In the news

Noise-free spectroscopy cleans up images

A ''reverse'' approach to spectroscopy can clean up images by eliminating background noise, according to researchers at the MESA+ Institute for Nanotechnology, University of Twente, The Netherlands [1].

Rather than starting with the laser beam, the trick by Herman L. Offerhaus and colleagues was to take the molecule under study as the starting point. This radical ''reversal'' led to a relatively simple modification of conventional coherent anti-Stokes Raman scattering (CARS) spectroscopy, which delivered better images (Fig. 1).

CARS was already a powerful technique, which used lasers to visualize molecules for such purposes as food testing and medical imaging. One advantage of CARS is that no fluorescent labels are needed to make the molecules visible. However, background noise complicates the task of interpreting the resultant images.

This new approach eliminates such noise completely, leaving only the ''real'' image. More information than ever before, such as accurate details of the concentration of the substance, can be obtained using this technique. It is easier to detect the signature of the molecule in question.

The key to side-stepping the overwhelming complexity involved lay in the exhortation by Professor Shaul Mukamel's exhortation to ''Look at the molecule!'' (Prof. Mukamel, who holds a post at the University of California, USA, collaborated on the publication [1]). So the Twente researchers did not focus on the way that light interacted with the molecule, as this made it very difficult – even impossible – ''to separate the wheat from the chaff'' and reveal the real image. Instead, they started by examining the energy levels inside the molecule. They translated this theory into the new technique of vibrational molecular interferometry (VMI), which they predict will vastly expand the uses of CARS and other techniques.

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Reference

[1] E.T. Garbacik, J.P. Korterik, C. Otto, S. Mukamel, J.L. Herek, H.L. Offerhaus, Phys. Rev. Lett.107 (2011) 253902.

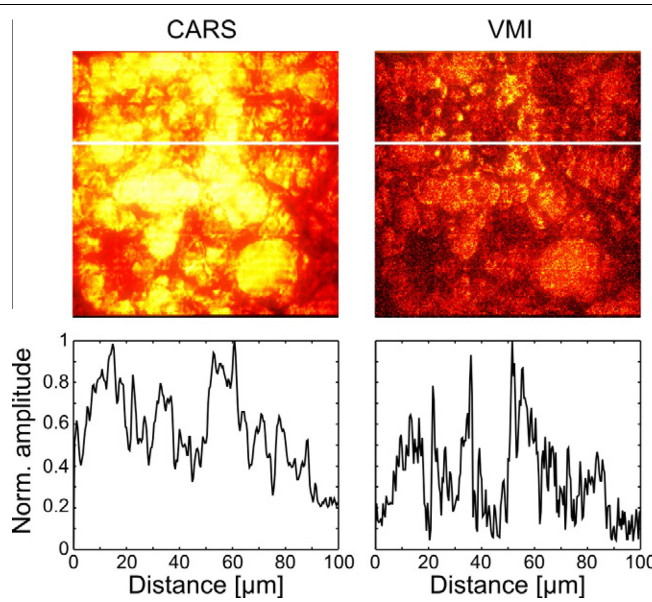

Figure 1. Images of mayonnaise using (left) coherent anti-Stokes Raman scattering (CARS) spectroscopy, and (right) the new vibrational molecular interferometry (VMI). Below, the intensity along the white line in the picture is given. Using VMI, the background signal vanishes.

0165-9936/$ - see front matter doi:10.1016/j.trac.2012.01.002

LGC Forensics finds new evidence that convicts two killers

Thanks to their painstaking, meticulous scientific work in preparing new evidence, LGC Forensics helped convict two of the racist killers of Stephen Lawrence (Fig. 2) – David Norris and Gary Dobson – whom the jury found guilty in early January – over 17 years after the murder in London.

Figure 2. Stephen Lawrence 1974–93.

After the verdict, Mr. Justice Treacy, the trial judge, sentenced Norris to 14 years and Dobson to 15 years – the prison terms reflecting their ages at the time of the murder, when both were juveniles, and the sentencing guidelines at the time. However, Attorney General Dominic Grieve was reviewing the sentences after a request from a member of the public, who complained that the jail terms were too lenient.

