

# Gas Chromatography at the 26th International Symposium on Capillary Chromatography and Electrophoresis

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**This month in "GC Connections," John Hinshaw files a report of the 26th International Symposium on Capillary Chromatography and Electrophoresis, which was held in May.**

Almost 350 scientists, researchers, students and industry representatives attended the 26th International Symposium on Capillary Chromatography and Electrophoresis — ISCCE 2003 — 18–22 May 2003 in Las Vegas, Nevada, USA. Returning to Las Vegas after two years, the symposium again was a clear success. The registration list included a wide sampling of European, American and Asian attendees, despite the last-minute cancellation of several foreign representatives from severe acute respiratory syndrome (SARS)-affected areas.

The meeting followed a format similar to past capillary symposium meetings in the United States. This year 85 lectures (in two parallel sessions) were presented concurrently with 169 posters. A conference workshop and a users' group session about gas chromatography  $\times$  gas chromatography (GC  $\times$  GC), each with more than 40 attendees still present at 10 pm, as well as sponsor seminars and an evening reception, rounded out the symposium activities. While the symposium sessions were underway in the hotel conference area upstairs, the casino and restaurants downstairs were in full swing. As I walked past the myriad slot machines, blackjack tables and roulette wheels, I became convinced that many of the same people were still in the casino two years after the first ISCCE symposium held in this location. Several chromatographers reported better luck this year, perhaps because they had honed their gambling skills in anticipation of their return.

## The Golay Award and Other Honours

2003 marks the 15th consecutive year that the Marcel Golay Award in Capillary Chromatography has been presented. First given in 1989 at the 10th International Symposium on Capillary Chromatography in Riva del Garda, Italy, and since then at each of the subsequent symposia in the United States, Japan and Italy, the Golay Award honours the inventor of capillary chromatography. The 2003 Golay Award, sponsored by PerkinElmer Instruments (Shelton, Connecticut, USA), was shared by three recipients — Andreas Manz (Imperial College, London, UK), D. Jed Harrison (University of Alberta, Edmonton, Alberta, Canada) and J. Michael Ramsey (Oak Ridge National Laboratories, Oak Ridge, Tennessee, USA) — each of whom are active in microsized separations science. Manz was recognized for his pioneering contributions in the field of micro-total analytical systems ( $\mu$ TAS), including microchip separations, fast and hyphenated separations, and information flow in the chemical and biochemical environment. Harrison's activities include original work in microchip separations, chemical microinstrumentation, and fabrication and material science as applied to biological and biochemical areas such as enzyme digestion, protein and cell manipulations, diagnostics and sensor technology. Ramsey, a past chair of the American Chemical Society's analytical chemistry division, has been instrumental in the development of microfluidics as applied

to capillary electrophoresis (CE) and related electrodriven liquid separations, laboratory-on-a-chip applications, and chemical and biochemical reactions for the acquisition of biomolecular information on proteins, deoxyribonucleic acid (DNA), peptides and cellular components.

A special session was held on Wednesday morning to honour Walter Jennings (University of California–Davis, USA) on the occasion of his 80th birthday. Jennings became involved with flavour and fragrance analysis early in the development of GC and subsequently co-founded J&W Scientific, a capillary column manufacturing company that now is part of Agilent Technologies.

## Gas-Phase Separations

The main conference sessions and the posters were split into parallel concurrent sessions: one about gas-phase separations and the other about liquid-phase separations. Of the 85 oral sessions, 33 (39%) concerned gas-phase separations and of those, 17 (52%) addressed comprehensive two-dimensional (2D) GC. Yet of the 169 posters, 101 (59%) dealt with aspects of gas-phase chromatography. Interestingly, all except two of the sponsor seminar topics were about gas-phase separations equipment and columns. Of necessity, this "GC Connections" column is limited to the gas-phase sessions. The choices of which items to include in this column are entirely my own and don't reflect upon the validity or significance of the work presented at the symposium.

The main focus of papers about gas chromatography continues to be comprehensive 2D GC (or GC × GC), as has been the case at other recent meetings. Other topics included selective sampling and sample preparation, column technology, high-speed separations, miniaturization, detectors and some other sample-specific applications.

