



## Connections

In this month's installment, John Hinshaw reports about the 25th International Symposium on Capillary Chromatography and Electrophoresis, which was held in May.

# Gas Chromatography at the 25th International Symposium on Capillary Chromatography

The 25th International Symposium on Capillary Chromatography and Electrophoresis convened in Riva del Garda, Italy, 13–17 May 2002. This symposium was the Silver Jubilee meeting of the ongoing symposium series that has met in Europe, Japan, and the United States. Next year the symposium returns to Las Vegas, Nevada, 18–22 May 2003. More than 600 scientists, students, and commercial representatives traveled from Europe, the United States, the Near East, Australia, and the Far East to attend this year's meeting. Two well-attended preconference workshops about comprehensive separations and liquid chromatography–mass spectrometry (LC–MS) were held. The meeting itself comprised 39 plenary lectures, 21 shorter keynote lectures that featured younger researchers, and more than 400 poster session contributions, which followed the format of the preceding symposia held in Europe with an emphasis on poster presentations. Several discussion seminars and sponsor lectures rounded out the program. Topics ranged from gas chromatogra-

phy (GC) to bioapplications and from sampling techniques to chromatography fundamentals, as listed in Tables I and II. As if that weren't enough, social programs that included a classical music concert, a disco party, and several receptions kept attendees busy in the evenings. Despite this after-hours activity, interest in comprehensive GC  $\times$  GC and GC injection was so high that the scheduled Thursday evening discussions, which ran until after 9 P.M., were packed as well.

With such a large number of presentations about such a comprehensive list of topics, it was impossible for one person to attend all the sessions or to view all the posters in detail. For reasons of time and space limitations — and not due to any personal opinion of their significance — many are not mentioned in this column. This report concentrates almost exclusively on GC activities and leaves mention of further areas to others who will report about the symposium. As a compliment to the organizers, the schedule allowed enough time between activities to visit the city of Riva del Garda briefly (see photograph), collect some souvenirs, and observe how this popular European tourist location has expanded since the first of the 11 international symposia at this location was held 19 years ago.

**Table I: Techniques presented at the 25th International Symposium on Capillary Chromatography**

Techniques
Fundamentals
Microcolumn separations
Capillary GC
(Micro) LC
Comprehensive techniques
Sample preparation and sampling systems
Trace analysis
Column technology
Supercritical fluid chromatography and extraction
Electromigration techniques
Instrumentation and automation
Coupled and multidimensional techniques

**Table II: Applications presented at the 25th International Symposium on Capillary Chromatography**

Applications
Environmental applications
Energy, petrochemical, and industrial applications
Biomedical and pharmaceutical applications
Analysis of natural products, food, flavors, and fragrances

### Awards and Recognition

Several awards and recognitions were conferred at this year's symposium. On Tuesday morning, session 1 of the symposium was dedicated to the memory of Arnaldo Liberti (1917–2001), who founded the Italian Chemical Society's Division of Analytical Chemistry and was very active in environmental analysis in Italy. The 2002 M.J.E. Golay Award, sponsored by PerkinElmer Instruments (Shelton, Connecticut), was presented to Keith D. Bartle (University of Leeds, United Kingdom) for his work in GC and to Stellan Hjertén (University of Uppsala, Sweden) for his research in ion separations.

On Tuesday afternoon, the 2001–2002 Martin Medals, sponsored by the Chromatographic Society of the United Kingdom, were presented to J. Michael Ramsey (Oak Ridge National Laboratory, Oak Ridge, Tennessee) for his work in miniaturized separation systems and to Paul R. Haddad (University of Tasmania, Hobart, Australia) for his contributions to electrochromatography and electrophoresis.

The opening session on Wednesday morning was dedicated to Leslie S. Ettre (Yale University, New Haven, Connecticut) on the occasion of his 80th birthday. Ettre presented some of his insights into the inception and evolution of chromatography and its associated instrumentation during the past 50 years.

### GC Technology

GC technology continues to progress toward higher speed, higher resolution, and broader application scope. This year's symposium saw sustained interest in comprehensive GC × GC separations, membrane and related sampling techniques, and high-

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speed GC. As a relatively mature discipline, GC generally doesn't spawn breakthrough technologies, but several participants presented creative ideas and concepts that have the potential to boost GC's analytical portfolio in the future.

