

UNIVERSITY OF YORK HOSTS MASS INVASION OF SPECTROSCOPISTS

The 30th BMSS 3-day meeting was held this year on 7th – 10th of September 2008 on the very pleasant Campus at the University of York. It was the biggest meeting to date with over 350 attendees, around 200 posters, 100 presentations and over 50 exhibitor stands. The lecture programme was structured such that there were keynote presentations followed by parallel sessions. A wide range of topics was covered from instrumental developments, informatics for MS and protein analysis through to 'Omics', Clinical & Toxicological and Imaging MS.

As ever the meeting was aimed at all levels of practicing spectroscopists from the relative novices to the 'I remember when...' generation and for the former group a series of workshops was organised by Brucker Daltronics and Waters Corporation on the afternoon of the 7th.

The meeting 'proper' started with the Maccoli Plenary Lecture given by Professor Graham Cooks from Purdue University, USA who spoke on the topic of 'Ambient Ionisation using Miniature Mass Spectrometers: In-situ Analysis'. The observation was made that in order to achieve 'point and shoot' analysis using MS two Instrument Developments need to be utilised viz, ionisation methods that operate in the ambient environment on unmodified samples and handheld Mass Spectrometers using combined technologies. Examples of the use of such systems were presented with applicability to the analysis of solids, vapours and solutions.

George McLeod (Brucker Daltronics) presented on "A leap forwards in sensitivity and selectivity for PAH analysis using GC-APLI-TOF/MS". Most polyaromatic hydrocarbons (PAH's) containing more than three aromatic rings are considered to be genotoxic and carcinogenic to humans. Therefore, PAH's are classified as priority pollutants to be controlled in food and in the environment. PAH analysis has not been performed effectively with atmospheric pressure ionisation techniques such as standard ESI or APCI. A novel technique using Atmospheric Pressure Laser Ionisation (APLI) coupled to a Brucker TOF or Qq-TOF mass spectrometer was described. Efficient and highly selective ionisation was obtained with an order of magnitude improvement in sensitivity over existing techniques. A method was developed using GC-APLI-MS for 16 PAH's. The method parameters were presented including precision & accuracy, calibration function and limits of detection that demonstrated its application to routine analysis.

Ole Vorm (Proxeon) spoke on "2D-LC-MS made automated, simple and fast, using a split-free nanoscale 1D-LC system". Analysis of complex biological samples often requires several separation steps. One of the favoured technologies in the field of proteomics is two-dimensional liquid chromatography couple with mass spectrometry (2D-LC-MS) using on-line strong cation exchange (SCX)/reverse phase (RP) separations. This involves a level of technical complexity to achieve successfully.

Spectroscopy Focus

The remainder of the morning session was devoted to presentations by Young Generation Spectroscopists from Universities across the UK. Workshops, vendor presentations and two parallel sessions dedicated to Instrumental and Integrated Developments and Informatics concluded the day with ample time to visit the Exhibition area and poster session.

Bob Bateman (Waters Corporation) gave the Tuesday Plenary Lecture on methods of obtaining 'From one to ten million spectra per hour and beyond'. Thereafter followed parallel sessions dedicated to Small Molecules and Interpretation, Post Translational Modification, Food, Protein Analysis, Trapped Ions and Separation Science for MS. The latter session is reviewed in more detail below.

The Tuesday afternoon parallel session, titled "Separation Science for MS", was chaired by Dr John Lough from Sunderland University who is currently president of the Chromatographic Society. The first talk was given by the Dr Tony Bristow (Astra Zeneca) on "Evaluation of a new interface to couple gas chromatography to time of flight – mass spectrometry.

The provision of robust technology to provide accurate mass GC-MS and GC-MS/MS for problem solving activities is an essential tool for impurity identification. A prototype atmospheric pressure chemical ionisation (APCI) source coupled to a Brucker Daltronics microTOF mass spectrometer has been developed that can be interfaced with both GC and LC without major modification. This approach allows both accurate mass LC-MS/MS and GC-MS/MS to be obtained from the same instrument. To test the potential of the new interface, ten potential impurity compounds used commonly in chemical synthesis were analysed using this new interface in GC-TOF-MS mode. Eight of the ten compounds were detected and gave the correct elemental formula when derived from the accurate mass measurement. This provisional data, produced with minimal optimisation, demonstrates the great potential of this technology.