## Citing recent government movements toward regulation of endocrine-disrupting compounds, Frank David discussed the need for rapid sample turnaround, multiresidue analyses and lower detection limits.

### Extraction and sample preparation:

After the Golay Award presentations and lectures by each recipient, the main programme's gas-phase itinerary began with several talks about extraction and sample preparation. Pat Sandra (Research Institute for Chromatography, Kortrijk, Belgium) opened by presenting his work in sorptive sample extraction from both liquid and gaseous samples, in which a relatively thick coating of absorptive material, such as polydimethylsiloxane (PDMS) or 100–300 µm o.d. particles of higher molecular weight siloxane gum, strongly retains sample analytes *in situ* until subsequent thermal or solvent desorption and analysis. The technique has been refined in recent years and applied to areas such as underground parking garages, fire and arson investigation, sick building syndrome, and endocrine disruptors, pesticides and plasticizers in urban environments.

Johan Roeraade (Royal Institute of Technology, Stockholm, Sweden) discussed techniques for thick-film trapping and release such as phase and cryofocusing on open-tubular traps coated with 100 µm films. He noted that a uniform film, as required for high chromatographic separation fidelity, is not necessarily desirable in such traps, and that the capacity of the traps increases rapidly with larger diameters if the phase ratio ( $\beta$ ) is held constant.

Abdul Malik (University of South Florida, Tampa, Florida, USA) demonstrated the use of sol-gel stationary phases made from zirconium and titanium oxides instead of silicon oxides for sample extraction and concentration. He described some unique selectivities and stable chemical properties.

Jan C.R. Demyttenaere and co-workers (Ghent University, Ghent, Belgium) compared classic solvent extraction techniques with solid-phase microextraction (SPME) of natural products. Maria I. Melecchi and

co-workers (Federal University of Rio Grande [UFRGS], Porto Alegre, Brazil) authored several posters about the uses of SPME for various environmental and biological analysis challenges.

Araceli Peña and co-workers (National University of Autonomous Mexico [UNAM], Mexico City) reported on their work with SPME to characterize natural products.

Claudia A. Zini (University of Waterloo, Ontario, Canada) presented several posters about sample preparation with SPME, supercritical fluid extraction (SFE), activated carbon and ion-exchange resins. I should note that numerous co-authors from various institutes and companies — including Federal University of Rio Grande, Klabin Riocell SA (Guaíba, Brazil), Zoex Corp. (Lincoln, Nebraska, USA), Eurofins (Galten, Denmark), Integrated Regional University of Alto Uruguai and Missões (URI, Santo Ângelo, Brazil), and Federal Center of Technological Education Paraná (CFET, Curitiba, Brazil) — also participated in these works.

Frank David (Research Institute for Chromatography) later spoke about the new analytical challenges that recent crises have created in food and environmental chemistry. Citing recent government movements toward regulation of endocrine-disrupting compounds, he discussed the need for rapid sample turnaround, multiresidue analyses and lower detection limits. Miniaturized sample preparation techniques use less solvent to produce faster results, but many laboratories are inhibited by older, entrenched methodologies. David suggested that many food residues can be analysed by a four-method hierarchy classified according to fat content and whether the analytes can be separated by GC or liquid chromatography (LC).

**Comprehensive 2D GC:** Arguably the most significant recent development in GC, comprehensive 2D GC has undergone a number of advances in the past year, although mainstream recognition remains elusive. Lectures and posters at the symposium revealed that the technique is beginning to find acceptance outside research laboratories. Barriers to broader acceptance remain in place, however, and these

hindrances include the cost of equipment, training of technical personnel and the lack of a perception that GC × GC offers unique and indispensable analytical information. At this year's symposium, though, many papers concentrated on specific application areas, as opposed to purely instrumental work or general applications.