### Sampling and sample preparation:

Overall, microextraction techniques dominated the presentations in the sampling area, including membrane extraction, solid-phase microextraction (SPME), and more-conventional headspace and bulk extraction techniques. T. Hyötyläinen, T. Tuutijärvi, K. Kuosmanen, and M.-L. Riekkola (University of Helsinki, Finland) presented their work with microporous membrane liquid-liquid extraction for GC analysis of pesticides in wine. J.C.R. Demyttenaere, J.I. Sánchez Martínez, M.J. Téllez Valdés, R. Verhé, and P. Sandra (University of Ghent, Belgium) compared dynamic headspace and SPME for the analysis of ester flavor components and found that SPME

provided higher overall sensitivity and reproducibility.

Jim Luong (Dow Chemical Canada Inc., Fort Saskatchewan, Alberta, Canada) presented a microvolume-injection system for capillary GC of pressurized fluids that combines a liquid injection valve with a conventional split-splitless inlet. K. Jinno (Toyohashi University of Technology, Japan) showed a new microfiber-packed capillary that can be used for sample concentration by SPME or as GC, capillary electrophoresis (CE), or LC columns. A. Malik (University of South Florida, Tampa, Florida) introduced some sol-gel materials, both in capillary and monolithic form, that provide more-rapid desorption



The Apollonia Tower on the Piazza III Novembre, Riva del Garda, Italy. Photography by John V. Hinshaw.

for high-boiling materials compared with conventional SPME extraction materials and that exhibit better stability toward solvent exposure.

Minhee Kim and Somenath Mitra (New Jersey Institute of Technology, Newark) presented a microfabricated microconcentrator that uses ohmic heating of a silicon

## Comprehensive separation techniques took the GC front stage again this year.

chip substrate to desorb semivolatile components from a polymer layer inside a micromachined passageway. They also characterized its operation in conjunction with a gas membrane or extractor for aqueous volatile organic compounds. Several authors reported their results with stir-bar sorptive sampling, which uses analyte absorption on a polymer-coated magnetic stir bar followed by thermal desorption into a GC system.

**Comprehensive separations:** Comprehensive separation techniques took the GC front stage again this year. The term *comprehensive* refers to instrumental methods that pass all of a sample through two different columns in such a way that all solutes experience the separating power of both columns as a series of orthogonal chromatograms and thereby benefit from a vastly increased resolving power. This method contrasts with conventional multidimensional chromatography in which only part of the sample, or the entire sample in continuum, experiences the resolving power of both columns only as a single combined chromatogram. In comprehensive separations, sequential solute fractions are trapped as they are eluted from the first column and then released into the second column. The technique is analogous to manually collecting separate fractions from one column across the entire sample and then reinjecting them one at a time into a second column. A separate chromatogram is recorded for each fraction and then a composite or convolute representation is constructed by combining the individual chromatograms. Although conceptually simple, in practice the technique uses many creative high-speed instrumental implementations.

Presenters described various column modulation systems for this purpose, and they fall into two broad groups: those that use thermal modulation and those that use a valve-switching arrangement to manipulate the flow of solutes from one column to another. In thermal modulation, a section of column tubing is rapidly and alternately heated and cooled so that material from the first column is collected for a short time and then released onward to the second column. Some devices use a moving heated zone that cycles across the trapping section of a column, and others direct jets of cooled or heated gas across the area. Groups from Dow Chemical Co. (Freeport, Texas, and Midland, Michigan), Dow Chemical Canada, and RMIT University (Melbourne, Victoria, Australia) presented posters and an oral session about a longitudinally modulated cryogenic system for GC  $\times$  GC as applied to polymer pyrolysis and various industrial samples. Valve-switching arrangements enable the modulation process by mechanical means using a high-speed multiple-port valve or a combination of two or more discrete valves. Gerard Rutten and Carel Cramers (Eindhoven University of Technology, The Netherlands) showed a multiple-port valve system with two sample loops that alternately hold and release effluent from one column into a second. Jacqueline F. Hamilton and Alastair C. Lewis (University of Leeds) described a valved system that permits independent carrier-gas pressure control of the second column.

Comprehensive GC  $\times$  GC generates large amounts of data, as much as one chromatogram per second from the second column. When combined with concomitant high-speed mass spectral scans, the resulting data sets can be quite large. Jens Dallüge, Leo van Stee, Jan Beens, and Udo A.Th. Brinkman (Vrije University, Amsterdam, The Netherlands) examined the use of target compounds and elevated peak thresholds to limit the amount of data acquired and diminish the amount of processing required to reduce the data to qualitative and quantitative results. Valentijn van Mispelaar and Albert Tas (TNO Nutrition, Zeist, The Netherlands), Peter Schoenmakers (University of Amsterdam, The Netherlands), and Hans-Gerd Jansen (Unilever Research, Vlaardingen, The Netherlands) discussed the application of multivariate analysis to the challenges of GC  $\times$  GC data handling.