Led by Roy Green, the LGC Forensics team started to re-examine the entire case in 2006, after the Metropolitan Police asked them to review the scientific aspects in the 1993 murder.

They initially examined Lawrence's jacket for traces of paint that may have transferred from a scaffold pole. During this examination, they saw red fibers that could have come from Lawrence's polo shirt, so the team pursued the possibility of these having been transferred to the

Figure 3. LGC Forensics analysts work within in the high-sensitivity DNA-profiling laboratory (Credit: Andrew Brookes).

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   Trends Anal. Chem. 22 (2003) 685.

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   Trends Anal. Chem. 30 (2011) 1598.

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    by A. Zgola-Grzeskowiak and T. Grzeskowiak
    Trends Anal. Chem. 30 (2011) 1382.

*In the 90 days before 9 January 2012

attackers. Tapings from the suspects' clothing, taken at the time of the initial investigation, were examined. The same red fibers were found on Dobson's jacket and Norris's sweatshirt.

This key finding led the team to search for fibers from other items of Lawrence's clothing that may have transferred to his attackers. The painstaking search revealed fibers, thought to be from Lawrence's jacket, on Dobson's jacket and cardigan, and fibers, which could have come from Lawrence's trousers, on Norris's sweatshirt. At the same time, short, cut human head hairs were noted on the tapings of both Lawrence's and the suspects' clothing, which initiated another line of investigation. This culminated in the finding of a hair in debris from Norris's jeans that matched Lawrence's DNA profile (Fig. 3).

Whilst using a micro-spectrophotometer to analyze the precise color of fibers, the LGC Forensics team identified an additional component that indicated blood on one of the fibers found on Dobson's jacket. Then, later on, during a search of the jacket's original Police packaging for fibers, blood fragments were found, including one with two blue fibers running through it that matched Lawrence's cardigan. The blood in this and one other fragment selected for testing matched that of Lawrence.

This led the LGC team to believe that blood may have been present on Dobson's jacket, after all, so they carried out a minutely detailed study of the jacket using a microscope, magnifying to 40x normal size. A blood stain was found on the collar of Dobson's jacket. Its DNA matched that of Lawrence. This was the key breakthrough for the LGC Forensics team. Their combination of experience, approach and advanced technology had produced crucial evidence lacking in the 1993 investigation.

The LGC Forensics team spent a great deal of time examining exactly how the blood got onto the clothing fibers, and this ruled out the possibility that it may have been caused by contamination of the evidence, as argued in court by the defense.

''I'm extremely proud of the work that LGC's forensic scientists did on this case,'' said LGC Forensics Managing Director Steve Allen. ''Persistence, meticulous science and innovation can help convict criminals years after they committed the crime. This case shows that the key to successful forensics is to assume nothing – which is all the more important in historic cases like the murder of Stephen Lawrence.

''LGC has an extraordinary record of success – largely because we look for evidence that may not have been the object of the original search. We keep an open mind at all times,'' he said.

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PET pinpoints Alzheimer disease

Positron emission tomography (PET) that can detect pathophysiologic change in the brain holds great promise for diagnostic assessment of patients with Alzheimer disease (AD) and dementia [1].

Researchers reviewed numerous PET studies to evaluate a molecular imaging technique that combines PET, which provides functional images of biological processes, with an injected biomarker called 18F-FDG to pinpoint key areas of metabolic decline in the brain that indicate dementia (Fig. 4). Having physiological evidence of neurodegenerative disease by imaging patients with PET could give clinicians the information they need to make more accurate diagnoses earlier than ever before.

In their review of the recent literature since 2000, Professor Nicolaas Bohnen of the Department of Radiology, University of Michigan, USA, and colleagues demonstrated that the evidence for 18F-FDG PET in assessment of dementia has increased with new studies that include autopsy confirmation, wide-diagnostic-spectrum recruitment in primary care settings, historical and prospective cohort studies, and multicenter data analyses.