Table 1 lists the application areas that were the focus of presentations about 2D GC. The applications fall into two general categories: complex multicomponent analyses that benefit from the additional second-dimensional information that GC × GC provides and applications that benefit from its greatly increased selectivity. The selective applications could be performed by more conventional heart-cut multidimensional analyses if one or a few closely related compounds were concerned, but the ability of 2D GC to deliver, in effect, hundreds of rapid sequential heart-cut runs makes it a very useful tool when multiple target compounds occupy significantly different sections of a primary chromatogram. Simpler mass spectrometry (MS) detection and selected-ion monitoring can provide a similar degree of selectivity in many instances, but these techniques are often better coupled with a heart-cut system, especially for isomeric and other related compounds that differ little in their fragmentation patterns or principal ions. Demands for scanning speed in a 2D GC system have driven the configuration of comprehensive systems to include time-of-flight (TOF) MS detection.

Several interesting developments in the hardware area also were presented. Paolo Magni (Thermo Finnigan, Milan, Italy) showed applications of large-volume splitless injection to trace analysis using 2D GC. John V. Seeley (Oakland University, Rochester, Michigan, USA) presented his latest work on obtaining GC × GC chromatograms without a thermal modulator by using a differential flow scheme instead. Alistair C. Lewis (University of Leeds, Leeds, UK) provided information about the effects of valve-based modulation on peak shapes. Mohamed Adahchour (Vrije Universiteit, Brussels, Belgium) discussed the use of 50 µm i.d. secondary GC columns for faster GC × GC analysis. Several papers about the data handling and presentation of GC × GC chromatograms were presented, as well.

In related work, H.-G. Janssen (Unilever Research and Development, Vlaardingingen, The Netherlands) discussed the coupling of LC columns with GC separations. These couplings are not new, and they can offer

an even greater orthogonality between the primary (LC) and secondary (GC) separations than does GC × GC. In particular, Janssen presented recent work with high-speed GC separations of LC fractions. By using rapid secondary GC column temperature programming rates as high as 200 °C/min, he could separate volatile components in a single LC fraction in less than 5 min. However, as many as four fractions per LC peak could be required for a comprehensive separation. The resulting 20 min time span for separating the fractions of a single LC peak exceeds many total LC run times, so a stopped-flow LC system would be required if full automation were to be realized. Janssen showed several LC × GC data sets, obtained with manual LC fraction transfer, that demonstrated the potential of such systems for the analysis of triglycerides.

**Miniaturized devices and high-speed analysis:** Miniaturized GC analysers were the topic of several presentations that certainly gained the audience's notice.

Richard D. Sacks (University of Michigan, Ann Arbor, Michigan, USA) updated the audience with his group's ongoing project to develop a wristwatch-sized, self-contained GC analysis system that would include sampling, separation, detection and wireless communications stages. Previously limited to larger-scale demonstrations of this principle, Sacks showed some chromatograms, both isothermal and temperature-programmed, from a micro-GC prototype column and discussed ways to produce stacked arrays of 100 µm i.d. micromachined columns with a circular cross-section. He discussed a chemical-sensor detector array with alkane–thiol bonded colloidal gold particles and showed plans for a preconcentrator device, as well as a prototype of an integrated assembly of the individual components.

An earlier talk co-authored and presented by Frank L. Dorman (Restek Corp., Bellefonte, Pennsylvania, USA), related recent developments in a series-column peak displacement technique that Sacks intends to use in the wristwatch GC system. Essentially, the technique uses pressure balancing to temporarily stop flow in the first column while continuing second-column flow, thereby increasing the separation between peaks stopped on the first column and those already traversing the second column. This technique provides a limited ability to displace coeluted peaks into adjacent unused areas in the chromatogram.

Mark Libardoni (University of Michigan, Ann Arbor, Michigan, USA) presented a related technique termed the band acceleration device (BAD) in which thermal ramping of part of a tandem-column pair changes the overall selectivity of a system in a controlled and predictable manner.

Curtis Tinker (Bristol-Myers Squibb Co., New York, USA) presented results obtained for residual solvents with a prototype dual-channel µ-ChemLab GC system from Sandia National Laboratories (Albuquerque, New Mexico, USA). The instrument has a preconcentrator; a sol-gel coated 98 cm × 100 µm, 400 µm d<sub>r</sub> rectangular cross-section column; and a surface-acoustic-wave (SAW) detector. Som Mitra (New Jersey Institute of Technology, Newark, New Jersey, USA) showed a microconcentrator also based on sol-gel technology.

