to enhance detection limits in 1-D and 2-D LC (Seminar)

Author: Vincent Pepermans, Vrije Universiteit Brussel
Country: Belgium
Co-Authors: Jelle De Vos, Gert Desmet, Sebastiaan Eeltink

Session: Comprehensive Chromatography - The State of the Art
Day & Time: Wednesday 24th January 2018, 11:20 - 11:40
Room: Room D

The detection of trace levels of analytes in liquid-chromatographic analysis constitutes one of the major analytical challenges. The fundamental nature of LC is such that the constituents in a sample mixture are diluted when they are distributed between the mobile and stationary phase, which negatively influences the limit of detection. This is even more detrimental in two-dimensional liquid chromatography (LC * LC), where dilution is even a larger bottleneck since the dilution factors in the two consecutive chromatographic processes are multiplicative.

We report on a generic method for the enrichment of analytes after their separation on an analytical column, prior to detection. In this strategy, the analytes are lead to a trap column packed with particles containing a strongly retaining stationary phase after which they are re-eluted using a strong solvent in a backward elution mode. During this re-elution, the trapped bands are refocused as the strong solvent chases the analytes out of the pores of the particles.

It will be demonstrated how, using an optimized trap configuration and elution/remobilization conditions, signal enhancement factors in the order of 10 to 20 can be achieved. Emphasis will also be put on the requirements in viscosity matching between the trapping solvent and the elution solvent. In addition, some general rules for the optimal conditions (trapping time, elution solvent composition, direction of elution) and trap design (particle type, length and cross-section) will be given, in combination with estimates of the maximal degree of refocusing that can be expected. Emphasis will also be put on the possibility to miniaturize the trap columns, and the entailing problems and limitations.

Applicability of retention modelling in hydrophilic-interaction liquid chromatography for algorithmic optimization programs with gradient-scanning techniques (Seminar)

Author: Bob Pirok, University of Amsterdam
Country: Netherlands
Co-Authors: Stef Molenaar, Rianne van Outersterp, Peter Schoenmakers

Session: Comprehensive Chromatography - The State of the Art
Day & Time: Friday 26th January 2018, 11:00 - 11:20
Room: Ferrier Hall

Comprehensive two-dimensional liquid chromatography (LCxLC) is an essential technique for the separation of highly complex samples of non-volatile molecules. In principle, the development of an LCxLC method requires establishing two separation dimensions with vastly different ("orthogonal") selectivities. However, with the advent of state-of-the-art instrumentation for LCxLC, the number of options to realize and optimize LCxLC separations is increasing dramatically. The challenge of optimizing tailored LCxLC separations is horrendous, yet it essential to address this challenge if sophisticated LCxLC systems are to be utilized to their full potential in an efficient manner. Method development lengthy and cumbersome and developing methods that make full use of the possibilities of the instrument (e.g. shifting gradients) is barely possible to date.

To facilitate rapid method development of LCxLC methods, the Program for Interpretive Optimization of Two-dimensional Resolution (PIOTR) was developed [2]. It allows rigorous, comprehensive optimization of LCxLC methods by interpreting a very limited number of scouting LCxLC(-MS) experiments. Using gradient-elution theory, large number of possible LCxLC methods are simulated for the interpreted sample, and evaluated using quality descriptors.

However, software tools such as PIOTR require accurate models to describe retention and the exact mechanism and thus model for retention in hydrophilic-interaction chromatography (HILIC) is known to be rather complex. We have evaluated the performance of five different retention models for hydrophilic-interaction chromatography (HILIC) for a wide range of analytes. In this presentation, the results are presented.

Quantitative proteomics for molecular diagnostics of public health (Seminar)

Author: Jack Rice, University of Bath
Country: United Kingdom
Co-Authors:

Session: Energy & the Environment
Day & Time: Thursday 25th January 2018, 15:10 - 15:30
Room: Room D

Wastewater based epidemiology (WBE) is an established technique that allows for the analysis of wastewater and environmental samples within a framework where the data is suitable for interdisciplinary use by epidemiologists. The biggest success of WBE is for the analysis of drugs of abuse, where near-real time monitoring of wastewater from across Europe using LC-MS/MS is combined with traditional sources of data, such as police seizures, to create a comprehensive overview of drugs abuse on a continental scale. The advantage of WBE over these traditional data sources is that a single sample of wastewater from a treatment works can be treated like a community wide pooled urine sample, meaning one sample can be representative of the whole community contributing to that treatment works.

Meanwhile in the field of clinical proteomics the use of mass spectrometry is becoming more wide spread, largely competing against single analyte detection methods like ELISA, focussing on multiplex analysis and the use of MS in biomarker identification. In many ways clinical proteomics is currently in the same state as drug of abuse monitoring before WBE, i.e. focussed on capturing data on individuals rather than whole communities at once. Attempts have been made to extend clinical proteomics to a community wide level through analysis of blood and urine samples. Whilst this studied generated very important results on the excretion on C - reactive protein, an inflammation biomarker, in a healthy population it required the analysis of ˜60,000 to analyse only 10% of the population surveyed, approximately ˜7800 people. If a WBE approach had been used this population could have been surveyed by collecting only one sample.

Mass spectrometry within clinical proteomics is generally focussed around the analysis of peptides produced via enzymatic digestion of protein targets or as part of a broad screening approach, which is known as bottom-up shotgun proteomics. This style of analysis uses similar classes of spectrometry to those used within WBE analysis of drugs of abuse. We have developed a method for the detection of proteins of infectious disease in wastewater using a hyphenated, dual instrument approach of either reverse-phase or HILIC coupled to Q-ToF MS for target screening or HILIC coupled to triple quadrupole MS for sensitivity and target quantification. This approach allowed for the selection and identification of peptides from four protein biomarkers and the detection of one of these within wastewater.

HTC-15

Separation of isomeric metabolites of carbamazepine by liquid chromatography and high resolution accurate mass. (Seminar)

Author: Joanne Roberts, Glasgow Caledonian University
Country: United Kingdom
Co-Authors: John MacLachlan, Moyra McNaughtan

Session: Hyphenated Techniques for Comprehensive Analysis
Day & Time: Wednesday 24th January 2018, 16:50 - 17:10
Room: Ferrier Hall

The environmental concerns about the presence of excreted pharmaceuticals in wastewater are well documented and the introduction of high resolution accurate mass (HRAM) spectrometers such as Time of Flight and Orbitrap instruments has aided their detection. Although HRAM is a highly specific technique, interferences can occur especially in a complex matrix such as wastewater. This paper describes how some of the problems encountered were overcome when analysing wastewater samples for carbamazepine and its metabolites using a Thermo Scientific Orbitrap Q Exactive HRAM instrument.