''The new data support the role of 18F-FDG PET as an effective addition to other diagnostic methods used to assess patients with symptoms of dementia,'' said Prof. Bohnen. ''The review also identified new literature showing the benefit of this imaging technique not only helping to diagnose dementia but also for improving physician confidence when diagnosing a patient with dementia. This process can be difficult for physicians, especially when evaluating younger patients or those who have subtle signs of disease.

''For the first time, imaging biomarkers of Alzheimer's disease are included in the newly revised clinical diagnostic criteria for the disease,'' said Prof. Bohnen. ''This is a major shift in disease definition, as previously an Alzheimer's diagnosis was based mainly on a process of evaluating patients to exclude possible trauma, hemorrhage, tumor or metabolic disorder. Now it is becoming a process of inclusion based on biomarker evidence from molecular imaging.''

The PET biomarker 18F-FDG comprises a radionuclide combined with fluorodeoxyglucose (FDG), which mimics glucose in the body. Cells metabolize FDG as fuel, and the variation in this uptake by cells throughout the body can then be imaged to detect a range of

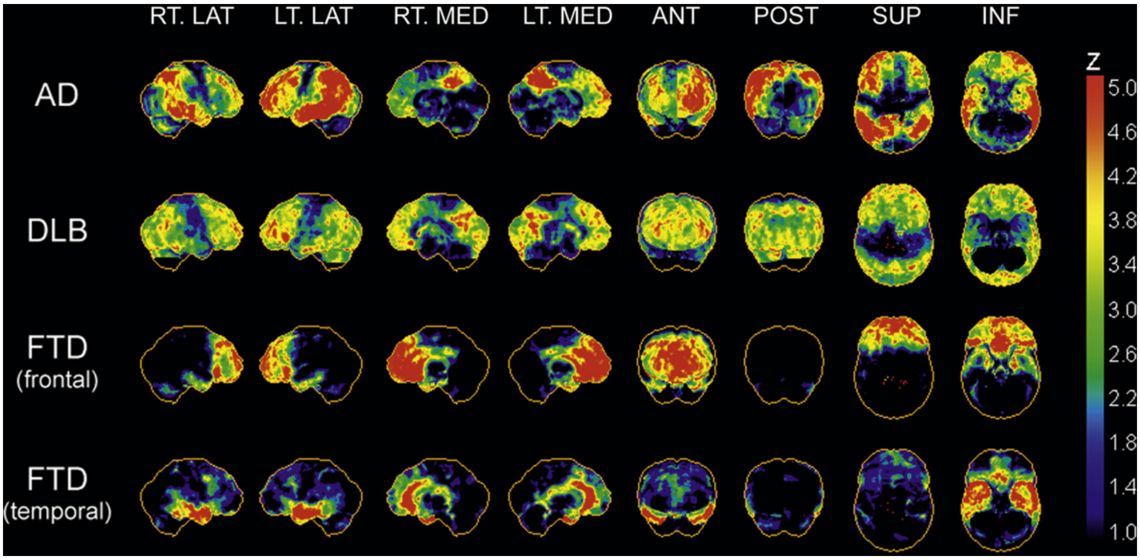

Figure 4. Typical regional cerebral 18F-FDG hypometabolism patterns in AD, DLB, and frontal and temporal FTD. Patterns are presented as z-score maps based on significantly hypometabolic voxels relative to non-demented comparison population. AD pattern of glucose hypometabolism involves predominantly temporoparietal association cortices and posterior cingulate and precuneus cortices. In advanced disease, prefrontal association cortices show additional hypometabolism. Primary sensorimotor and visual neocortices are relatively spared. DLB has cortical hypometabolism similar to that of AD but with additional involvement of occipital cortex. FTD demonstrates frontal lobar or frontal and temporal polar cortical hypometabolism with relative sparing of parietal association cortex and preservation of primary somatomotor and visual cortices. ANT 5 anterior; INF 5 inferior; LAT 5 lateral; MED 5 medial; POST 5 posterior; SUP 5 superior [1].

abnormalities. In the case of dementia, marked reductions in the metabolism of different lobes of the cerebral cortex can confirm that the patient has the disorder. Physicians can tell AD from other dementias, depending on the specific cortices affected.

