

Chromatography

Visiting the 14th International Symposium on Hyphenated Techniques in Chromatography and Separation Technology (HTC-14)

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The 14th International Symposium on Hyphenated Techniques in Chromatography was held in Ghent, Belgium from 27th to 29th January 2016. An optional short course on Hyphenated Techniques in Supercritical Fluid Chromatography was held the day before HTC-14 on Tuesday 26 January, 2016.

The location for HTC-14, Ghent is a historic city in northwest Belgium, with roots back to the Middle Ages, and sits at the junction of the Leie and Scheldt rivers. Today it's a university town and cultural hub with a cobbled pedestrian zone centre known for medieval landmarks such as 12th-century Gravensteen castle and the Graslei, a row of guildhalls beside the Leie river.



Picture of the rear of het Pand, the venue for HTC-14.

The symposia were again jointly organised by the Royal Flemish Chemical Society (KVCV) and the Separation Science Group of the Royal Society of Chemistry (RSC) and took place in the het Pand which dates from the thirteenth century. Het Pand was used initially as a hospital (Utenhove Hospital) until a lack of capacity forced a change of use and it became a Dominican friary and convent. Since 1963, the building has been owned by the University of Ghent and is now the culture and congress centre.

The site, whilst very grand, has rooms which on the whole are compact, as with most historic sites, but still housed three parallel sessions, provided space for posters where the Belgian beer tasting took place and two exhibition areas housing 15 exhibitors, the majority of which were in an external tent (which thankfully was heated). Coffee breaks and lunches were taken in the exhibition areas.

As usual, industry (52%), academia (22%) and students (26%) were all well represented among the speakers and over 260 delegates from 22 different countries were in attendance. The short course covered different aspects of analytical separation techniques that employ compressed CO₂ as the main mobile phase component, typically referred to as Supercritical Fluid Chromatography (SFC). Starting from an overview of the fundamental aspects of SFC, the unique retention and selectivity properties are highlighted, as are the possibilities of the technique in pharmaceutical analysis. Hyphenation of SFC with MS and its possibilities in multidimensional separations were covered in detail.

When asked what the expectations for HTC-14 were, Frederic Lynen the HTC Chairman said: "The ongoing success of the HTC symposium series is strongly related to the impressive evolution that both high-end separation techniques and mass spectrometry underwent in recent decades. When considering the significant progress which has, for example, been made in the development of increasingly powerful comprehensive separation tools or in the field of increasingly higher resolution mass spectrometry, the

expectations for HTC-14 were that if it should be possible to bring together the main actors in these fields, while also providing a suitable platform for the vibrant community of young and emerging scientists active therein." He continued: "The choice was made to move the location of the HTC-14 conference to Ghent, reflecting the changes the HTC conference has undergone recently and to allow the participants of the conference to appreciate the hospitality of Ghent and its excellent symposium venues. As the preparations of HTC 14 evolved the organising committee became increasingly convinced that HTC14 was heading for a success."

HTC-14 covered many innovative applications based on hyphenated techniques, taking place in 3 parallel sessions, of different lengths, making it difficult and a little frustrating when attempting to participate in the multitude of interesting discussions. The Poster flash sessions started at HTC-13 were continued with great success and five vendor lunchtime seminars were also available.

This review will now focus on a selection of the presentations given during the HTC-14 meeting. However an overview of the main topics covered during the symposium is given here:

- Petrochemical, Environmental, Toxicology and Industrial Analysis
- High-Performance Separations
- Hyphenated Approaches and multi-dimensional LC and GC-MS separations
- Ionisation Techniques and Mass Spectrometry
- New Method Development Strategies and Chemometrics
- Emerging Applications
- Emerging Technologies for Industry
- On-line Analysers and GC
- LC-MS Profiling and Method Development
- The Fundamentals and Power of Hyphenation
- Advances in Column Technology
- Fundamentals in Separation Science
- GC and GCxGC
- Biological Sample Preparation, Analysis of Biopharmaceuticals and BioAnalysis
- Food Analysis
- New Possibilities in Separation Science and Detection
- New Detection Possibilities and Pushing Detection Limits in 2D-LC
- Advances in SFC

The opening session of HTC-13 chaired by Dr John Langley (University of Southampton, UK) and Professor Frederic Lynen (Ghent University, Belgium) consisted of two plenary lectures, the first was to be by Professor Jim Jorgenson (University of North Carolina, USA) who sadly could not make the trip due to illness. Gert Desmet (Vrije University Brussels, Belgium) discussed the 'Building the Ideal Chromatography System: Why, What and How' and expressed the opinion that "instrumentation needs to change radically – getting rid of connection tubing" and "that in the future (20-50 years all columns will be made by 3D printing". Dr Koen Sandra (Research Institute for Chromatography, Belgium) then discussed 'The Power of LC-MS and LCxLC in the Characterization of Recombinant Proteins, Monoclonal Antibodies and Antibody-drug Conjugates'.