Carbamazepine is a widely prescribed drug used to treat epilepsy and neuropathic pain and it is known to be a persistent environmental pollutant. Many drugs are metabolised prior to excretion, however metabolite presence in wastewater is rarely reported. Carbamazepine is excreted mainly as the di-hydroxy metabolite but also forms an epoxide metabolite which is known as being toxic in the environment. In addition to the epoxide and di-hydroxy metabolites there are five mono-hydroxy metabolites of carbamazepine having the same precursor ion exact mass as the epoxide. As well as having the same precursor ion they are all structurally similar and on fragmentation yield the same product ion in high abundance. This makes it difficult to distinguish between the different metabolites even using HRAM and requires careful interpretation of the data. Thus the presence of mono-hydroxy metabolites could result in higher quantities of the epoxide being reported if they are not adequately separated. Therefore, robust chromatography and careful interpretation of the precursor and product ion data is required to ensure the correct analyte was selected and accurately measured. The analysis is further complicated by the breakdown of other carbamazepine metabolites in the ion source to the same precursor and product ions as the epoxide.

The excellent sensitivity and resolution of the instrument aided the ability to inject the samples directly on to the liquid chromatography mass spectrometry (LCMS/MS) system hence, no analytes were missed due to poor recovery during a sample clean-up or concentration step.

A robust LC method was developed ensuring baseline resolution of any interferences, including those partially broken down in the ion source. (Due to the lack of standards, a sample of waste water was used to determine the retention times for all the metabolites.) The MS transition m/z 253.10 _ 210.0921 was the most abundant product ion for the epoxide and interferences. Analysis of wastewater samples using the developed LC method determined eight peaks with this transition. With careful selection a more specific transition (m/z 253.10 _ 182.0971) was determined for the epoxide which further distinguished it from the interferences. Using the improved chromatography method and the specific transition afforded a good quantitation method for the epoxide.

HRAM is extremely sensitive and selective however, robust chromatography is essential for complex mixtures and care has still to be taken interpreting the HRAM data to prevent interferences and false positives.

All Ion Differential Analysis in Product Control Applications using GC/MS and Comprehensive GCxGC/MS (Seminar)

Author: Marco Ruijken, MsMetrix
Country: Netherlands
Co-Authors:

Session: Advanced Analysis of Food and Beverages
Day & Time: Friday 26th January 2018, 11:20 - 11:40
Room: Ferrier Hall

Many applications in industry, using GC/MS or Comprehensive GCxGC/MS, relate to finding differences between a newly measured Sample and a so-called Reference Sample. These questions may typically arise in application areas like Product Control or during Trouble Shooting. Examples are: what new impurities are present in a new batch compared to a reference batch, or why does this product behave differently compared to our reference batch, or the comparison of samples in Food Fraud applications to detect illegally added substances. Typically for the above examples is the limited time available to solve these problems. Furthermore, most of the time only a few samples are available, which excludes the use of statistical comparison tools as applied in the field of Metabolomics.

Although GCxGC-MS has become an invaluable laboratory analysis tool, the procedure may produce gigabytes of data per sample in four dimensions, which makes data analysis time consuming and complicated. In the presentation new methods and software tools will be presented to quickly find differential components from a comparison between two samples only, applicable to GC/MS and GCxGC/MS.

Certainly, comprehensive GCxGC/MS is a technique having superior separation capabilities compared to 1-dimensional GC/MS, but co-elution or near co-elution still might occur, especially in complicated matrices. Whereas most software tools for GCxGC/MS use processing of "TIC" data only, our new methods apply data analysis using the "All Ions" approach. The implemented method allows for the detection and deconvolution of differential components that are not or badly separated, even in two dimensions. It will be demonstrated that processing using the "All Ion" approach will substantially detect more (differential) components, compared to the analysis using TIC data only.

Technical details of the algorithms will be explained and examples will be given from applications like Food Analysis, Product Control in Flavor & Fragrance industry and from Base Chemistry industry.

Recent advances in the analysis of protein biopharmaceuticals (Keynote)

Author: Koen Sandra, Research Institute for Chromatography
Country: Belgium
Co-Authors: Emmie Dumont, Pat Sandra, Mieke Steenbeke, Jonathan Vandenbussche, Isabel Vandenheede, Gerd Vanhoenacker

Session: (R)evolutions in Biopharmaceutical Analysis (KVCV)
Day & Time: Wednesday 24th January 2018, 10:30 - 11:00
Room: Ferrier Hall

Protein therapeutics are becoming a core aspect of the pharmaceutical industry. Together with a huge therapeutic potential, these molecules come with a structural complexity that drives state-of-the-art chromatography and mass spectrometry to its limits. The present contribution reports on recent advances in the analysis of protein biopharmaceuticals at protein, peptide and glycan level. The versatility of hydrophilic interaction chromatography (HILIC), the benefits of instrument and column inertness, the power of comprehensive two-dimensional liquid chromatography (LCxLC) and the possibilities of micropillar array columns (µPAC), all in hyphenation to high-resolution mass spectrometry (MS), will be discussed and exemplified using therapeutic enzymes, monoclonal antibodies (mAbs) and antibody-drug conjugates (ADCs).

Multi-dimensional liquid chromatography of complex mixtures (Plenary)

Author: Peter Schoenmakers, University of Amsterdam
Country: Netherlands
Co-Authors:

Session: Comprehensive Chromatography - The State of the Art
Day & Time: Friday 26th January 2018, 15:20 - 16:10
Room:

Liquid chromatography (LC) is a well-established, robust and reliable analytical separation technique. It is pervasively applied to address a wide variety of analytical questions. However, LC is not inherently a high-resolution technique. The 100,000 theoretical plates that can routinely be obtained by gas chromatography or capillary electrophoresis can only be obtained in LC at the expense of relatively long analysis times. This feeds into the need for hyphenated system. On-line sample-preparation techniques and hyphenated detection methods, such as LC-MS, reduce the burden on the LC separation. Alternatively, we can strive to increase the separation power of LC.

One of the best ways to achieve the latter is comprehensive two-dimensional liquid chromatography (LCxLC). This technique has several major advantages. It provides high peak capacities (up to ca. 10,000, as compared to 1,000 for conventional one-dimensional LC), additional selectivity and, in many cases, structured and readily interpretable chromatograms. LCxLC is a highly worthwhile approach, provided that incompatibility issues (the effluent of the first-dimension separation is, in principle, the injection solvent of the second-dimension separation) can be overcome and that method development is adequate and efficient.

Potentially, LCxLC separations may also be performed in a "spatial" mode, using a flat-bed stationary phase, development in the first dimension ("separation in space") and elution in the second and final dimension ("separation in time", hence xLCxtLC). Because all second-dimension separations are performed simultaneously, spatial LCxLC, when implemented successfully, is expected to outperform conventional, column-based LCxLC.

