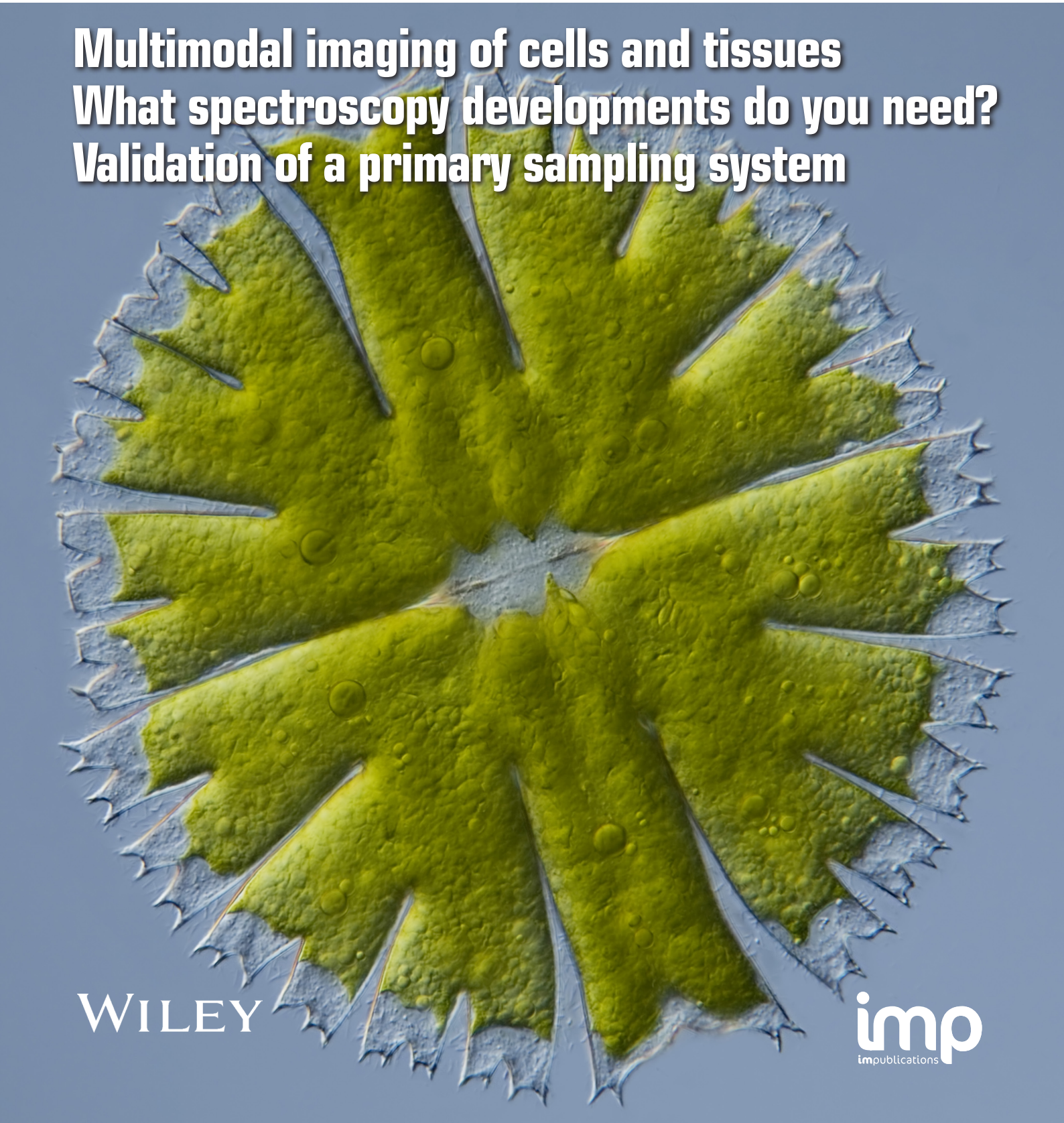
Vol. 29 No. 5 October/November 2017

SPECTROSCOPY europe

Informing European spectroscopists for over 40 years

Multimodal imaging of cells and tissues
What spectroscopy developments do you need?
Validation of a primary sampling system