Ole Vorm described fast, simple and automated on-line separation achieved on a split free 1D nano-LC-MS system. The approach was validated using the analysis of mouse placental cell proteome. The 2D approach yielded about 2000 protein identifications whereas 1D analysis gave approximately 600 but avoided the complexity of the 2D methodology.

Dr Gwyn Lord gave a presentation on "Adsorption of compounds onto injectors in LC-MS". Carry-over in quantitative analysis is a common occurrence and can often determine the limit of detection of a particular assay. He described a particular problem in developing an assay for a drug where there was differential adsorption of the analyte with respect to its deuterated analogue internal standard. The degree of adsorption depended on the relative concentrations of the two compounds and was believed to be taking place on the face of the rotar seal in the injector. The solution was, by increasing the relative concentration of the analyte injected by concentrating sample extracts, the adsorption process reached saturation. The relative percentage of carryover to analyte response was thus reduced, minimising the problem to an acceptable level.

Pamela Stoddart (Applied Biosystems) presented on 'Analysis of water for pesticides at low parts per trillion (ppt) levels using two dimensional LC-MS/MS without and sample pre-treatment'. The provision of clean, uncontaminated drinking water is of paramount importance to the water industry. In recent times, the requested limits of detection for pesticides have been decreasing as methodologies improve. Typically water companies need to achieve limits of quantitation for analytes between 0.02 – 1 µg/L (20 – 1000 ppt). To achieve these limits of quantitation, samples require extraction and sample concentration. This sample pre-treatment adds time and cost to the analysis. Pamela presented a method where on-line sample extraction followed by LC-MS/MS using novel

Author Details:

Bernie Monaghan (1)
and Richard Houghton (2)

(1) Separations Science and Spectroscopy
Editor, International Labmate

(2) Principal Scientist,
Drug Development Services, Quotient
Bioresearch, Newmarket, UK

software, allowing a dynamically changing lists of MRM transitions (scheduled MRM), was used to screen for a large number of pesticides in a single injection. Quantitative and qualitative data, in the range of 20 – 1000 ppt, was obtained showing this was a valid approach.

The final talk of the session was given by Richard Houghton (Quotient Bioresearch) on "The application of small particle stationary phases to the analysis of biomarkers by LC-MS/MS". One of the major drivers in clinical and pre-clinical bioanalysis is the reduction in analytical run-time to increase sample throughput. Recent developments in the areas of small particle stationary phases have offered the potential for increased speed and enhanced resolution of chromatographic separations. Richard presented the theory and practical examples of how sub 2 μm particles had been used to successfully develop assays for steroid biomarkers with run-times of under half that of more conventional 3 μm particles. This enabled the accurate and precise quantitation of thousands of samples in a fraction of the time previously required.

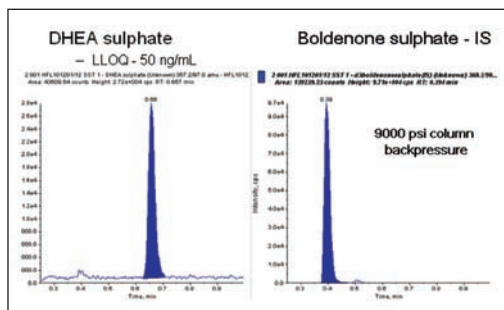


Figure 1. Slide from Richard Houghton's presentation showing a chromatogram of DHEA sulphate using uHPLC-MS/MS.

The final day again started with a plenary lecture this time delivered by Dr Graham Lappin from Xceleron Ltd. on the topic of 'Accelerator Mass Spectrometry (AMS): An unlikely marriage between Archeology and Biomedical Science'. AMS is an extremely sensitive isotope ratio method being capable of measuring a few atoms of ^{14}C , putting its sensitivity into the zeptomole range (10^{-21}). Its applicability to metabolic research was discussed and examples presented.