The real challenge, with great potential rewards, is to perform spatial three-dimensional LC separations (xLCxxLCxtLC). Analytes are separated "in space" (i.e. based on their x-y coordinates after the first two separations), followed by elution in the z-direction. For extremely complex samples, with many thousands of relevant analytes, spatial three-dimensional LC is ultimately the way to go, with predicted peak capacities of the order of one million.

In this presentation, the advantages and practical application of column-based LCxLC will be demonstrated based on examples from (industrial) practice and progress towards spatial two- and three-dimensional spatial chromatography will be discussed.

HTC-15

Matching 1st and 2nd dimension chemistries in the 2D-LC. Active Solvent Modulation (Seminar)

Author: Konstantin Shoykhet, Agilent Technologies
Country: Germany
Co-Authors:

Session: Fundamentals in Separation Science (KVCV)
Day & Time: Wednesday 24th January 2018, 14:50 - 15:10
Room: Lower Hall

Two-dimensional liquid chromatography (2D-LC) is often used to solve difficult separation problems ranging from targeted analyses of structurally similar molecules to untargeted separations of highly complex samples. In a 2D-LC system, aliquots of effluent from the first dimension column (1D) are transferred to the second dimension column (2D) with the goal to separate the compounds that might have co-eluted in 1D.

One of the well-known challenges in the 2D-LC is matching the elution strength of the 1st dimension effluent to the separation method in the 2nd dimension. In many cases (RP xRP; HILIC x RP; pH-mismatch) the 1D sample matrix (solvent in which compound was sampled for injection into the 2nd dimension) may be too strong for sufficient retention on the 2D column. This can lead to severe peak distortion and a loss of resolution and sensitivity, especially if the aliquot volumes transferred to 2D are significant.

It is thus desirable to adjust the aliquoted solvent prior to injecting it onto the 2nd-D column in order to avoid elution artifacts. In the case of on-line conjugation of the 1st and 2nd dimension, i.e. when the aliquots are provisionally stored in loops (rather than in vails), such solvent adjustment can be challenging.

This presentation shows the concept of Active Solvent Modulation (ASM). This novel valve-based approach not requiring additional hardware enables sample dilution by a pre-configured dilution factor prior to application of the sample to the 2nd-D column.

The effect of ASM on the gradient shapes arriving on the column in the 2ndD and baseline shapes in sensitive applications, like those using TFA or FA as eluent additive is discussed. Improvement of the separation performance in the 2nd dimension by applying ASM is demonstrated on a case study.

Flow chemistry: A synthetic chemist's perspective (Tutorial)

Author: Anna Slater, University of Liverpool
Country: United Kingdom
Co-Authors:

Session: Microfluidics & flow process technology
Day & Time: Wednesday 24th January 2018, 16:00 - 16:30
Room: Room D

Flow processes have been used in analytical science for decades; an early example is the AutoAnalyzer invented by Skeggs in the 1950s. Process analytical technology (PAT) is increasingly used in industry to monitor flow and batch processes in real time, with attractive advantages over offline methods. However, opportunities exist to develop new methodologies for monitoring laboratory and plant scale processes to achieve both final product quality, and ease the transition between lab discovery of new materials and their industrial use.

This tutorial will focus on the flow process to be monitored, giving examples of chemistry carried out under continuous flow conditions. An introduction to the benefits and implications of moving from batch to flow will be covered, discussing which classes of reactions are best suited to such a transfer.

Emphasis will be placed on the benefits analytical process monitoring can bring, such as greater understanding, improved safety, and higher R&D efficiency. Finally, some of the challenges of monitoring flow processes will be discussed, with examples from the flow chemistry literature used to show recent advances in the area.

Monitoring the effect of post-harvest storage on fruit quality by TD-GCxGC-TOF MS (Seminar)

Author: Natasha D. Spadafora, University of Calabria/Markes International
Country: United Kingdom
Co-Authors: Maria B. Bitonti, Nick Bukowski, Laura McGregor

Session: Comprehensive Chromatography - The State of the Art
Day & Time: Friday 26th January 2018, 13:50 - 14:10
Room: Ferrier Hall

The fruit quality (FRUITY) project aims to provide a better understanding of post-harvest storage conditions of fruit, to allow improved sensorial and internal quality of fruit throughout the supply chain.

The project uses a multi-trait approach - including sensory profiling, monitoring of the volatile organic compounds (VOCs) produced by the fruit and investigation of biochemical reactions - with the overall goal of providing a suite of simple diagnostic checks to monitor fruit quality.

In this presentation, we will focus on the VOC bouquets from peach cultivars in an attempt to identify molecular markers for objective quality assessment.

Thermal desorption (TD) enables rapid and robust in-situ sampling of VOCs, on to sorbent tubes that can be subsequently capped for safe transport to the laboratory for analysis. Here, we use comprehensive two-dimensional gas chromatography coupled with time-of-flight mass spectrometry (GCxGC-TOF MS) to provide enhanced separation of these complex samples. The pre-concentration effect of TD, combined with improved separation and highly-sensitive detection by GCxGC-TOF MS provides a comprehensive chemical fingerprint in a single analytical run.

The VOC profiles at the time of harvest and after storage at low temperature will be compared and correlated with results from sensory evaluation.

HTC-15

Rapid evaporative ionization mass spectrometry for high throughput screening in food analysis: the case of boar taint (Seminar)

Author: Sara Stead, Waters Corporation
Country: United Kingdom
Co-Authors: Nathaniel Martin, Zoltan Takats, Lynn vanhaecke, Kaat Verplanken, Jella Wauters

Session: Advanced Analysis of Food and Beverages
Day & Time: Friday 26th January 2018, 13:30 - 13:50
Room: Ferrier Hall

Introduction

Increasing awareness on animal welfare has led to a European Treaty announcing a voluntary ban on the surgical castration of piglets by 2018. An alternative is to raise entire males however; the limitation is the possible occurrence of "boar taint" an off-odour caused by the accumulation of indole (IND), skatole (SK) and androstenone (AEON) in adipose tissue. IND and SK are indolic compounds derived from the degradation of L-tryptophan in the hindgut and their odour is often described as faecal-like. AEON is a pheromone produced in the Leydig cells of the testis having a urinary or sweaty like odour. To prevent adverse consumer reactions there is an urgent need for rapid methods for detecting "boar taint" at the slaughter line.

Methods

Rapid Evaporative Ionization Mass Spectrometry (REIMS) was used as an emerging direct analysis technique to train a predictive model for accurate high-throughput identification of boar taint above the odour threshold using representative samples of characterised pig adipose tissue. Adipose tissue was sampled using the iKnife handheld monopolar device, which was connected directly to a Xevo G2-XS Q-TOF mass spectrometer equipped with a REIMS source (Waters corporation, Manchester, UK). For each sample, 2 technical replicates were taken with a sampling time of 3-5 seconds. Untargeted mass spectrometric profiling in both negative and positive ionisation mode of pig neck fat samples enabled the construction of statistical models for the classification of pig carcasses in boar taint positive or negative groups and sows.

