

Process Spectroscopy Anchors the 31st FACSS Conference

Through symposia, posters, and workshops, representatives from industry and academia addressed a broad range of technology and implementation issues that are beginning to change the landscape of modern spectroscopy.

Michael MacRae

The 31st meeting of the Federation of Analytical Chemistry and Spectroscopy Societies (FACSS) took place October 3–7, 2004, in Portland, Oregon. At the outset of the conference week, nearby Mount St. Helens began belching steam and ash for the first time since 1986, charging the FACSS atmosphere with anticipation of a major eruption.

The restless mountain provided an apt metaphor for another significant force being felt during FACSS. Process analytical technology, like the noted volcano, also has been gathering strength since the mid-1980s and is expected to alter the future terrain of the spectroscopy world significantly.

This article briefly touches upon some of the highlights of the FACSS program, focusing specifically on techniques and applications for process monitoring and related measurements in the industrial environment. One major factor behind the current momentum of process spectroscopy is FDA's Process Analytical Technology (PAT) initiative, which is discussed later. As the pharmaceutical industry considers the ramifications of the PAT effort, the spectroscopy community is making changes of its own to prepare for increased opportunities in pharma and other regulated industries. The growing prominence of PAT — in fact, the very use of this novel nomenclature — at a meeting considered by many to be the leading indicator of spectroscopy's state of the art is one sign of this development.

Of course, FACSS is a very broad-based meeting with a scientific program that extends well beyond process analysis. For a more detailed view of the meeting, visit the organization's Web site at www.facss.org.

A New Type of Spectroscopy Gathers Steam

Although analytical instrumentation had been employed for years in on-line or field-based industrial applications, the demand for ever-more challenging measurements has given rise during the past few decades to new and more sophisticated analytical solutions. Enabling technologies such as fiberoptic sampling and powerful chemometrics software began to emerge in the 1980s, bringing the power of exotic laboratory techniques to the process line, and catalyzing process spectroscopy into its own specialized field of research and technology transfer.

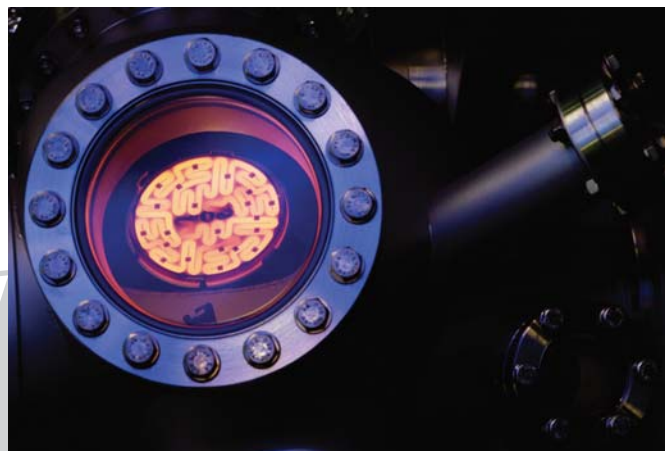


IMAGE COURTESY OF PHOTODISC

Advances in process analytical chemistry have been steady ever since. Early signs of this growth include the advent of focused academic/industrial research initiatives such as the University of Washington's Center for Process Analytical Chemistry (CPAC), which marked its 20th anniversary in 2004, and topical conferences such as the International Forum on Process Analytical Chemistry (IFPAC). As a result, popular spectroscopy-community meetings such as FACSS began to make room for process-related sessions in conference programs traditionally devoted to fundamental studies in techniques and instrumentation.

Despite all this activity in process analytical chemistry, many would argue that the process industries largely have failed to take full advantage of the available technologies. A gulf still exists between the scientists who design the measurement instruments and the process engineers who must do the measuring. Particularly in well-established production industries where basic analyzer technology has been entrenched for many decades, new technologies are often resisted as too risky, unnecessarily complex or overly expensive. Changes in one key industry, however, can overcome these barriers.

The US pharmaceutical industry, with its giant research and quality-control budgets, has taken major steps toward embracing advanced real-time analytical methods. FDA launched its PAT initiative in 2001, at last signifying a willing-

ness not only to recognize newer analytical techniques but also to provide standards and guidance for their implementation. Following FDA's lead, the US Pharmacopeia and the ASTM also have organized committees to create measurement standards and educate the industry in the use of new measurement technologies to reduce production cycle times, reduce waste and error, increase safety, and manage product quality.

Although PAT extends well beyond spectroscopy and chemical measurement to include physical, microbiological, mathematical and risk analyses, the initiative creates new opportunities for spectroscopists to take part in the scientifically rich world of pharmaceutical process control.