WILEY

imp
impublications


See Spectroscopy in a New Light
Learn • Experience • Engage
Discover more at www.pittcon.org
PITTCON 2018
CONFERENCE & EXPO
February 26 – March 1, 2018
Orlando, FL USA
Orange County Convention Center

EDITORIAL

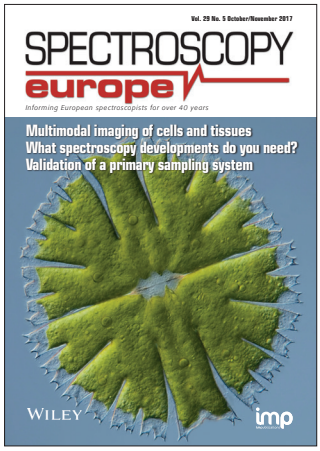
Different spectroscopic techniques have been combined to provide additional and complementary information for decades. Increasingly, this is being expanded beyond just two techniques and may include spatial/imaging information as well. All of which bring their own challenges. In our first article, "Multimodal imaging of cells and tissues: all photons are welcome", David Perez-Guaita, Kamila Kochana Anja Rüther, Phillip Heraud, Guillermo Quintas and Bayden Wood report an example of these new approaches. They look at the use of infrared, Raman and X-ray fluorescence spectroscopies to obtain combined imaging data of whole algal cells and discuss how to overcome the challenges.

Next, in the Tony Davies Column, it is your turn to contribute: to the potential development of new spectroscopic instrumentation! Tony argues that many spectroscopic fields have seen little real development: and there are many reasons for that. Now, you have your chance to let us and, more importantly, the instrument vendors know what innovations (hardware and software) you would like to see in order to help you in your work. There is a short (three-question) survey at https://www.spectroscopyeurope.com/survey; please use this opportunity to help influence the future of the instrumentation you will be using.

In the Sampling Column, Karin Engström and Kim Esbensen detail the "Experimental validation of a primary sampling system for iron ore pellets". This practical article demonstrates the importance of regular validation and bias testing.

The News section covers a diverse range of applications (from cocaine to carrots) and developments (from antimatter to miniature NMR) of spectroscopy.

A Product Focus on Mass Spectrometry is followed by New Products and an extensive Diary of future events to help you plan your 2018 conference schedule.



Micrasterias algae are the subject of the article on multimodal imaging of cells and tissues starting on page 14.

Publisher
Ian Michael
E-mail: ian@impublications.com

Advertising Sales
UK and Ireland
Ian Michael
IM Publications, 6 Charlton Mill, Charlton, Chichester, West Sussex PO18 0HY, United Kingdom. Tel: +44-1243-811334, Fax: +44-1243-811711, E-mail: ian@impublications.com.

Americas
Joe Tomaszewski
John Wiley & Sons Inc.
Tel: +1-908-514-0776
E: jtomaszews@wiley.com

Europe and
the Rest of World
Stephen Parkes
John Wiley & Sons Ltd, The Atrium, Southern Gate, Chichester, West Sussex PO19 8SQ, UK, Tel: +44-1243-770367, Fax: +44-1243-770432, E-mail: stephen.parkes@wiley.com

Paid subscriptions
Journals Subscriptions Department, John Wiley & Sons, Ltd, The Atrium, Southern Gate, Chichester, West Sussex PO19 8SQ, UK.
Tel +44-1243 779777, Fax +44-1243 843232, e-mail: cs-journals@wiley.co.uk

Spectroscopy Europe is a joint publication of John Wiley & Sons Ltd, The Atrium, Southern Gate, Chichester, West Sussex PO19 8SQ, UK, and IM Publications LLP, 6 Charlton Mill, Charlton, Chichester, West Sussex PO18 0HY, UK.

Vol. 29 No. 5
October/November 2017

ISSN 0966-0941

CONTENTS

Spectroscopy Europe is a controlled circulation journal, published seven times a year and available free-of-charge to qualifying individuals in Europe. Others can subscribe at the rate of €152 (Europe), £108 (UK), $208 (ROW, postage included) for the seven issues published in 2016. All paid subscription enquiries should be addressed to: *Spectroscopy Europe*, John Wiley & Sons Ltd, Journals Administration Department, The Atrium, Southern Gate, Chichester, West Sussex PO19 8SQ, UK.