Preliminary data

To demonstrate the classification potential of REIMS, blank (sow), boar taint positive and negative carcasses were included, in total of 150 samples. Both negative and positive ionization modes were investigated to increase the range of detected metabolites and were considered separately. In negative ionization, better classification accuracy (98%) was observed compared to positive ionization (94%). The OPLS-DA model showed good separation between sow and boar groups. The two boar groups showed some overlap, nevertheless, cross-validation demonstrated the model had a total correct classification rate of 99% and consequently could be used as an accurate predictive tool for boar taint. All blank and negative samples were correctly classified, whereas of the boar taint positive samples, 98% were correctly classified. The remaining 2% were classified as negative. The classification results obtained by chemical and sensory analysis, (which were used as Y-information for model building) could form the basis of the miss classification of these samples. Based on the sensory scores of the neck fat samples, these samples were severely tainted. The validity of the model was evaluated through R2(Y) and Q2(Y), CV-ANOVA testing and permutation tests. The results obtained in this study demonstrate that despite the lack of sensitivity for the boar taint compounds in targeted detection, tainted carcasses could be correctly classified by an untargeted approach. This makes REIMS suitable for discrimination between gender samples (sow versus boar) and for discrimination within gender (tainted versus untainted). This discrimination originates from alterations in lipid profiles, primarily in the fatty acid and phospholipid regions. As REIMS eliminates extensive sample pre-treatment procedures, analysis takes <10 seconds, it offers potential as the first technique enabling in-situ detection of boar taint combined with accurate classification. REIMS is a promising and powerful tool for other applications in food quality, whereby rapid characterization of food products is requisite.

Novel aspect

Direct screening method for the classification of boar taint positive carcasses generating results within 10 seconds.

HTC-15

UHPLC quantitation and identity confirmation in drug development with a multi-detector approach (Seminar)

Author: Frank Steiner, Thermo Fisher Scientific
Country: Germany
Co-Authors: Mauro De Pra, Katherine Lovejoy, Michael Menz

Session: Challenges in Quantitative Analysis
Day & Time: Thursday 25th January 2018, 11:20 - 11:40
Room: Lower Hall

The analysis of API is required at several stages during the drug development. Monitoring of chemical entities during process development and accelerated stress studies, are just few of the examples where peak identity assignment and quantitation of chemical components is needed in early development stage. However the availability of standards of relevant chemical compounds, such as related impurities, process intermediates etc., at this point of development is limited. The lack of standards prevents quantitation based on calibration curve; additionally peak assignment based on retention time relative to run of the standard is impossible without the respective compounds. Peak assignment could be addressed by monitoring the UV absorption spectra measured with a diode array detector. This approach has severe limitations, for instance the need of a spectra database and most of all it is unsuitable when components co-elute. Therefore it is preferred to hyphenate mass spectrometry to UV detection. In most of cases the detection of the intact mass of the peak is sufficient to confirm an identity, and the availability of the MS spectrum may provide an extra level of confidence in the identity assignment. However this approach does not solve the problem of quantitation. Quantitation without standards is achievable with Charged Aerosol Detector (CAD). With the CAD, the response of non-volatile analytes is highly consistent, provided that the composition of the mobile phase at detector inlet is the same for the all the analytes. This is the case for isocratic elution, or for gradient elution with inverse gradient approach [1]. Thanks to the uniform response, the calibration curve determined with a molecule of choice, for instance the API, will deliver accurate estimation of the amount of the rest of components.

In this work we demonstrate an approach that relies on a multidetector set-up based on LC-UV-CAD-MS. The single quad MS provides m/z values of the peaks to ensure correct peak assignment, and enables detection of co-eluting components. The CAD provides universal quantitation, based on the calibration curve of an available component; in order to ensure that constant mobile phase composition reaches the CAD an inverse gradient will run in parallel and will merge to the analytical flow gradient prior entering the CAD (Figure 1). Moreover the UV detector will complement the data collected by MS and CAD, and acts as valuable tool for method troubleshooting and method transfer.

Laboratory Automation: The Rise of the Machines (Keynote)

Author: Scott Summerfield, GSK
Country: United Kingdom
Co-Authors:

Session: Automating Complex Sample Workflows
Day & Time: Friday 26th January 2018, 10:30 - 11:00
Room: Lower Hall

The dynamics of drug discovery and development are demanding the generation of increasingly complex analytical data to support the progression and filing of drug candidates; whether this be PK-related, biomarker measurements or monitoring safety endpoints. Against this backdrop, Pharma companies are looking to use internal and externally deployed resources to maximum effect in order to mitigate against the spiralling costs of drug progression.

Effective deployment of automation in the laboratory environment is a key strategy to better utilise the costly resource we have in highly trained scientists. In our division this has involved assessing the critical bottlenecks that impact the working practices of our people, not necessarily 'big ticket' automation items but those that facilitate increased agility of staff, better use of lab time and better flow of quality data to project teams. Critically automation equipment on its own is not enough because a culture of acceptance for the implementation of new technologies has to play its part. This presentation will cover elements of automation from the small to transformational, building trust with lab scientists and also what lies ahead in the bioanalytical sciences supporting drug discovery and development.

Structure driven prediction of retention :Improvement of accuracy (Seminar)

Author: Roman Szucs, Pfizer
Country: United Kingdom
Co-Authors:

Session: Other
Day & Time: Thursday 25th January 2018, 15:10 - 15:30
Room: Ferrier Hall

Identification of a suitable starting point for chromatographic method development (stationary phase and mobile phase) is essential to ensure maximum robustness and ruggedness of the final method. In routine practice, this is typically achieved by screening a fixed number of stationary phases which offer significant differences in selectivity in combination with multiple mobile phases. Although such an approach represents a significant improvement over random selection of stationary phases, there are certain limitations. The entire selectivity space is not sufficiently covered, as only 4-6 stationary phases are typically screened with up to 6 mobile phases. Screening and subsequent data processing is time consuming, costly and also generates large amount of waste in the form of toxic solvents as well as data.

A study on the use of analyte chemical structures to permit prediction of retention times and thereby to select optimal chromatographic conditions is reported. In this approach, relevant molecular descriptors (features), generated from molecular modelling, are selected utilizing various statistical techniques and evolutionary algorithms. Mathematical relationships are then developed between selected features and measured retention times for training sets of compounds. These were chosen taking into account structural similarity of the "new compound", retention time of which is to be determined.