Several authors discussed applications of GC  $\times$  GC. R.A. Shellie (RMIT Univer-

sity) discussed his group's experience with the characterization of flavors and fragrances, including the use of an enantioselective column to elucidate the stereoisomerism of these materials. M. Harju (Umeå University, Sweden) demonstrated the separation of polychlorinated biphenyls (PCBs) and also showed how data from two conventional one-dimensional separations can predict a GC  $\times$  GC result. L.L.P. van Stee (Vrije University) gave the results from his examination of the selectivity obtained with different column combinations in the GC  $\times$  GC analysis of various household chemicals.

The theoretical resolving power of comprehensive systems is the product of that which is available on either column separately. As Phillip Marriott (RMIT University) pointed out, however, even without a general theory of comprehensive separations, a little effort toward optimizing GC  $\times$  GC results could far outstrip conventional methodology. Indeed, it is the richness and complexity of GC  $\times$  GC information that engenders new interpretive techniques in information science. Leonid Blumberg (Fast GC Consulting, Hockessin, Delaware) proposed several new metrics to help characterize and understand GC  $\times$  GC separations. S.E. Reichenbach (University of Nebraska, Lincoln) presented numerical techniques for removing background images in GC  $\times$  GC and introduced to many in the audience the term *blob* in reference to the appearance of GC  $\times$  GC peaks when plotted in two dimensions.

### GC Applications

The large number of GC applications presentations demonstrated again the wide range of analytical challenges to which chemists can apply GC. Just a few of the papers are discussed here.

In the environmental area, water, air, sediments, and soils were the subjects of multiple contributions. For example, L. Elflein, E. Berger-Preis, and K. Levsen (Fraunhofer Institute of Toxicology and Aerosol Research, Hannover, Germany) and G. Wünsch (University of Hannover, Germany) presented methodology for the analysis of common household pesticides in air, with minimum detectable levels ranging from 0.5 pg to 52 pg. I. Viden and V. Janda (Institute of Chemical Technology, Prague, Czech Republic) discussed SPME of dimethylpolysulfides in water. Patricia Schossler, Fabiane Trombetta, Priscila Schütz, and Elina Bastos Caramão



(Instituto de Química, UFRGS, Brazil) used ultrasonic extraction and GC–MS to analyze polyaromatic hydrocarbons (PAH) in lagoon sediments. I.N. Glazkov, A.A. Martynov, E.D. Virus, and I.A. Revelsky (Lomonosov Moscow State University, Russia) used supercritical fluid extraction (SFE) to analyze semivolatile organic compounds, such as PAH and PCBs, in soil.

In the energy, petroleum, and industrial application area, G. Castello, M. Perata, and L. Gardella (University of Genova, Italy) examined the presence of volatile organic compounds in car exhaust that lead to the formation of ozone (ozone precursors). N.P. Kulshreshtha and S.C. Moldoveanu (Brown & Williamson Tobacco Corp., Macon, Georgia) presented a detailed poster about the quantitative analysis of pyridines in tobacco smoke.

Elena I. Savel'eva and Andrew S. Radilov (Institute of Hygiene, Occupational Pathology, and Human Ecology, St. Petersburg, Russia) and Igor G. Zenkevich (Chemical Research Institute of St. Petersburg State University, Russia) provided results from their study of GC–MS and retention index–based identification of the by-products of the chemical destruction of

phosphorus-containing VX warfare agents. This paper was one of the most remarkable at the symposium. Looking back at the previous International Symposia on Capillary Chromatography that were held at the same location during the past 15 years, I remember how rare and difficult it was for any scientist from the former Soviet Union to attend. Many managed to do so then, and in 2002 scientists from what is now the Russian republic can present their information freely. This development emphatically points out the ability of chromatography to bring together scientists from diverse backgrounds.

### Conclusion

In addition to the brief descriptions above, numerous GC papers were presented at the 25th International Symposium in the environmental, energy, petrochemical, industrial, pharmaceutical, natural products, food, flavor, and fragrance areas. Even a summary description of those presentations goes far beyond the available space. Perhaps, it is sufficient to say that GC is alive, healthy, and thriving in the beginning of its second 50 years. Comprehensive GC × GC, high-speed GC, microextrac-

tion techniques, and miniaturized instrument systems, along with all the peripheral equipment and devices that enable them, form the basis for the advancement of GC technology. The applications to which they will be applied provide the impetus for ongoing development. I look forward to May 2003 when the next International Symposium on Capillary Chromatography will provide a new snapshot of the state of chromatography.

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