Printed in the UK by Headley Brothers Ltd, Ashford, Kent.

WILEY

imp
impublications

SHIMADZU
Excellence in Science

Tri-Light system through Photomultiplier, PbS and InGaAs detectors

Tri-Light Zone

The UV-VIS-NIR spectrophotometer UV-3600 Plus provides high technology features for liquid and solid samples in a wide variety of fields. With three detectors (PMT, InGaAs, PbS), the UV-3600 Plus is optimized for UV, VIS and NIR spectral ranges.

Highest sensitivity
in the entire wavelength range covering 185 - 3,300 nm

High resolution and ultra-low stray light
through high performance double monochromator system

Multipurpose applications
from basic use to high-end research

Complete range of accessories
enables advanced applications

www.shimadzu.eu/uv-3600-plus

ARTICLE

Multimodal imaging of cells and tissues: all photons are welcome

David Perez-Guaita,[a] Kamila Kochan,[a] Anja Rüther,[a] Phillip Heraud,[a,b] Guillermo Quintas[c] and Bayden Wood[a]

[a]Centre for Biospectroscopy, Monash University, Australia. E-mail: david.perez.guaita@monash.edu
[b]Department of Microbiology and the Biomedical Discovery Institute, Monash University, Australia
[c]Leitat Technological Center, Valencia, Spain

Research groups around the world are studying the spatial location and distribution of molecules within cells using an increasing number of analytical techniques such as infrared (IR), Raman and X-ray fluorescence (XRF) spectroscopy. The information obtained from these techniques in terms of lipids, proteins and the general metabolome is complementary, but commonly the analysis of the data is performed individually on each technique. These three techniques are based on different interactions of the sample with light with different energy and wavelengths, leading to dissimilarities in the spectral features offered by each technique. Table 1 summarises the main features of Raman and IR microspectroscopy, both representing vibrational spectroscopic methods. Raman spectroscopy is a scattering technique, in which energy is transmitted from a photon to a molecule, resulting in a shift in the wavelength of the incident light beam. Fourier transform (FT)-IR spectroscopy, on the other hand, is an absorbance technique where the molecule absorbs a photon and gains energy moving from a lower to a higher vibrational energy state. These methods are complementary in terms of providing molecular information on samples, as molecules or functional groups that tend to be strong Raman scatters are usually weak IR absorbers and *vice versa.* The techniques also complement each other in terms of their advantages and disadvantages for the investigation of biological systems. FT-IR spectroscopy is a non-destructive method with a good signal-to-noise (S/N) ratio and a high efficiency. Raman may lead to a thermal destruction of a cell or tissue due to the high output power of the light source and has a considerably lower S/N ratio, unless the energy of the incident light is close to an electronic transition of the analyte. In that case, resonance Raman enhances the S/N by several orders of magnitude. Surface enhanced Raman spectroscopy (SERS) can also be used for increasing the S/N ratio of Raman spectroscopy. However, as the light source in Raman is a typically a laser with wavelength ranging from the ultraviolet to near IR (240–1064 nm), the achievable spatial resolution is higher depend-

Table 1. Comparison of Raman and IR spectroscopy imaging techniques.

Feature	Raman	Infrared
Origin	Scattering of light: molecular vibrations with changes in the polarisability tensor of a functional group or a molecule	Absorbance of light: molecular vibrations with changes in the dipole moment
Information	Symmetric molecules Non-polar oscillating nuclei	Asymmetric molecules or fucntional groups Polar molecules
Efficiency	Low	High
S/N ratio	Low (unless resonance or SERS is used)	High
Spatial resolution	~0.2–0.7 µm (wavelength-dependent on the incident radiation)	2–10 µm (wavelength-dependent)
Living cells	Good applicability (water is a weak Raman scatterer)	Possible (strong background contributions from water)
Destructiveness	Thermal- and photo-denaturation possible	Non-destructive