"Using 18F-FDG PET in the evaluation of patients with dementia can improve diagnostic accuracy and lead to earlier treatment and better patient care," said Prof. Bohnen. "The earlier we make a diagnosis, the more we can alleviate uncertainty and suffering for patients and their families."

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[1] N.I. Bohnen, D.S.W. Djang, K. Herholz, Y. Anzai, S. Minoshima, J. Nucl. Med. 53 (2012) 59.

SAMMI sees through materials

A millimeter-wave sensor can see through non-transparent materials without harmful radiation, according to researchers at Fraunhofer Institute for High Frequency Physics and Radar Techniques (FHR), Wachtberg, Germany.

The Stand Alone MilliMeter wave Imager (SAMMI) (Fig. 5) can see through all non-transparent materials.

"The system detects wooden splinters lurking in diapers, air pockets in plastics, breaks in bars of marzipan, and foreign bodies in foodstuffs," said Helmut Essen, head of the FHR's millimeter-wave radar and high-frequency sensors department. "It can even detect and monitor the dehydration process in plants and how severely they have been stressed by drought."

This makes the scanner extremely versatile – it is suitable for industrial product and quality control and analyzing materials in the laboratory. Because the system can detect dangerous substances (e.g., explosive powder hidden in letters), it can protect vulnerable people (e.g., politicians or freight handlers).

SAMMI's most striking feature is its ability to pick out the smallest differences in materials that are invisible to X-rays. SAMMI can differentiate between different fillings of chocolates, or rubber composites that have similar or identical absorption qualities.

Inside the system housing, there are transmitting and receiving antennae on each of two opposing rotating plates. A conveyor belt transports the sample – perhaps a package whose contents are unknown – between the antennae, while these send electromagnetic waves at 78 GHz. Different areas of the sample absorb the signal to different degrees, leading the varying material composition across a sample to show up in distinguishable contrast.

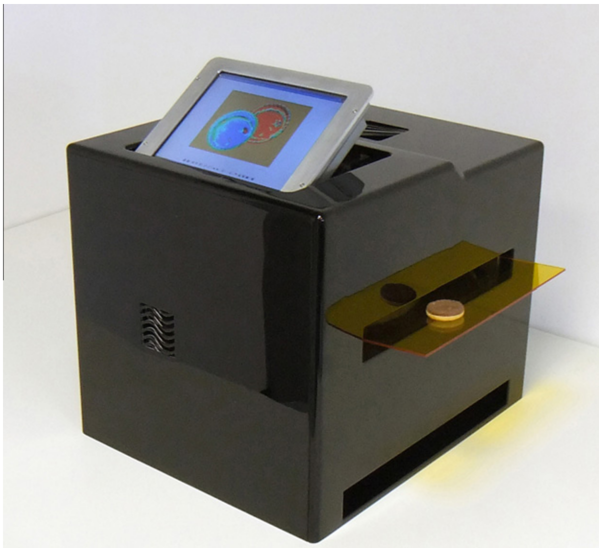

Figure 5. Stand Alone MilliMeter wave Imager (SAMMI).

''Basically, we examine the scanned objects for dissimilarities,'' explained Essen.

The content of the sample appears in real time on the scanner's fold-out display. If the package contains a knife, even the grain of the handle is discernible. If the handle is hollow, the millimeter-wave sensor would show that too. The device scans an area of 30 x 30 cm in 1 min.

''Our system can be operated without safety precautions or safety instructions, and, since it weighs just 20 kg, it's eminently portable,'' said Essen. ''It can also be adjusted to various measuring frequencies.''

In future, the researchers aim to upgrade the system for frequencies of 2 THz.

''Then, we'll be in a position not just to detect different structures but also to establish which type of plastic a product is made from. That's not possible at the moment,'' said Essen.