Opening Session in the Refter room.

The three parallel sessions started after the morning coffee break with the High Performance Separations being well attended. Oral presentations were given on:

- Towards Hyperformance Two-Dimensional Liquid Chromatography. Professor Peter Schoenmakers (University of Amsterdam, The Netherlands)
- Hyphenation of Intact Protein Separation and Mass Spectrometry for Characterization of Biopharmaceuticals. Professor Govert Somsen (VU University Amsterdam, The Netherlands)
- Kinetic Gain Factors and Peak-Compression Effects in Ultra-High-Pressure LC. Professor Sebastiaan Eeltink (Vrije Universiteit Brussel (VUB), Belgium)
- Successful Generic Approaches for Heartcutting 2DLC with Focus on User-friendliness. Mrs Isabelle François (Waters, Belgium)

The afternoon session selected for review was on 'New Method Development Strategies and Chemometrics' chaired by Dr Achim Treumann (Newcastle University, UK) featured four oral presentations which were given by Dr Stefan Lamotte (BASF SE, Germany) on the 'Bridges over Troubled Waters: Strategies for HPLC method Development of Complex Samples' who discussed why multidimensional LC is impractical, time consuming and expensive and supported the concept of 'Peak Recycling Chromatography' where as soon as a fraction leaves a column it is switched to a second and back to the first and so on.

- On the Use of Bayesian Methods for Automated Data Analysis. Dr Gabriel VIVO Truyols (University of Amsterdam, The Netherlands)
- Nano UHPLC-ESI-MS/MS for the In Vivo Monitoring of Neuropeptides in the Rodent Brain. Professor Ann Van Eeckhaut (Vrije University Brussels (VUB), Belgium)
- Peaks Behaving Badly: Application of 2D-LC to Determine and Control the Reactivity of Samples of a Drug Product Under Different Analytical Conditions During Method Development. Dr Claudio Brunelli (Pfizer, United Kingdom)

After the final presentation on Wednesday evening a Belgian Beer tasting (sponsored by Shimadzu) was held in conjunction with the poster viewings. The area was rather narrow and made for a cosy atmosphere where everyone mingled, sampled the beers and enjoyed passed hors d'oeuvres, all whilst talking hyphenated chromatography.

On Thursday morning, to 'honour individuals deserving special recognition of their innovation or influential work in the field of Separation Science', the John Knox award was presented by John Langley (RSC) to Dr Keith Bartle (University of Leeds, UK) who captivated the packed auditorium with a fascinating trip down SFC memory lane with a plenary lecture on SFC – No Regrets. Keith very adeptly recalled how in 1958 he started in chromatography after reading the 'Golan paper on Capillary GC' and moved on to his utilisation of SFE for Taxane extractions and then Capillary SFC "where it all started to go wrong". He then recalled packed column SFC and the collaborations which led to a unified chromatograph which comprised of GC/SFC/HPLC all in one unit. He then covered a range of applications – commented on the future of SFC and then thanked all his students, graduate students, post doc's and collaborators, a veritable who's who of chromatography, for helping him over the years.

During the subsequent selection of one of the three parallel sessions before the morning coffee break, 'The power of Hyphenation' was discussed in two keynote lectures including:

- Hyphenated Techniques for Organic and Inorganic Analysis in Order to Ensure Patient Safety. Dr David Clicq (UCB Pharma, Belgium)
- Smaller, Better, Faster - Strategies for Metabolite Profiling and Phenotyping. Professor Ian D. Wilson (Imperial College London, UK)

The post break session selected covered, Fundamentals in separation science, with a tutorial and oral presentations:

- Description and Prediction of the Shape of Chromatographic Peaks: A Tutorial. Dr Wim Kok (University of Amsterdam The Netherlands)
- Assessment of Intra-particle Diffusivity in Hydrophilic Interaction Chromatography (HILIC) and Reversed-phase Liquid Chromatography (RPLC) under Conditions of Identical Packing Structure. Professor Deirdre Cabooter (KU Leuven, Belgium)
- The Art of Column Thermostatting in the Presence of Frictional Heating? Dr Frank Steiner (Thermo Fisher Scientific, Germany)
- Simpler, Better, Faster - The Hyphenated Potential of Planar Chromatography. Professor Gertrud Morlock, (Justus Liebig University Giessen, Germany).