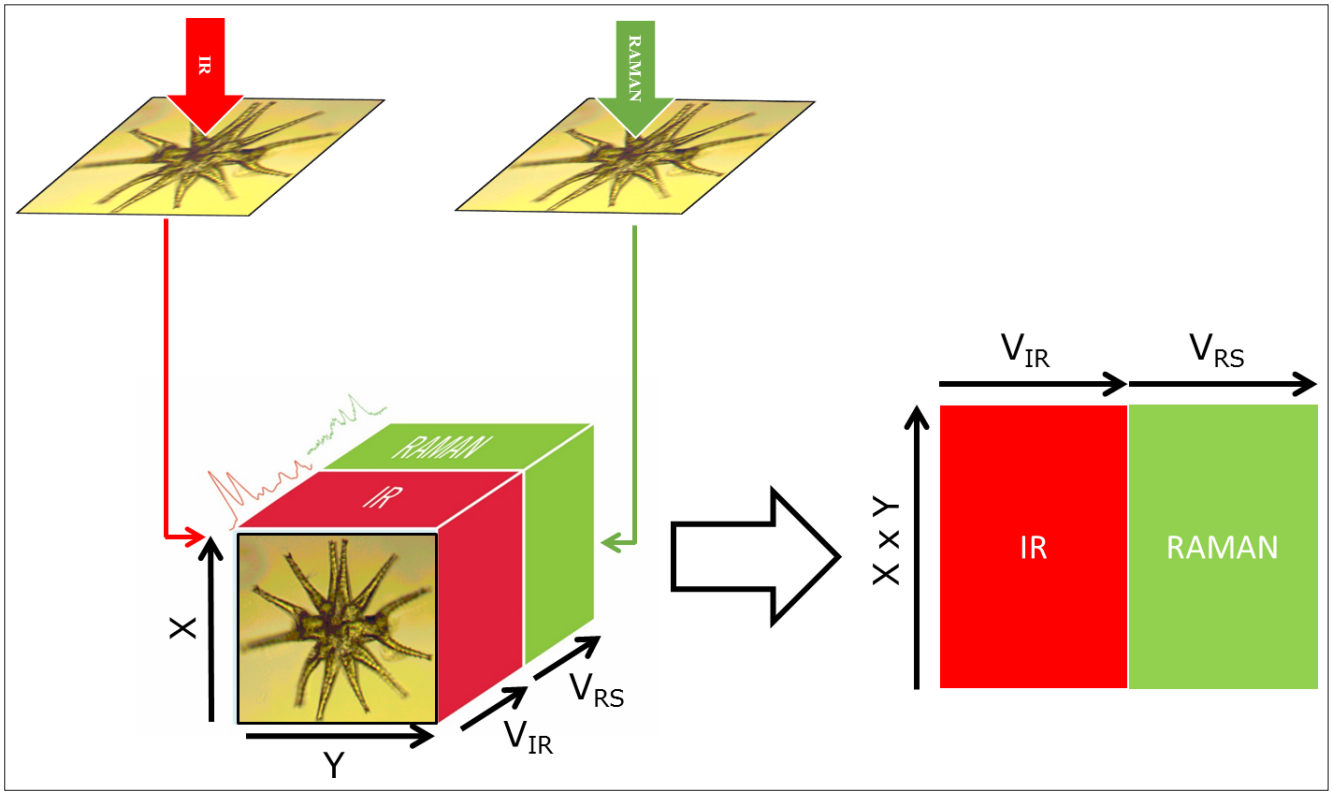


Figure 1. Conceptual scheme of the multimodal imaging of a *Micrasterias* algae, depicting the registration of IR and Raman hyperspectral images for creating an extended data matrix.

ing on the wavelength of the incident photons. Furthermore, as water is a weak Raman scatterer, cells and tissues can be studied using Raman spectroscopy under physiological conditions.