Figure 2. Exhibitors and Attendees at the wine reception.

Parallel session on Omics, Clinical and Toxicology and Imaging MS followed before the final presentation, the Chair Plenary Lecture delivered by Prof Alan Marshall from Florida State University. He covered the topic of 'Reading Chemical "Fine Print": The Key to Exploiting Nature's Isotopic Complexity'.

By utilising Fourier Transform ion cyclotron resonance mass spectrometry (FT-ICR-MS), high mass resolving power ($m/\Delta m_{50\%} > 400,000$ over a wide mass range) offers two advantages. First, it becomes possible to separate complex mixtures without prior chromatographic or gel separation. Second, elemental composition may be determined from accurate ($t_0 < 1\text{ppm}$) mass measurement alone for unknown molecules up to $\sim 1,000$ Da. Examples across a wide application area were shown.



Figure 3. L to R, Paul Humphrey (Thermo Scientific), Ann McLaren and Steve Lennon (both Peak Scientific Instruments).

EXHIBITION

As with previous BMSS the Exhibition area was well cited and offered a focal point for the attendees during the coffee breaks being adjacent to the Poster sessions. A good pointer as to the interaction between delegates and exhibitors was the vendor presentations during the Monday afternoon, which were generally well received by novice and experienced users alike.

The exhibition area was managed by Dave Collison and this year featured 56 companies from the UK and overseas. From Instrument manufacturers through to service providers, CRO organisations and companies offering related services (Chromatography Columns, Solvents etc.) the choice on offer was quite outstanding for the attendees.

Never ones to miss an opportunity some exhibitors and associated companies made the opportunity to support the event even stronger by sponsoring various events and activities.

Activity	Sponsor
Lanyards and Badge Holders	Agilent Technologies
Monday Evening Social Event	Thermo Fisher and Agilent Technologies
Exhibitor Wine Reception	Jaytee Biosciences and the BMSS
Conference Dinner Reception Drink	The University of York
Exhibitor WiFi	The University of York
Barber/Bardoli Prizes (best young person's poster and oral presentation)	
Barber Prize	Pfizer Ltd.
Bordoli Prize	Waters Corporation

Figure 4. Meeting Sponsors.

There will be no BMSS Annual Meeting in the UK in 2009. Instead the Society will be supporting the IMSC which will be held in Bremen, Germany from the 30th August to the 4th September www.imsc-bremen-2009.de

Particle Counters Give Air Pollution Data in Beijing

Sold in Europe by **Copley Scientific**, wide-range particle spectrometers from MSP Corporation are being used to deliver precise, accurate data about air pollution in and around Beijing in conjunction with the Olympic Games.

Ahead of the Olympics, the Hong Kong Polytechnic University and Shandong University, working with Chinese environmental authorities, implemented MSP's Model 1000XP Wide-Range Particle Spectrometers to measure airborne particles. Measurements were made before, and continued during and after the Games in central, upwind and downwind locations around the city.



MSP's M-1000XP Wide-Range Particle Spectrometer is a unique particle-measuring instrument that uses laser light scattering, differential mobility analysis and condensation particle counting to measure and sort aerosol particles as small as 10 nanometers into 120 different size channels. Ten nanometers is the size of the smallest known virus, or roughly 1/10,000th the thickness of a human hair. Researchers will use these data to learn about particle formation and growth in different locations in Beijing.

The Wide-Range Particle Spectrometer was developed by a team of scientists and engineers at MSP, an aerosol/particle technology company based in Shoreview, MN. The instrument is used by researchers worldwide for highly precise and accurate airborne particle measurement in air pollution studies and laboratory research.

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Mid Infrared Fibre ATR Probes for Process Spectroscopy



Wavelength Range	2-18 μm
ATR Element	Diamond, ZnSe
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Suitable for the analysis of liquids, powders, polymers or tissue.



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www.fibrephotonics.com info@fibrephotonics.com

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Contact Gwyneth Astles on
+44 (0)1727 855574
or email:
gwyneth@intlabbmate.com