At present, SAMMI is suitable for spot checks only. However, the FHR researchers are adapting it for fast, automatic inspection of goods on industrial assembly lines. They envisage mounting a line of sensors over the conveyor belt, so that in future products can be scanned at up to 6 m/s.

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Fluoroacetate identified directly in water

Excellent recovery and detection of fluoroacetate (FA) can be achieved at sub-ppb concentrations using reagent-free ion chromatography (RFIC) coupled to mass spectrometry (MS) detection [1].

FA is a strong metabolic poison, commonly used as a rodenticide and predacide. Fluoroacetic acid is also an intermediate metabolite of many compounds (e.g., anticancer drugs 5-fluorouracil and fluoroethyl nitrosourea). FA is inexpensive, simple to synthesize, tasteless, and very soluble in water. Due to the high level of toxicity when ingested, and there being no known antidotes, its use has been banned or restricted in many countries. The US Environmental Protection Agency has placed sodium fluoroacetate in Toxicity Category I, indicating the highest degree for acute oral toxicity. Dionex therefore considered it crucial to develop a method for determination of fluoroacetic acid.

Matrix effects that lead to signal suppression were minimized by using a high-capacity column that separated FA from matrix ions. By diverting these matrix ions to waste prior to entering the electrospray-ionization source, low detection levels were achieved. The MSQ Plus spectrometer provided molecular-ion analyte selectivity, and the selected-ion monitoring (SIM) function achieved good low-level quantification.

Reference

[1] Dionex, Direct Determination of Fluoroacetate in Water by IC-MS, Application Note 276 (www.thermoscientific.com/dionex).

Tumor markers sought in breath

Through the Interreg project, Medisen, Tecnalia in Spain is contributing to developing biosensors capable of detecting the presence of tumor markers of lung cancer in exhaled breath.

Patients with lung cancer, treated in the Section of Medical Oncology of the Institute of Onco-Hematology of the Donostia Hospital (IDOH) have collaborated in this project, after the Ethics Committee of the Clinical Research of Euskadi authorized the clinical trials.

Human breath, whether from a healthy or ill person, comprises hundreds of organic compounds (e.g., acetone, methanol, butanol, and hydrocarbons). There is no single specific component in the exhaled breath capable of acting as a biomarker for the diagnosis of lung cancer, but compounds of interest, generally found at 1–20 parts per billion (ppb) in healthy human breath, can be increased 10–100-fold in the breath of sick patients. To detect these changes required development of novel materials.

During the first phase of the project, breath samples were collected by the hospital staff by a breath collecting device (Fig. 6). Detailed analysis of the most representative compounds present in the breath samples was carried out and the compounds required to act as markers for the presence of lung cancer selected.

Organic compounds were analyzed using gas chromatograph/ mass spectrometry (GC/MS), and algorithms used to discriminate and to identify ''healthy'' and ''cancerous'' patterns that provided information for the design of the sensor.

In parallel, Tecnalia developed novel materials for the detection of the selected organic compounds in order to increase the sensitivity of the devices. Participating together with Tecnalia in this project were the Instituto de Tecnologías Químicas Emergentes de La Rioja (Inter-Química) – to design the sensor device – and the University of Perpignan (France) – to test the novel materials.

By diagnosing lung cancer early, the biosensors could increase considerably the chances of survival.

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Figure 6. Breath-sample collection.

Waters Symposium reviews portable, handheld XRF

The 23rd James L. Waters Annual Symposium at Pittcon 2012 is to review ''The Development and Application of Portable Handheld X-Ray Fluorescence Spectrometers''.

To be held on Monday, 12 March, at Orange County Convention Center, Orlando, Florida USA, it will focus primarily on the later generation of products that continue to drive explosive growth in the use of XRF in the field.