Program Highlights

Building on this momentum, PAT was featured prominently throughout the FACSS program. Along with workshops, oral presentations, and poster sessions dedicated to process topics,

process-related talks infiltrated other sessions focused on specific techniques or instrumentation groups. The following summary merely provides a flavor of the range of topics covered at the conference; readers again are referred to www.facss.org for more detailed program and speaker information.

Oral and poster presentations.

A Monday session on PAT in the pharmaceutical industry featured application-oriented talks focused on NIR for particle size distribution determinations, IR and NIR for fluid-bed drying process monitoring, and Raman and ATR-based FT-IR monitoring of polymorphic transformations in acetaminophen. The session also featured more general talks by speakers from companies such as Pfizer, AstraZeneca and GlaxoSmithKline discussing how they have implemented mid-IR, chemometrics, and other approaches.

Another Monday session discussed advances in miniature spectroscopic sensors. A mix of speakers from academia, government laboratories, and the

instrument industry presented the latest achievements in shrinking conventional lab instrumentation for use in previously unimagined production situations.

On Thursday, a session titled "State of the Art Infrastructure for PAT Spectroscopy in Industry" focused more on the delicate implementation issues in process analysis than on the technologies themselves. Speakers addressed the need to develop productive partnerships within the plant, and to effectively integrate new measurement approaches with other key manufacturing elements. A second session on Thursday reviewed some of the up-and-coming technologies being adapted for process applications. These included a portable integrated UV resonance fluorescence and Raman chemical sensor, a micro-NMR system, a portable surface plasmon resonance instrument, a vapochromic system for detecting and quantifying relative humidity and organic solvent vapors, a system for RF/microwave dielectric spectroscopy, and the application of modular sampling systems.

A Wednesday session focusing on Raman brought together speakers who shared their results from using Raman techniques to analyze dyed fibers, fat composition in high-protein whey emulsions, and nuclear waste. Other speakers talked about Raman instrumentation optimized for forestry and environmental monitoring, as well as for potential use in future Mars explorations.

This year's Charles Mann Award for contributions to applied Raman spectroscopy went to Michael Carrabba, principal scientist for Hach Homeland Security Technologies and a long-time advocate for nontraditional uses of Raman in the field and in process settings. The Mann Award symposium featured several talks that demonstrated Raman's growing acceptance as a process-monitoring tool in a range of industries such as nuclear waste management, semiconductors, and environmental engineering.

The chemometricians convened for a Tuesday afternoon session on pharmaceutical process applications. Speakers addressed the modeling of batch processes with in situ spectroscopic and

calorimetric measurements; case studies in the development of chemometrics tools for PAT; process development for active pharmaceutical ingredients using in-line FT-IR and Raman spectroscopy; and the challenges to FT-NIR transmission spectroscopy posed by low-drug-content tablets.

Numerous process-based posters were presented throughout the week at FACSS, including a dedicated poster session on emerging technologies in process analysis on Thursday. Among the novel techniques presented, a group from the Lund Institute Technology and AstraZeneca presented a poster titled “Non-Invasive Determination of Porosity in Solids by Diode Laser Oxygen Spectroscopy: Application to Pharmaceutical Tablets.”

Workshops. Several FACSS workshops also gave attendees the opportunity for in-depth discussion of key topics. “Process Analytical for Faculty,” offered by Lynn Melton of the University of Texas, Dallas, attempted to coach instructors on ways to introduce

process concepts into the analytical chemistry curriculum. The workshop aimed to help instructors with no industrial experience explain how familiar laboratory technologies are adapted for the process environment, and how economics play an important role in a company’s decision to adopt a process analytical technology. Other process-focused workshops included “Process Analytical Chemistry: Out of the Lab and Into the Pipe,” taught by Christian Hassell of Los Alamos National Lab and James Rydzak of GlaxoSmithKline; “Chemometrics Without Equations (or Hardly Any) — Hands On,” taught by Barry M. Wise of Eigenvector Research; and “FDA Guidelines and Expectations for Validation of PAT Methods,” taught by Emil W. Ciurczak of Integrated Tech Solutions.

Conclusion

The strong showing of process topics at this year’s meeting does not mean that FACSS is shifting away from its

traditional structure as a broad-based analytical chemistry meeting. Rather, it has begun to update its format so that important application areas such as PAT, proteomics, nanotechnology, biodefense, and so forth are discussed in thematic sessions more accessible to their interdisciplinary audiences. PAT is clearly one of those areas where people from diverse backgrounds must come together and collaborate to solve scientific and engineering problems that otherwise would go unchallenged. This summary of PAT at the 2004 FACSS conference documents just one example of how the traditional spectroscopy community is preparing for the eruption of opportunities that process analytical technologies might yet create. ■

Michael MacRae, former editor of *Spectroscopy*, lives in Portland, OR, and writes about science and technology. E-mail: msmacrae@yahoo.com.