In this contribution we focus on optimization of retention models, selection of relevant features and provide initial insights on the impact of structural similarity between the training and test sets on accuracy of retention time prediction. The required size of the training sets will also be discussed for various scenarios of similarities between training sets and test compounds. Finally, the above described approach is applied to alternative separation mechanisms such as RP-LC, HILIC and Ion Chromatography.

From GC-MS to LC-MS/MS: Further Advances in Adrenal Cancer Diagnosis (Seminar)

Author: Angela Taylor, University of Birmingham
Country: United Kingdom
Co-Authors: Wiebke Arlt, Irina Bancos, Michael Biehl, Alice Sitch

Session: Advances in Clinical Analysis
Day & Time: Friday 26th January 2018, 13:10 - 13:30
Room: Room D

Discriminating adrenocortical adenoma (ACA) from adrenocortical carcinoma (ACC) represents a continuous challenge in patients with adrenal masses.

We previously established urine steroid metabolomics, (the combination of urinary steroid profiling by GC-MS and machine learning-based data analysis), to distinguish adrenal cancer from benign adrenal tumours, achieving higher sensitivity and specificity than currently available imaging techniques. (JCEM2011; 96 (12): 3775-84). However, GC-MS is labour-intensive, expensive and low-throughput. Here we developed a urinary steroid profiling method using liquid chromatography tandem mass spectrometry (LC-MS/MS) and report method optimisation (using Multi-platform Unbiased-optimisation of Spectrometry via Closed Loop Experimentation software MUSCLE), validation, and cross validation to GC-MS.

We analysed 24-hr urinary steroid excretion by GC-MS and LC-MS/MS in: 481-anonymised comparison urines from a range of disorders associated with steroid excess and deficiency 129 healthy controls 99-ACA 40-ACC Comparison of the mass spectrometry methods using correlation and Bland-Altman plots showed significant $P<0.001$ correlations for all steroids (range 0.78-0.90).

To determine diagnostic accuracy, (the ability to distinguish ACC from ACA), ROC curves were generated. GC-MS and LC-MS/MS demonstrated similar diagnostic accuracy, (area under the ROC curve average 0.969 (SD=0.044) and 0.954 (0.067) respectfully).

Development of this novel LC-MS/MS method represents a significant advance diagnosis of ACC.

Petroleomic Depth Profiling of Staten Island Soil by GC and FT-ICR MS (Seminar)

Author: Mary Thomas, University of Warwick
Country: United Kingdom
Co-Authors:

Session: Energy & the Environment
Day & Time: Friday 26th January 2018, 11:40 - 12:00
Room: Room D

The New York/New Jersey Harbor (NY/NJ) Estuary is in a region with one of the highest population densities in the United States, with many heavy waste generating industries based in the area. Unsurprisingly, the NY/NJ Estuary was ranked among the most chemically contaminated waterways in the United States based on surface sediment concentrations. Elevated body burdens of toxic substances including heavy metals, petroleum hydrocarbons, and aromatic hydrocarbons have also been detected in a wide range of aquatic wildlife.

Environmental samples, particularly those contaminated with petroleum-related products, are highly complex. Using state-of-the-art analytical techniques, comprehensive molecular characterization of petroleum-related samples can be achieved. Following such analyses, unique fingerprints can be established for the samples, allowing any transmission into the environment to be traced. The characterization of petroleum-related samples using mass spectrometry has been termed 'petroleomics', with the molecular formulae of tens of thousands of components observed in a single spectrum.

In this study, soil was sampled at 5 depths in Staten Island, New York, with the aim of correlating the compositional fingerprints of the petroleum extracts with the history of the area. The extracts were profiled by a combination of gas chromatography (GC) and Fourier transform ion cyclotron resonance mass spectrometry (FT-ICR MS). GC provided a summary of the bulk properties of the extracts including total petroleum hydrocarbon (TPH), polycyclic aromatic hydrocarbon (PAH), and sediment toxicity (microtox) analyses. FT-ICR MS revealed more detailed compositional differences between organic contributions at different depths. The relative contribution from more highly oxygenated organic compounds was found to increase as the sampling depth increased. Changes in the contributions from sulfur-containing classes were also observed. Each individual component and its contribution can be visualised further in a plot of double bond equivalents against carbon number for each compound class.

The detailed molecular characterization obtained by FT-ICR MS coupled with the bulk information determined by GC allows a fingerprint of the petroleum-based contaminants of the soil to be developed. Petroleomic profiles of soil as a function of depth allow contamination to be correlated with the site history, and aging of petroleum-based compounds over time to be better understood.

Mass Spectrometric Investigation of Compounds of Interest to the Chemical Investigation Programme (CIP) within Environmental Matrices: Homogenate Analysis (Seminar)

Author: Rachel Townsend, Swansea University
Country: United Kingdom
Co-Authors: Claire Desbrow, Ruth Godfrey, Geertje van Keulen

Session: Screening Enviroment Pollutants, what can the data tell us?
Day & Time: Friday 26th January 2018, 11:20 - 11:40
Room: Room D

The environmental persistence of organic pollutants, such as pharmaceuticals is a growing area of research. Until the introduction of Environmental Quality Standards (Directive 2008/105/EC), the impact of drug emission into the environment through wastewater treatment plants has been largely unconfirmed and unrestricted. Research has shown that compounds with high octanol-water partition coefficient (Kow), like many common pharmaceuticals, are not biodegraded during wastewater treatment and are able to bioaccumulate, adsorbing to soils and sludge. As treated sludge is routinely deposited on land, it is important to understand the extent of any chemical accumulation. Focussing on compounds of interest to the Chemical Investigation Programme; a British research initiative concentrating on the monitoring of pollutants in sludge, a reverse-phase liquid chromatography-mass spectrometry (LC-MS) method has been developed for multi-residue detection and quantitation of 10 pharmaceuticals, with a modified QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) sample preparation method. The modifications made to the QuEChERS method have proven to be effective for other chemical pollutants and offer increased recoveries of pharmaceuticals, with reduced matrix interferences, to reliably quantitate these compounds in complex environmental matrices, such as sludge and biota, using an internal standard approach.