The panel of speakers will recognize the pioneers who were critical in the development of this innovative instrumentation:

- Lee Grodzins, AS&E, ''The Birth and Maturation of Handheld XRF Spectrometers'';
- Andrew T. Elllis, Oxford Instruments, ''X-MET HHXRF Analyzers: A Stainless History'';
- Stanislaw Piorek, Thermo Scientific Niton Analyzers, ''The Evolution of Analytical Capabilities of Field Portable and Handheld XRF Analyzers – From Pencil and Slide Rule to Fundamental Parameters Based Algorithms'';
- Alan Huber, Amptek, Inc., ''The Development of Detectors for Handheld XRF''; and,
- Charles Jensen, Moxtek, Inc., ''Performance Improvements in Miniature X-ray Sources''.

Complete speaker biographies are available at www.pittcon.org.

Biosensors control water quality

The Robotics Institute of the University of Valencia, Spain, is coordinating the Hydrobionets project to save 45% of the costs of desalinating water and to reduce the energy consumption in purification facilities by 74%.

Researchers at the University are designing intelligent networks intended to optimize the operation of sewage-treatment plants (STPs) and water-desalination plants (WDPs). The European project, which has a budget of €3.5m ($4.5m) over three years, has the goal of developing, for the first time in the world, an intelligent, interconnected, wireless network of biosensors able to control bacterial activity and to determine the ideal injection of biocides, and so increase the efficiency of these facilities.

The research platform, which has been working since last autumn, is made up of an international multidisciplinary team. Apart from University of Valencia, participants include the National Center of Microelectronics of the Spanish Superior Council of Scientific Research, several Swedish, Hungarian, Greek and British research centers, and the company Acciona Agua.

University Vice-Principal for Research and Scientific Policy, Pedro Carrasco stressed that the research is ''not only pioneering but another sample of knowledge transfer from the University to society, in this case, through improvements in economy and sustainability.''

Hydrobionets director Baltasar Berefull explained that the main goal of this initiative is to fundamentally increase plant productivity and reduce costs, thanks to greater durability of osmosis membranes in the case of the WDPs and bioreactors in the STPs, and thanks to more accurate use of chemical products. Better management of the facilities will be possible by allowing access and visualizing the different processes more efficiently, he added.

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Whisky galore – but is it the real thing?

In support of the global whisky-manufacturing industry, LGC, the UK's National Measurement Institute for chemical and bioanalytical measurement, has produced a reference material to help identify adulterated high-end whiskies.

Adulteration is a major problem for the global drinks industry and, in order to keep control of this issue and to identify adulteration accurately when it occurs, alcohol manufacturers monitor marker substances in their products. It is these marker substances that can be profiled to prove authenticity. Congeners – volatile organic compounds formed during the whisky-fermentation process – are used as marker substances to help distinguish and differentiate between brands. They also dictate the unique flavor of whisky brands.

LGC has produced the reference material specifically for whisky congeners to help manufacturers uphold the protection of their brands, ensure process and quality control, and enhance research and development.

''For manufacturers of high-end spirits to be confident in the quality of their analysis, they need to be able to validate and monitor procedures using reference materials close in matrix composition to the test samples,'' said Gill Holcombe, LGC Head of Reference Material Production. ''This whisky congener reference material does just that, and is yet another weapon in the arsenal against alcohol adulteration.''

This reference material, LGC5100, is available from LGC Standards, which provides products and services to improve measurement in the laboratory.

Contact:
E-mail: askus@lgcstandards.com
Website: http://shop.lgcstandards.com/

Pittcon announces 2012 Awardees

The 2012 Pittsburgh Analytical Chemistry Award is to go to Professor Alan G. Marshall of Florida State University (Fig. 7).

This award acknowledges Prof. Marshall's contributions to the field of analytical chemistry through his continuing development of Fourier transform ion-cyclotron resonance mass spectrometry (FT-ICR-MS). His current research spans FT-ICR instrumentation development, fossil fuels and environmental analysis, and mapping the primary and higher-order structures of biological macromolecules and their complexes.