While IR and Raman are very useful in establishing the chemical functional groups of a sample, XRF enables compositional elemental analysis. XRF spectra are obtained by irradiating a sample with X-rays and recording the emitted fluorescence.[1] The complementarity of the information obtained from the three techniques makes their combination extremely powerful in understanding both the molecular and atomic compositions. Biological samples, such as cells or tissues, are complex entities composed of a wide range of chemical compounds including organic and inorganic molecules as well as monoatomic ions. Changes due to an external factor (e.g. inoculation of a drug, radiation or infection by a pathogen) affect the complex network of interactions between the metabolome, proteome and metallome. However, with a single technique only a portion of the molecular phenotype can be studied, thereby neglecting the contributions of the non-detectable analytes, which remain as "dark spots in the whole picture". The biochemical interpretation of these changes is challenging if only individual sections of a phenotype provided by one instrument are analysed and studied independently. The integration of different modalities will enable a holistic comprehension of the biological system under study obtaining correlation between IR (polar/asymmetric molecules), Raman (chromophoric/symmetric molecules) and XRF (elementary composition). In addition, the use of hyperspectral images from different modalities enables spatial correlations based on molecular composition within cells and tissues. Figure 1 depicts the conceptual framework of a multimodal Raman and FT-IR hyperspectral image using a giant algal cell from the genus *Mictrasteria* as the model. In short, a cell or tissue is measured using FT-IR and then Raman with a similar spatial resolution per pixel. Alternatively, if the initial images contain different pixel sizes, pixels can be binned to match the lowest pixel resolution. In our case, IR images were registered to match the Raman image by rotating and/or cropping the image. Image processing registration algorithms such as the ones available in the Image Processing Toolbox™ from Matlab (Mathworks) are very useful in this process. After registration, an augmented data matrix (X, Y, $V_{IR}+V_{RS}$) is obtained, with X and Y being the size of the image, and V_{IR} and V_{RS} the number of variables in the IR and Raman images, respectively. Then, the image can be treated in a similar way to a standard dataset by reshaping the 3D image into a 2D matrix ($X \times Y$, $V_{IR}+V_{RS}$). In a previous study, we pioneered the use of multimodal vibrational (IR and Raman) imaging for the complete study of cells.[2] In this article, we highlight the challenges and advantages on analysing cells through multimodal imaging of cells and we provide two examples performed with algae.

Data analysis and technical challenges

Two main challenges have to be considered for the creation and analysis of multimodal images. First, there are technical impediments on acquiring the images using different modes. Creating a hyperspectral multimodal image containing unique IR, Raman and/or XRF spectra requires i) exactly the same area of the sample to be measured with the different techniques and ii) the use of the same pixel size, overcoming the dissimilarities in special resolution by binning pixels or over- or under-sampling. The selection of substrates that enable the measurement of images through several platforms is a crucial aspect for obtaining successful results. Substrates should be compatible with the different techniques and not present any strong signals, which discards the use of low emissivity slides substrates and regular CaF_2 windows for Raman. Alternatively, silicon wafers and Raman grade CaF_2 windows are suitable substrates for performing Raman, reflection IR and transmission IR measurements, respectively. In addition, it is important to consider a sequence of operations that will ensure that the non-destructive techniques are performed first (e.g. perform FT-IR first). Another pitfall is finding the same cell or tissue section of interest under the microscope, which can be also troublesome under high magnification, and requires the use of flags such as marker points to locate the exact region. The same flags can be used for ensuring that cells are measured in the same spatial orientation, which facilitates the process of registering the images.

The second challenge to overcome is to data mine the combined images to extract meaningful biological information. The lack of analytical data tools for integrating information obtained from different platforms makes the comprehension of complex biological systems a challenge. To analyse single hyperspectral images is a complex issue *per se*, but when different modalities are integrated, the analysis should additionally deal with correlations between the variables from the different spectra. Advances in multimodal chemical imaging technologies and hyphenated analytical systems require new multivariate approaches to extract meaningful data and determine correlations in complex biological systems. Data fusion can be defined as the process of integrating data obtained from different sources. Data acquired from complementary sources can be jointly analysed for studying the relationship between variables obtained from different modalities. This enables a comprehensive understanding of the system, which can lead to an improved molecular phenotyping.[3] Literature shows recent attempts at integrating data provided by different platforms: i) Statistical heterospectroscopy is used for the co-analysis of spectral datasets obtained from different spectroscopic platforms with multiple samples. The methodology performs a covariance map between the spectral dataset measured by the different techniques. This approach has already been employed for the correlation of NMR and IR spectra[4] and NMR and CE spectra. ii) Orthogonal partial least squares (O-PLS and O2-PLS) was used in the field of metabolomics and proteomics to integrate for example data from NMR and MS analytical platforms. iii) Joint and Individual Variation Explained (JIVE) is a method that separates the shared patterns among data sources (i.e. the joint structure) from the individual structure of each data source that is unrelated to the joint structure.[5]

