

Chromatography Focus

THE CHROMATOGRAPHIC SOCIETY'S SPRING SYMPOSIUM



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The Chromatographic Society recently held its Spring Symposium and AGM in Milton Keynes, hosted by Shimadzu UK, in their newly completed laboratories.

With the Chromatographic Society celebrating half a century of supporting chromatography and chromatographers and Shimadzu celebrating 50 years of business in gas chromatography, Shimadzu's laboratories made an appropriate venue for the symposium. In addition, attendees were able to see the various and significant technical achievements made by the company over the fifty years which were incorporated into a wall display at Shimadzu with space for future advancements.

The symposium was divided into two sessions before and after a buffet lunch and exhibition.

The morning session was introduced by Dr. Chris Bevan, President of the Society who emphasised that the symposium had something for everyone, dealing with the future of sample preparation, detection and data processing before moving onto one of the days highlights in awarding Dr. Kevin D. Altria of Pharmaceutical Development, GlaxoSmithKline R&D Harlow, the Society's Silver Jubilee Medal for his outstanding contribution to the development, application and popularisation of capillary electrophoresis. The award was given in the presence of Dr Altria's mother who proudly accompanied her son for the occasion. *[Further biographical details of Dr Altria's achievements are given on the Chromatographic Society's website www.chromsoc.com]*

Addressing the audience, Dr. Bevan stated that the presentation could so easily have gone wrong. He had checked the medal the night before and discovered that the sterling silver had become tarnished during storage and was black rather than silver! Panic ensued and with no silver polish in the house a propriety brass cleaner was used to achieve the desired effect!

Following his award Dr Altria gave an elegant presentation introducing us to the application of micro-emulsions in HPLC and micro emulsion electrokinetic chromatography (MEEKC).

Micro-emulsion droplets of nanometer size were prepared as oil in water or water in oil. Generic MEEKC procedures using oil in water had application for the separation of acidic, basic and neutral drugs and steroids and were particularly useful for creams and ointments which could be solubilised and analysed successfully by the technique. For GSK marketed compounds significant savings could be made using MEEKC compared with HPLC. For nicotine and related impurities better selectivity could be achieved than with HPLC. For competitor

analysis, generic MEEKC was able to achieve separations in 30 seconds leading to high sample throughput. For water in oil MEEKC, reverse migration occurred, compared to oil in water – with the procedure being particularly useful for ointments.

Dr Altria concluded his presentation by discussing micro-emulsion HPLC (MELC) a very recent technique having about ten published papers to date. MELC in isocratic and gradient form was proving useful for parabens analysis and compounds in suppositories and creams. The next presentation by Dr Stephen Smith from the University of the West of England dealt with the rapid diagnosis of Campylobacter infection using SPME followed by GCMS, and volatile organic compound (VOC) analysis as a potential diagnostic tool for cancer detection?

Dr Smith initially discussed disease diagnosis for the agricultural / food industries; especially for stored foodstuff susceptible to fungal and bacterial attack, which have both major health and economic impact. For example, diagnosis of soft rot in potatoes, where vapour profiles of healthy potatoes obtained by GCMS were compared with profiles obtained from potatoes inoculated with bacteria.

Dr Smith then discussed the importance of developing a rapid VOC analysis for the early detection and rapid diagnosis of gastrointestinal disease and diarrhoea, particularly for developing and poorer counties. VOC from healthy faeces were compared with those from diseased faeces, extracting the VOC using SPME followed by GCMS profiling. Headspace volatiles analysis took around 20 minutes compared with the more traditional extraction procedures which took two hours. A number of statistical techniques were used to compare results. Finally it was hoped that techniques under development will have application in the early detection and diagnosis of colorectal cancer.