Hyphenated microdialysis and chromatography to monitoring protein free drug for pharmacokinetic study in rat (Seminar)

Author: Tung-Hu Tsai, National United University / National Yang-Ming University
Country: Taiwan, Republic of China
Co-Authors:

Session: Hyphenated Techniques for Comprehensive Analysis
Day & Time: Wednesday 24th January 2018, 11:20 - 11:40
Room: Lower Hall

To explore the pharmacokinetic of protein unbound drug, a hyphenated microdialysis and chromatography was developed to monitoring the level of protein free drug for absorption, distribution, metabolism and excretion study in rat. The microdialysis membrane is semi-permeable depending on the molecular weight cut-off and does not allow larger molecules penetration. Only protein free drug is permitted to penetrate through microdialysis membrane. Due to the protein free fraction is the pharmacological active form, which is available for pharmacokinetic and pharmacodynamic action. Besides, only protein free form of the drug molecule can be delivered to the target sites for therapeutic actions. Then, this technique is gaining the popularity in preclinical pharmacokinetic and pharmacodynamic studies. To investigate the brain, muscle, liver or kidney distribution of the drugs, single or multiple microdialysis probes has been applied in the experimental animal for blood, brain, muscle, liver, kidney ...etc. multiple targets. The regional brain distribution, the portion of drug that passes through the blood-brain barrier, liver and kidney distribution can be defined by the area under the curve ratio of blood-to-brain, blood-to-liver and blood-to-kidney, respectively. P-glycoprotein (P-gp) is a product of the multidrug resistance gene, which also corresponds to diminished intracellular accumulation of the drugs. P-gp also contributes to the barrier functions of the brain by extruding compounds that are potentially toxic to the brain from the capillary endothelia cells. In addition, biliary excretion has been recognized not only to depend on bile salts secretion, but also to require the active participation of the P-gp. To investigate the mechanism of P-gp modulation on the blood brain barrier and biliary excretion, a P-gp inhibitor was administered in the experimental animal. Furthermore, the pros and cons of microdialysis will be discussed, including the detailed surgical techniques in animal experiments from rat blood, liver and kidney for the analysis of protein-unbound drug.

The comparison of Unispray and Electrospray for the ionization of neuropeptides (Seminar)

Author: Yannick Van Wanseele, Vrije Universiteit Brussel
Country: Belgium
Co-Authors: Ilse Smolders, Ann Van Eeckhaut, Marijn Van Hulle, Laurence Van Oudenhove

Session: Interfacing and Ionisation
Day & Time: Wednesday 24th January 2018, 14:50 - 15:10
Room: Room D

In the world of bioanalytical chemistry, electrospray ionization (ESI) is one of the most prominent ionization techniques. Its soft ionization mechanism and compatibility with liquid chromatography are the main incentives leading its popularity. However, the technique is prone to low ionization efficiency and low ion transmission towards the mass spectrometer vacuum chambers. To tackle these drawbacks, liquid chromatography infusion flow rates were reduced over the years to nanoL/min, creating smaller droplets and as such aiding desolvation. In parallel, variations in source design were explored for increasing the ion transmission through the sample cone.

More recently, a new ionization source was developed and commercialized as 'Unispray'. In this atmospheric pressure setup, a grounded solvent flow is nebulized onto a high voltage charged rod. In addition to ionizing the compounds of interest, Coand_ and vortex effects aid droplet break-up and desolvation. In the last two years, differences in ionization between ESI and Unispray were already thoroughly investigated by Lubin et al for the analysis of small molecules . In our experiments we compared the ionization of seven neuropeptides, varying between 6 and 36 amino acids, via Unispray and ESI at different impactor and capillary voltages respectively. All peptides were infused in acetonitrile/water (30:70 V/V) containing 0.1% formic acid at 100 µL/min. We also examined the effect of different additives such as m-nitrobenzyl alcohol, dimethylsulfoxide and sulfolane on the peptide charge state distribution and signal intensity.

Chromatographic fingerprints: chemometrics and application in method development (Seminar)

Author: Yvan Vanderheyden, Vrije Universiteit Brussel
Country: Belgium
Co-Authors:

Session: Big Data Chemometrics and Method development (In-Silico) (KVCV)
Day & Time: Thursday 25th January 2018, 14:30 - 14:50
Room: Ferrier Hall

A fingerprint is a profile which represents the composition of a sample and thus is characteristic. Various separation (chromatographic, electrophoretic) and spectroscopic (NIR, FTIR, Raman, NMR) techniques can be applied to obtain such profile. The fingerprints can be applied in the context of quality control aspects of food products, dietary supplements and pharmaceutical samples. Despite the diversity in techniques to measure fingerprints, they all have the common feature of containing a large amount of information, which requires chemometric tools for a systematic data analysis. For all applications, either the complete fingerprint is considered, or a number of selected variables is used. The data handling techniques applied depend on the purpose of the fingerprinting application, which can for instance be identification and quality control, classification, modeling and prediction of activities. Different applications and some possible data handling approaches for each situation will be discussed and illustrated.

Additionally, fingerprints can also be applied in the optimization of given methods, for instance, in the optimization of the extraction of herbal samples. This application also will be illustrated.

A paradigm shift for (big) data analysis in chromatography: on the use of Bayesian statistics (Keynote)

Author: Gabriel Vivo-Truyols, Tecnometrix
Country: Spain
Co-Authors:

Session: Big Data Chemometrics and Method development (In-Silico) (KVCV)
Day & Time: Thursday 25th January 2018, 14:00 - 14:30
Room: Ferrier Hall

Data analysis methods applied to chromatographic data, including base-line correction, peak detection, alignment and peak tracking, calibration and/or classification are a routine part of most modern analytical workflows. With the emergence of hyphenation (especially high-resolution mass spectrometry) and two-dimensional methods (e.g. LCxLC) new challenges for the data analysis are emerging. We are witnessing a boom of the amount of data to be processed, so we can start to talk about Big Data in Analytical chemistry. Analysing these enormous and complex quantities of data becomes a tremendous challenge, especially because of the need to do it automatically. Traditionally, chromatographic data has been processed using the so-called frequentist approach. With this approach, we get just a final answer about the hypotheses we are testing, but we have no information about its probability of being true.

Contrary to the frequentist approach, Bayesian statistics offers a very interesting alternative, estimating the probabilities of the processes mentioned above. This way of thinking opens a new world of possibilities, especially in the area of automated massive data treatment. In this way, the chromatographer has no longer to "trust" the results of the data analysis, but (s)he has to decide on the different configurations that explain the data, based on the probabilities of each one.

We have applied this way of thinking to a broad range of situations. One example concerns toxicological screening, in which the probabilities of a list of compounds being present in the sample, analysed with LC-MS. Using a Bayesian approach, it is easy to build up evidence about the presence/absence of a compound by taking into account adduct formation, isotope ratios, retention times and mass values, resulting in more accurate values of probability. Another example is a Bayesian view of the well-known peak tracking methods. In (traditional) peak tracking methods, peaks of the same compound are recognized in different chromatographic conditions. A Bayesian thinking approaches the problem in a probabilistic way, i.e. assigning different possibilities of peaks to the different compounds available.

In my opinion, the use of Bayesian statistics to deal with massive data treatment in chromatography constitutes a shift in the way we think about data analysis. Basically, we are proposing to work with probabilities of hypotheses (and update them as long as more information/data is taken into account), opposed to deliver the final answer to the chromatographer.