Principal component analysis (PCA) of a hyperspectral image of a whole algae

Figure 2 depicts the PCA of a hyperspectral multimodal image combining IR and Raman spectroscopies. The dataset was created using the procedure explained in Figure 1. Raman and IR hyperspectral images were registered and a PCA was performed over the extended dataset using second derivative and mean centring as the pre-processing steps. Prior to the data fusion, the spectra of the two images were normalised independently using standard normal variate normalisation to eliminate dissimilarities between the ranges of Raman intensity (1–1000 counts) and IR absorbance (0–1 AU) values. Figure 2a shows a 3D image corresponding to the PC1 scores values for each pixel in the hyperspectral multimodal image. It can be seen that PC1 values are not distributed homogeneously along the cell; the centre and arms of the cell show low values whilst the edges of the cell show high values. This distribution evidences that the PC1 captures variability related to differences between the spectra of the cellular wall and the rest of the cell. To gain insight into the changes in the spectra, which are caused by differences of the chemical composition of the cell, the loading vector of PC1 is investigated (see Figure 2a and b). The PCA was performed over the second derivative of the data, so the loading was integrated twice for a better interpretation. It can be seen that some bands are strongly correlated (range 1540–1142 cm^{-1}) for both modalities, which indicates that they correspond to molecules that show absorbance in both Raman and IR. Other bands such as the ones assigned to C=O (1750 cm^{-1}), and Amide I (1650 cm^{-1}) show a strong negative value in the IR spectra, whilst the band assigned to Amide II (1540 cm^{-1}) shows a small negative value in Raman and IR. This indicates that the proteins are concentrated in the regions with a negative value of the PC1 score, i.e. in the centre of the cell, and the edge of the cell is lower in proteins. Interestingly, the region between 1200 cm^{-1} and 900 cm^{-1} presents a derivative shape, being highly positive between 1100 cm^{-1} and 900 cm^{-1} and negative between 1200 cm^{-1} and 1100 cm^{-1}. In this region, several bands including the ones associated with C–O and P–O stretching modes are present, making it difficult to assign them to lipids, phospholipids or carbohydrates. The composition of the edge of the cell, which shows positive PC1 values is related to the positive contribution of the 1100–900 cm^{-1} band, but the position of the band does not give enough information by itself to elucidate its origin. At this point, the multimodal approach can contribute to solve the vague assignment of the IR bands. In the 3050–2700 cm^{-1} region (see Figure 2b), it can be seen that the Raman loadings vector shows a broad negative band