Jon Curtis of GSK gave a presentation illustrated with a number of short videos dealing with high frequency sonics in sample preparation and management using adaptive focused acoustics. High frequency sonics,

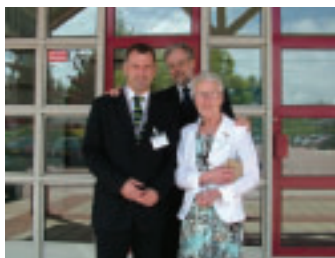
200-800 kHz, can be focused on an area of 3-4 mm² within a sample preparation cell to solubilise samples such as animal tissue and other samples previously considered insoluble in short time periods of ca 20 seconds at room temperature. The technique can be used on a wide range of samples, in either batch or continuous flow mode without heat or aerosol generation or compound degradation – major advantages over conventional ultrasonics. Adaptive focused acoustics is easy to use, robust and can be

automated for integration into automated sample analysis procedures. The technique has found application with RNA, muscle, heart and brain tissue and with the solubilisation of emulsions and lubricants. Covaris Inc. manufacture commercial kit for adaptive focused acoustics which is marketed in Europe by K Biosciences. Lunch and exhibition with ESA, Thames Restek, Aitken Scientific and K Biosciences exhibiting was followed by the Chromatographic Society's AGM attended by the Society's members.

The afternoon session was initiated by Dr Peter Myers of Xtec who's presentation on 'Sample Preparation and Chromatography without Pressure Pumps' discussed recent work at the universities of Leeds and York, partially sponsored by Waters on miniaturisation and portable chromatographic devices. Chips that had grooves were difficult to seal on top and provided difficulties with interfacing and scaling, leading to the development of a polycarbonate chip - a 1536 well sample plate in CD format. Centrifugal force combined with surface tension provided the transport mechanism whilst valves made of sacrificial wax guided solvent / solute direction. Elegant presentations on 'Radio-Chromatography in Positron Emission Tomography (PET) Research' by Adam McMahon and on 'Gonadotropin Glyco-Fingerprinting' by Ray Iles followed, with the final presentation being given by Ian Mutton of GSK Stevenage, 'Towards Generic Quantitation Protocols - Initial Experiences with the Corona CAD'

The object of the research was to obtain qualitative and quantitative confirmation of target compounds and their impurities in terms of yield and purity. Quantitation proved difficult as response factors for compounds were unknown and the number of reference standards limited at the time of first synthesis. Further significant difficulties included quantity of sample available – possibly 100 µg in 10 µl, together with impurities such as water, salts, solvents, catalysts, silica and vacuum grease and the requirement for high sample throughput -100,000s samples run per annum.

If calibration wasn't possible, how was quantitation possible at the Discovery stage? What detector? What calibrants, and how could accuracy be obtained as 'fit for purpose?' The Corona CAD appeared a suitable universal detector. However, it was necessary to evaluate the effect of the matrix composition on CAD response, to target compounds, using single and multiple calibration procedures. Comparing NMR, ELSD and CAD and correlating the results with true known values for 12 test compounds results obtained were NMR $r^2 = 0.97$, ELSD $r^2 = 0.62$ and CAD $r^2 = 0.96$, from which it was concluded that ELSD was unreliable for the given requirements and that CAD compared well with NMR but requires further evaluation.



Silver Jubilee medal winner Dr Kevin Altria with his mother and Society president Dr Chris Bevan

Separation Science / Spectroscopy Meetings Calendar 2006/2007

Meeting	Venue	Date	Contact
26th International Symposium on Chromatography	Copenhagen, Denmark	21-25th August	www.isc06.dk
17th International Mass Spectrometry Conference	Prague, Czech Republic	27 August - 1st September	www.imsc2006.org
Festival of Science	Norwich, UK	2-9th September	www.the-ba.net
Desty Memorial Lecture for Innovation in Separation Sciences	London, UK	20th September	www.desty.info
50th Golden Triad Symposium -Chromatographic Society	Charnwood, UK	11th October	www.chromsoc.com
50th Golden Triad Symposium -Chromatographic Society	Sandwich, UK	24-25th October	www.chromsoc.com
23rd Montreux Symposium on LC-MS	Montreux, Switzerland	8-10th November	www.iaec.ch
50th Golden Triad Symposium -Chromatographic Society	Stevenage, UK	21-22nd November	www.chromsoc.com
2007			
58th Pittsburgh Conference	Chicago, USA	25th Feb-1st March	www.pittcon.org
55th ASMS Conference	Indianapolis, USA	3-7th June	www.asms.org
17th International Reid BioAnalytical Forum	Guildford, UK	9-12th June	www.chromsoc.com
HPLC 2007	Ghent, Belgium	17-21st June	www.hplc2007.org
4th International Peptide Symposium "From Discovery to Therapeutics"	Cairns, Australia	21 - 26th October	www.peptideoz.org

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