Improving Untargeted Metabolomics with Ion Chromatography-Mass Spectrometry (Seminar)

Author: John Walsby-Tickle, University of Oxford
Country: United Kingdom
Co-Authors: Joan Gannon, David Hauton, James McCullagh, Elisabete Pires

Session: Hyphenated Techniques for Comprehensive Analysis
Day & Time: Friday 26th January 2018, 13:50 - 14:10
Room: Room D

Metabolomics is a rapidly growing area of mass spectrometry (MS) which maps molecular changes in cells, tissues and bio-fluids. Liquid chromatography (LC) coupled with mass spectrometry (LC-MS) is frequently used to identify molecular phenotypes associated with diseases such as cancer and the effect of pharmacological intervention on metabolic pathways. The principal LC separation methods currently used are reversed-phase (RP) and hydrophilic interaction chromatography (HILIC). HILIC has been shown to perform well for polar metabolites in both targeted and untargeted analysis due to its solvent compatibility with MS and relatively fast run times. However, it requires careful column equilibration and pH balance to maintain a delicate aqueous layer on the stationary phase, leading to poor retention time reproducibility across large numbers of samples.

To date ion chromatography (IC) coupled directly to MS (IC-MS) is relatively rare in metabolomics but has a number of characteristics which makes it an attractive chromatographic approach for coverage of highly polar and ionic metabolites. We have been developing this technique for targeted and untargeted applications and have demonstrated exceptional retention time stability and lower limits of detection when compared to HILIC. This performance is due to on-line eluent generation from water and a concentrated ion solution which leads to highly reproducible chromatographic conditions and low chemical noise. IC-MS also requires minimal sample preparation as cell lysates and bio-fluids can be run directly after filtration, minimising metabolite degradation and intra-sample variability.

Here we demonstrate a number of advantages in using IC as part of multiplatform LC-MS for untargeted metabolomics applied to the profiling of metabolites from glioblastoma cells expressing isocitrate dehydrogenase 1 (IDH1) mutations. IDH1 is a change of function mutation which disrupts the normal citric acid cycle conversion of isocitrate to 2-oxoglutarate (2-OG) and catalyses the production of 2-hydroxyglutarate (2-HG) from both species. IC is an ideal method for the separation of tricarboxylic acid cycle metabolites due to their high polarity and similar functionality which present a challenge for analysis using standard chromatographic techniques.

We analysed cell extracts from multiple glioblastoma cell lines with and without the IDH1 mutation in different media conditions. Three LC-MS approaches were used to ensure maximal coverage of the metabolome: IC, RP and derivatised RP. This ensured that consecutive pathways could be followed and the cumulative impact of pathway modification measured. Our aim was to determine whether metabolic changes occurred as a result of the presence of the IDH1 mutation and if any such changes were also affected by glucose concentration. Here we show the power of IC for untargeted metabolomics and the effect of glucose concentration and IDH1 mutation on the metabolome, especially on glycolysis and pentose phosphate pathways.

UPLC-MS for Metabolic Phenotyping: Advantages, Assays and Applications (Keynote)

Author: Elizabeth Want, Imperial College London
Country: United Kingdom
Co-Authors:

Session: Advances in Clinical Analysis
Day & Time: Wednesday 24th January 2018, 14:00 - 14:30
Room: Ferrier Hall

Ultra performance liquid chromatography coupled to mass spectrometry (UPLC-MS) has become increasingly prevalent in the field of metabolic phenotyping over the past decade. Applications to date have included clinical, toxicological, and epidemiological investigations, improving our understanding of drug metabolism and toxicity, as well as disease diagnosis and prognosis. UPLC has advantages over conventional HPLC, including increased speed and chromatographic resolution, resulting in deeper metabolome coverage with higher sample throughput. This makes it an ideal analytical tool for large scale epidemiological studies, as well as for in depth interrogation of individual biological sample types. In this talk, the field of metabolic phenotyping will be introduced, the typical analytical workflow will be discussed and factors which affect the metabolic profile will be examined. Applications of UPLC-MS in metabolic phenotyping will be reviewed, illustrating advances in biomarker discovery and our understanding of human health and disease.

Despite the 3-5 fold increase in sample throughput that UPLC-MS offers over HPLC-MS, researchers remain interested in further improving speed without compromising metabolite coverage, in order to accommodate large sample numbers and to reduce solvent usage. Improvements to sample throughput will be mentioned, such as rapid microbore metabolic profiling (RAMMP) technology and other miniaturisation approaches. As part of the metabolic phenotyping pipeline, targeted UPLC-MS assays are crucial for quantitation of key metabolites and biomarker validation. Therefore, targeted assays will be introduced, with examples of successful applications in disease diagnosis. Crucially, the importance of quality control measures in all of these assay types will be covered. Finally, the future of this exciting and ever growing area of research will be discussed.

50 shades of Green SFC (Keynote)

Author: Caroline West, University of Orleans
Country: France
Co-Authors:

Session: Green Separations
Day & Time: Thursday 25th January 2018, 14:00 - 14:30
Room: Lower Hall

Green chemistry is defined as chemical processes that reduce or eliminate negative environmental impacts. Supercritical fluid chromatography (SFC) is usually presented as a "green" chromatography technique. How compelling is this supposed greenness of the method? Is it a reality or just green washing? Having practiced SFC for fifteen years, I am willing to question the point, based on a thorough discussion of the features that should pertain to green chromatography: not only the generation of waste and the use of toxic chemicals, but also energy consumption, extra chemical reactions (derivatization) for solubility, separation or detection reasons, possible choice of less toxic co-solvents, analysis time, method development time, and the risk of accidents. In this respect, modern SFC will be compared to current chromatography methods as gas chromatography (GC) and ultra-high pressure liquid chromatography (UHPLC), based on sustainability indicators and case studies.

Direct infusion MS - who needs hyphenation anyway? (Seminar)

Author: Sam Whitmarsh, BP
Country: United Kingdom
Co-Authors: Chrissie Wicking

Session: Analysis of Complex Energy Products
Day & Time: Friday 26th January 2018, 13:10 - 13:30
Room: Lower Hall

It is not hyperbole to say that benchtop, high resolution mass spectrometry (MS) has revolutionised analytical science over the past 20 years and changed what we understand of our world. Direct infusion experiments have existed since the very first mass spectrometers but hyphenation with liquid or gas chromatography has been the dominant application of these instruments over most of that time. The advance of ambient MS techniques and the requirement to reduce analysis times for large data sets - particularly in the omics fields has seen the proportion of direct infusion experiments increase. As MS instruments improve there is perhaps a tendency to view chromatography as old technology that is too slow and awkward to be part of a modern workflow.