The Thursday afternoon, three parallel sessions, covered Biological Sample Preparation, Food Analysis and BioAnalysis with the day culminating in 'the battle of the gurus' where Dr Pat Sandra, Professor Alexander Makarov and Professor Peter Schoenmakers during a sometimes lively and humorous debate vied for the 'head guru' position over a bottle of wine.



The Battle of the Gurus

The HTC-14 Conference Dinner was held on Thursday Evening and was the main social event of the symposium.

The opening session on the Friday, saw a lifetime-achievement-award presented to Hernan Cortes (H.J. Cortes Consulting LLC/Dow Chemical (Retired), USA). The criteria for this award are 'for outstanding achievements in hyphenated techniques in chromatography and for distinguished service to the international chromatographic community'. The award was presented by Dr John Langley, president of the scientific committee.



Hernan Cortes receiving the award

Hernan Cortes in receiving the award discussed "An academic mentality in an industrial environment" and how the end result of research, whether conducted in academia or industry is the same – despite very different approaches and philosophies.

The following session selected for review was on 'Novel Separation Systems' chaired by Dr Frank Sobott (University of Ghent, Belgium) featured four short fifteen minute oral presentations. The oral presentations were as follows:

- Extending the Performance of Microfluidic Membrane Suppressors and Hyphenation with On-Chip Conductivity Detection for Ion-Exchange Chromatography. Ing. Sam Wouters (VUB, Belgium)
- Prospects of Flow Field Thermal Gradient GAS Chromatography (FF-TG-GC). Dr Peter Boeker (University of Bonn, Germany)
- Design Aspects for the Construction of a Microfluidic Device for Comprehensive Spatial Three-Dimensional Liquid Chromatography. Ing. Bert Wouters (Universiteit van Amsterdam, The Netherlands)
- Vacuum Ultraviolet Spectroscopy as a Complementary Detection System to Mass Spectrometry for One- and Comprehensive Two-Dimensional GAS Chromatography. Mr Thomas Gröger (Helmholtz Zentrum München, Germany).

The final award presented during the closing session on Friday afternoon – the HTC award, was presented to Professor Paola Dugo, University of Messina, Italy). The nomination was based on the lecture 'Hyphenated chromatography techniques for complex analysis of lipid samples', judged to be the most valuable and innovative contribution of the conference.

Poster prizes were also presented, the first and second of which were awarded to Morgan Sarrut, (University of Lyon, France) and Sam Wouters, (Free University Brussels, Belgium) for

their posters titled, Optimisation of conditions for the analysis of ADC's by comprehensive on-line 2D HIC x RPLC hyphenated to Mass Spectrometry and – Design of Microfluidic devices for 2D spatial separations and evaluation of flow characteristics respectively. The third prize was awarded to Maud Bonichon, (Free University Brussels, Belgium) for the poster titled, Development and Characterisation of immunosorbents for the selective extraction of butyrylcholinesterase from human plasma, prior to enzymatic micro reactor digestion and micro-LC/MS analysis.

After the successful 14th edition of the symposium on Hyphenated Techniques in Chromatography the next and 15th edition will be organised in Cardiff, Wales, during the last week of January 2018.



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When asked what the focus would be for 2018 Professor Lynen answered: "I'm looking forward to incorporate sessions on hyphenated microfluidic techniques in 2018. We will also try to offer further contributions in the field of bio-therapeutics as this is particular field of interest where there is a further need for improved transfer of knowhow of the most recent hyphenation strategies."

The meeting is also expected to cover the fundamental aspects regarding hyphenation in further detail and more specifically in the field of interface design.

As HTC is a conference which brings together industry and academia and established and young scientists in a unique way, further emphasis will be set towards achieving these goals even more.

HTC-14 in Ghent