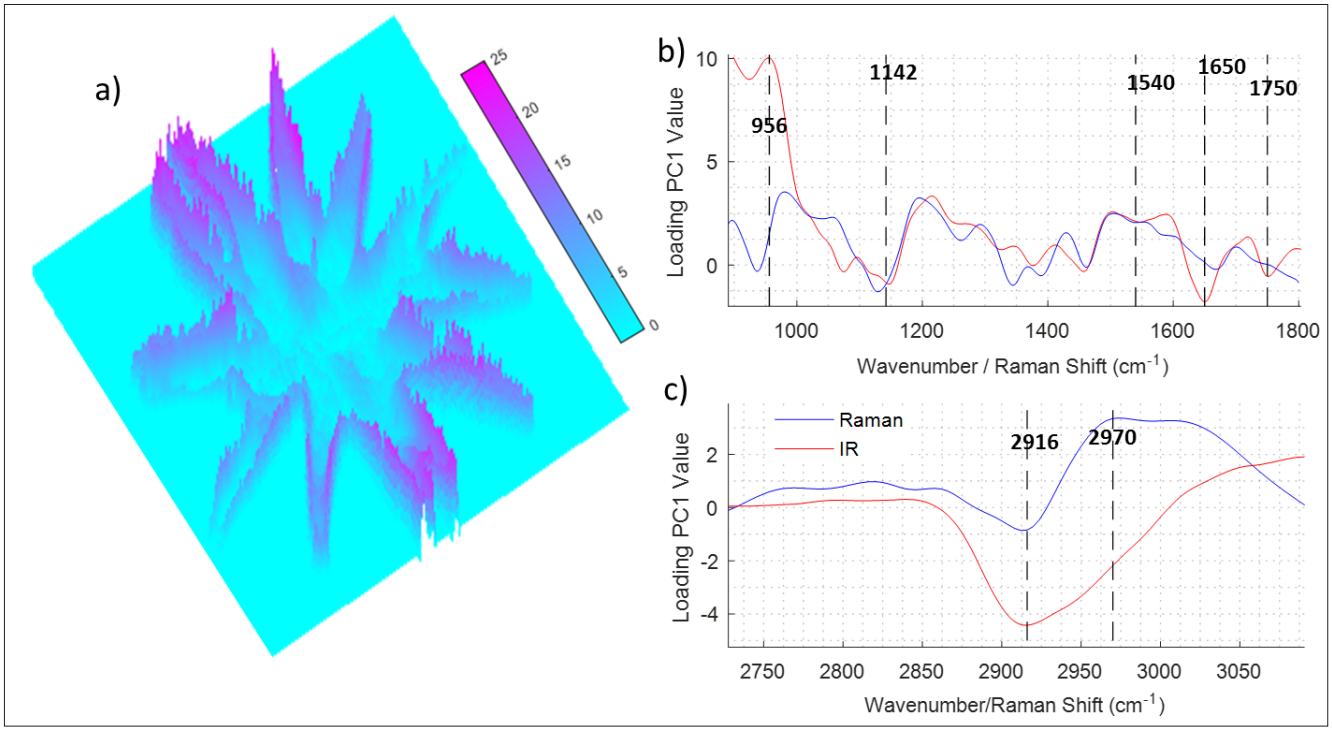


Figure 2. Example of data treatment for the data fusion of infrared and Raman imaging measurements of an algae. a) PseudoColor image representing the first score. b) and c) Loading values of the first score for the 900–1800 cm^{-1} and 2700–3050 cm^{-1} regions.

in the regions associated with the C–H stretching vibrational mode. The broad Raman band at this position is presumably associated with the presence of lipids, which are highly symmetric molecules with a large Raman cross-section. This indicates that lipids are concentrated inside the cells and not on the edges. The fact that the Raman band at 2916 cm^{-1} is inversely correlated to the IR band at 1100–900 cm^{-1} eliminates the possible assignment of this band to lipids. That indicates that the IR bands located at 1100–900 cm^{-1} are caused by a high concentration of carbohydrates.

In summary, the use of multimodal imaging can be technically challenging and requires the use of complex data analysis procedures for resolving the sophisticated relationships between the different variables. However, it provides a comprehensive picture of the biological system under study.

References

1. N.J. de Winter and P. Claeys, "Micro X-ray fluorescence (μXRF) line scanning on Cretaceous rudist bivalves: a new method for reproducible trace element profiles in bivalve calcite", *Sedimentology* **64,** 231–251 (2017). doi: https://doi.org/10.1111/sed.12299
2. D. Perez-Guaita, K. Kochan, M. Martin, D.W. Andrew, P. Heraud, J.S. Richards and B.R. Wood, "Multimodal vibrational imaging of cells", *Vib. Spectrosc.* **91,** 46–58 (2017). doi: https://doi.org/10.1016/j.vibspec.2016.07.017
3. E. Acar, A.J. Lawaetz, M.A. Rasmussen and R. Bro, "Structure-revealing data fusion model with applications in metabolomics", in *Conf. Proc. Annu. Int. Conf. IEEE Eng. Med. Biol. Soc.* **2013,** 6023–6026 (2013). doi: https://doi.org/10.1109/EMBC.2013.6610925
4. D.J. Crockford, E. Holmes, J.C. Lindon, R.S. Plumb, S. Zirah, S.J. Bruce, P. Rainville, C.L. Stumpf and J.K. Nicholson, "Statistical heterospectroscopy, an approach to the integrated analysis of NMR and UPLC-MS data sets: application in metabonomic toxicology studies", *Anal. Chem.* **78,** 363–371 (2006). doi: https://doi.org/10.1021/ac051444m
5. J. Kuligowski, D. Pérez-Guaita, Á. Sánchez-Illana, Z. León-González, M. de la Guardia, M. Vento, E.F. Lock and G. Quintás, "Analysis of multi-source metabolomic data using joint and individual variation explained (JIVE)", *Analyst* **140,** 4521–4529 (2015). doi: https://doi.org/10.1039/C5AN00706B

TONY DAVIES COLUMN

What developments do you need in spectroscopy?