In the fast GC area, Jim Luong (Dow Chemical, Fort Saskatchewan, Alberta, Canada) updated the audience with the

latest results from a high-performance pressurized liquid injection system coupled with a high-speed GC.

George A. Reiner (International Flavors and Fragrances, Hazlet, New Jersey, USA) related his experiences in the migration of high-speed GC methodology from the research laboratory into production. He used method translation software to develop a new method using 100 µm i.d. capillary columns that was 3.5-fold faster than the existing method. Well-suited for the existing high sample load in the production laboratory, the new method was challenged by the difficulties in obtaining the nonstandard 16 m × 100 µm, 0.16 µm d<sub>r</sub> columns called for by the method translation process, as well as by difficulties matching stationary-phase polarities — measured by an ester retention-index scale (IE) — with the existing columns. On methylsilicone columns, the new method matched the old

**Table 1: Application areas for comprehensive 2D GC at the 26th International Symposium on Capillary Chromatography and Electrophoresis.**

Application	Principal Author	Affiliation
Allergens in fragrances	Alain Chaintreau	Firmenich (Geneva, Switzerland)
Chemical warfare agents	Edward B. Ledford Jr.	Zoex Corp. (Lincoln, Nebraska, USA)
Dioxins and PCBs* in food	Jacob de Boer	Netherlands Institute for Fisheries Research (Ijmuiden, The Netherlands)
Essential oils	Tincuta Veriotti	Leco (Las Vegas, Nevada, USA)
Food analysis	Luigi Mondello	University of Messina (Messina, Italy)
Forensics	Glenn S. Frysinger	US Coast Guard Academy (New London, Connecticut, USA)
Genetically modified plant material	Jack Cochran	Leco
Organic air pollutants	Alastair C. Lewis	University of Leeds (Leeds, UK)
PCB analysis	René J.J. Vreuls	Vrije Universiteit (Brussels, Belgium)
Pesticides in fruits and vegetables	Jack Cochran	Leco
Petroleum hydrocarbons in coastal marsh sediments	Christopher M. Reddy	Woods Hole Oceanographic Institute (Woods Hole, Massachusetts, USA)
Polyaromatic hydrocarbons	James B. McQuaid	University of Leeds
Pyrolysis	Hernan J. Cortes	Dow Chemical Co. (Midland, Michigan, USA)
Residual solvents in pharmaceuticals	Jean-Marie D. Dimandja	Spelman College (Atlanta, Georgia, USA)
Wine flavour and aromas	Jack Cochran	Leco

\*PCBs = polychlorinated biphenyls.

IE values to within 61.5%, with nearly the same resolution. Suitable polar columns were not obtainable, however. Ultimately, Reiner believes that the new rapid method will displace the older, slow method as laboratories' internal customers experience the increased throughput and the results' equivalency.

Luigi Mondello (University of Messina, Messina, Italy) presented two posters about ultrafast GC — one about essential oil composition and the other about the analysis of lipids — that show the potential of capillary GC in the fast lane.

## Conclusion

It is impossible to mention all of the GC work presented at this symposium. Many authors related their results in column, detector and other hardware areas that aren't mentioned in this column. Suffice it to say that GC continues to demonstrate its viability and continues to evolve toward new capabilities. Advances in comprehensive 2D GC abound, and the technique is poised for mainstream acceptance as researchers apply it to a range of application areas. High-speed GC and miniaturization go hand-in-hand and offer great potential. I'm not sure that we'll be wearing wristwatch GC systems anytime soon, but the side benefits of pursuing such a goal only can enhance the field. The combination of specific sampling methodology with the extremely high resolving power of multidimensional GC and GC-MS will continue to yield significant advances for years to come.

Next year, the International Symposium on Capillary Chromatography and Electrophoresis returns to Riva del Garda, Italy, 31 May-4 June 2004. I'm looking forward to seeing how much further the researchers and contributors will advance the science of chromatography.

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For an on-going discussion of GC issues with John Hinshaw and other chromatographers, visit the Chromatography Forum discussion group at <http://www.chromforum.com>