This presentation looks at both the advantages and disadvantages of direct infusion mass spectrometry and hyphenated techniques through the lens of the study of complex mixtures such as crude oil and its derivatives. Examples from both the upstream and downstream will be discussed using different atmospheric pressure ionisation, ambient ionisation, and field ionisation MS techniques along with hyphenation to field asymmetric ion mobility spectrometry (FAIMS), liquid chromatography and gas chromatography.

FAIMS and fortune (Seminar)

Author: Christianne Wicking, BP
Country: United Kingdom
Co-Authors:

Session: Ion Mobility - Mass Spectrometry (BMSS)
Day & Time: Thursday 25th January 2018, 11:00 - 11:20
Room: Room D

Petroleum and petroleum related compounds are multi-component mixtures containing compounds with wide ranging chemistries. Integrated oil companies have a requirement to understand these complex mixtures to price crude oil, understand refinery and chemicals process and to develop new products such as fuels and lubricants.

Many techniques are available to characterise these fluids, however often these can be time consuming and produce data that summarises the composition at a low level of granularity. Orbitrap high resolution mass spectrometry has proven effective in the characterisation of complex petroleum samples at a molecular level. However, high resolution mass spectrometry alone is not a panacea. Direct infusion approaches that rely on the resolving power of the instrument alone suffer from C-trap filling issues with reduced detection of low abundance ions where even semi-quantitation proves to be difficult.

A variety of separation solutions have been utilised to combat these issues with this paper focussing on Fast Asymmetric Ion Mobility Spectrometry (FAIMS) using an Owlstone chip system coupled to an Orbitrap using electrospray ionisation mass spectrometry (ESI-MS). ESI-MS is ideally suited for the identification of many petroleum related compounds and is a powerful tool when coupled with FAIMS. FAIMS is an orthogonal separation technique to MS that separates ions by applying an asymmetric field after ionisation and can be used to filter out/separate interfering ions. The research shown here will discuss how FAIMS has been applied to separate different components to better understand petroleum products and for quantification of these compounds.

FAIMS, already a proven technology for the analysis of crude oil scavengers, coupled to an Orbitrap high resolution mass spectrometer has the potential to expand the toolbox of the analytical petroleum chemist and revolutionise how we characterise and quantify petroleum related compounds.

Big Data - When Less is More (Seminar)

Author: Benjamin Woolford-Lim, GlaxoSmithKline
Country: United Kingdom
Co-Authors:

Session: Big Data Chemometrics and Method development (In-Silico) (KVCV)
Day & Time: Thursday 25th January 2018, 11:20 - 11:40
Room: Ferrier Hall

How many different file formats do you use in your work? Word docs and PDFs for analytical methods, Excel spreadsheets for calculations, a multitude of scientific file formats for raw data... One of the challenges of Big Data, also faced by scientists at the bench, is how to reconcile all these different inputs and contexts to make sense of the whole.

The goal of the Allotrope Foundation is to provide a consistent, coherent, and high performance data format. Representatives from the pharmaceutical industry, academia, hardware and software vendors, and government institutes have all come together to build a flexible approach that can be developed and extended to store scientific data of all kinds. Using innovative Semantic Web technologies on top of the well-established HDF5 file format, the Foundation's approach is starting to enable consistent, context-rich data capture from the beginning of the scientific process right to the very end.

This talk will share details of the Allotrope Data Format, a new file format for scientific data that simplifies data sharing, brings toolset flexibility to scientists at the bench, and enables easier access to Big Data techniques. It will also outline the usage of this and other Allotrope products to provide Big Data capabilities in companies today.

Sequential Three-Dimensional Gas Chromatography with Accurate Mass Spectrometry: A Novel Tool for High-Resolution Characterization of Multicomponent Samples (Seminar)

Author: Dandan Yan, Unversity of Tasmania
Country: Australia
Co-Authors:

Session: Comprehensive Chromatography - The State of the Art
Day & Time: Wednesday 24th January 2018, 15:10 - 15:30
Room: Room D

Innovations on the theme of multidimensional gas chromatography (MDGC) and comprehensive two-dimensional GC (GCxGC) separations, with the aim of expanding peak capacity, are of continuing interest in chromatography. This presentation will highlight our work on the development of an integrated multidimensional three-dimensional gas chromatography (3D-GC) approach for the study of secondary metabolite compounds in essential oils. This integrated system incorporates a non-polar first-dimension (1Dnp) separation step, prior to microfluidic Deans switch heart-cutting of a targeted region(s) into a polar second-dimension (2DPEG) column for multidimensional separation (GC-GC). If further separation is required, the effluent from 2DPEG may be modulated to produce a GCxGC separation, using an ionic liquid phase as 3D column (3DIL). In practice, it operates with preliminary 1D separation of a complex matrix using GC-FID, followed by extraction of target regions into a second (GC-GC) and/or a third column (GC-GCxGC) for additional successive separations; i.e. it operates as a sequential GCnp-GCPEGxGCIL. The analytical benefits of employing three varied chemical selectivities in the 3D separation, coupled with accurate mass time-of-flight mass spectrometric detection will be discussed. The described system may be used in a number of modes, but one useful approach is to target specific classes of compounds for improved resolution. This is demonstrated here through the separation and detection of oxygenated sesquiterpenes in hop (Humulus lupulus L.) and agarwood (Aquilaria malaccensis) essential oils.

Online extraction and determination of carotenoids from food sample by means of supercritical fluid extraction-supercritical fluid chromatography-mass spectrometry (Seminar)

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Session: Green Separations
Day & Time: Thursday 25th January 2018, 15:10 - 15:30
Room: Lower Hall

Extraction and detection of bioactive compounds from food samples is a very hard task. Conventional extraction procedures are usually time consuming and can require a very high amount of organic solvents. Moreover bioactive compounds can be photolabile and/or thermolabile, in this cases must be immediately injected to avoid any kind of degradation.

The aim of this research was focused on the development of an online method coupling supercritical fluid extraction and supercritical fluid chromatography (SFC) for the extraction and detection of carotenoids from Tamarillo samples. The online nature of the system, compared to offline approaches, improves run-to-run precision, enables the setting of batch-type applications, and reduces the risks of sample loss and contamination.

Carotenoids were extracted and detected in less than 17 minutes by using few mL of methanol, including free carotenoids, carotenoids monoesters and carotenoids diesters, in a very fast, and efficient way. Multiple extractions, until depletion, were performed on the same sample, in order to evaluate the extraction yield and to obtain quantitative data. The online supercritical fluid extraction supercritical fluid chromatography method developed was then compared with the traditional solid-liquid extraction HPLC analysis giving comparable results. The proposed methodology developed in this work provides a new platform for the online extraction and separation of relatively nonpolar compounds, showing the capability of this methodology to greatly reduce the analytical times and solvent consuming in a very fast and efficient way.