''It has been wonderful to have helped enable the explosive growth and impact of accurate mass measurement for addressing previously intractable analytical problems, ranging from petroleomics (i.e., prediction of the properties and behavior of crude oil and its products from their detailed organic composition) to mapping of contact

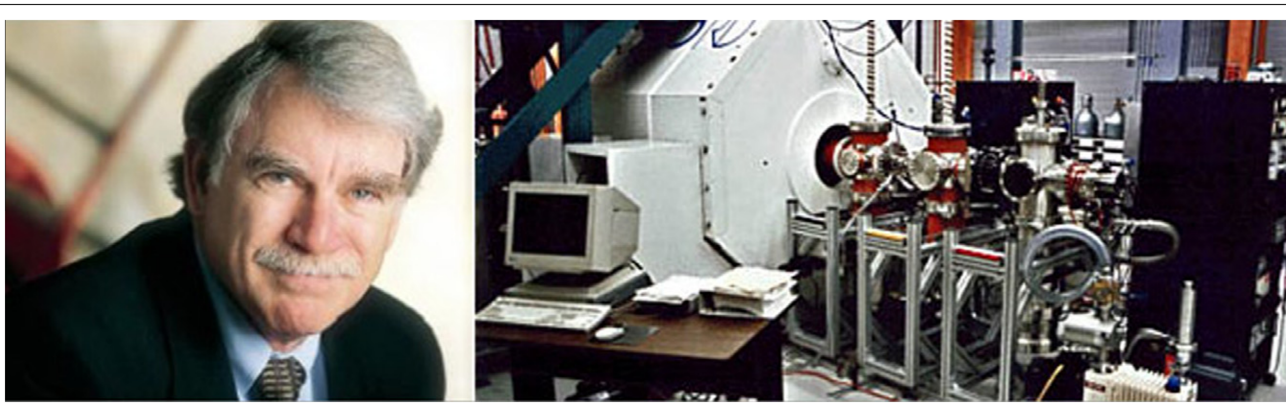

Figure 7. 2012 Pittsburgh Analytical Chemistry Awardee – Professor Alan G. Marshall of Florida State University.

surfaces in protein complexes inaccessible by NMR or X-ray diffraction,'' he said. ''I especially look forward to the role of mass spectrometry in elucidating epigenetics (chemical modification of the genome without change in the nucleotide sequence) as the next grand challenge for biochemistry.''

Prof. Marshall obtained his PhD from Stanford University. Before joining the faculty at FSU, he was a faculty member at the University of British Columbia and at Ohio State University. His recognitions include: Alfred P. Sloan Fellow; American Chemical Society Award in Chemical Instrumentation; Eastern Analytical Symposium Award; American Chemical Society Field-Franklin Award in Mass Spectrometry; Spectroscopy Society of Pittsburgh Maurice F. Hasler Award; New York Society for Applied Spectroscopy Gold Medal; and, the American Society for Mass Spectrometry Distinguished Contribution Award. He is a Fellow of the American Physical Society and the American Association for the Advancement of Science, and is an FSU Distinguished Research Professor.

The Pittcon 2012 Program Committee also announced the recipients of 10 other prestigious awards honoring scientists who have made outstanding contributions to analytical chemistry and applied spectroscopy, as follows:

- Pittsburgh Spectroscopy Award – W.E. Moerner, Stanford University.
- Pittcon Heritage Award, honoring Genzo Shimadzu, Sr. (1839–1894) and Genzo Shimadzu, Jr. (1868–1951), Shimadzu – Shijehiko Hattori, Shimadzu Chairman of the Board, to accept the award.
- Pittsburgh Conference Achievement Award – Christy L. Haynes, University of Minnesota.
- ACS Division of Analytical Chemistry Award for Young Investigators in Separation Science – Jared L. Anderson, University of Toledo.
- Bomem-Michelson Award – Joel M. Harris, University of Utah.
- Dal Nogare Award – Purnendu K. Dasgupta, University of Texas.
- Charles N. Reilley Award – Debra Rolison, Naval Research Laboratory.
- Young Investigator Award – Lane Baker, Indiana University.
- Ralph N. Adams Award – Jonathan V. Sweedler, University of Illinois.
- Williams Wright Award – Richard Crocombe, Thermo Fisher Scientific.

Complete details and biographies are at: www.pittcon.org.