Antony N. Davies[a,b]
[a]Strategic Research Group – Measurement and Analytical Science, Akzo Nobel Chemicals b.v., Deventer, the Netherlands
[b]SERC, Sustainable Environment Research Centre, Faculty of Computing, Engineering and Science, University of South Wales, UK

I have been involved recently in quite heated discussions around what developments we need in the field of spectroscopy which are not currently being provided by our vendors. Phrases like "surely it should be capable of…" or "but it is common sense that the instrument must be able to…" and more frequently "in this day and age it is unacceptable that…".

Do YOU recognise this? Anyway, I thought it was time to look at the developments we would all like to see and to get some feedback from the readership which could help our vendors' Product Managers in deciding on development priorities and market justifications in upgrading the technology we are able to purchase. It has also been suggested to me that we should explore what is a reasonable price to pay for such developments!

What is "the State of the Art"

Take a step back and look at your instrument park. How much has it changed since you took on your current role? How much has it changed since your predecessor took on your role? Do you get the feeling that in your specialism time has actually stood still? Now we know there are specialist labs around with seemingly unlimited funding, full of gleaming kit leant as prototypes by the vendors. But I am talking about a standard analytical laboratory. Very important for our suppliers, as they will represent the source of the majority of their turnover.

For example, if you are an infrared spectroscopist, what is the biggest technology development which has helped you in your work? Probably the biggest development seen in the instrument park in this area was the step—resisted by some vendors—to move from dispersive instruments to Fourier transform instruments by adding a Michelson interferometer as the wavelength selection device. Now, there have been advances in the sample handling attachments that you can add to a base spectrometer with better microscopes or optical fibres for somewhat remote measurements. Clearly, significant focus and advances have been made on the use of advanced statistics (chemometrics) to pull information out of the noise or low resolutions and background contamination signals, but the fundamentally low speeds of the instruments (time per sample for good signal-to-noise ratios) remain. There have also been some micro-spectrometer developments, but these make use of the significantly more powerful computing capabilities to deliver acceptable results in some specific areas of activity from significantly worse data sets, and can only be rather specialist. Probably one of the most amazing facts is the widespread acceptance of an energy source for the spectrometers with such poor linearity in the actual energy delivered to the samples giving significantly different signal-to-noise ratios across the range of wavelengths we expect it to operate in. In an age of the rapid development and vast increases in energy efficiency of, for example, LED light sources, why has the promise of the quantum cascade laser technology not resulted in easily available laser-based infrared spectrometers—even if you have to multiplex arrays of the sources.

Mass spectrometrists are somewhat better served than their optical spec-

troscopy brothers and sisters, with a steady stream of incremental instrumentation improvements leading to fundamentally new ways of approaching their analyses, such as accurate mass approaches to analyte identification and different sample inlet technologies making more difficult samples amenable to a mass spectrometric approach. Nuclear magnetic resonance has seen field strengths increase significantly, although the cost of these restricts their use to a few laboratories.

How does development prioritisation happen?

Money! Pure and simple. I sometimes feel quite sorry for our product managers who have to fight for resources to get new developments to market. If your lab is hanging on to outdated spectrometers for 10–20 years, there are issues for a keen product manager to show where they can produce the required return on the necessary vendor investment. Unlike some other laboratory science areas, spectroscopy usually does not rely on the use of vast amounts of consumables to keep a vendor division's turnover moving. For some of our smaller, innovative start-ups there are lower barriers to get their innovations to market, but much bigger challenges in supporting their innovations on a global stage in the longer term. The necessary overheads of training, deploying and retaining support engineers inevitably start to take up an ever-larger proportion of their precious cash flow, diverting badly needed funding from the work on the next "innovation". This can often lead to their takeover by one of our global multinational vendors with the inevitable corporate culture clash. So, what do we need as a society of spectroscopists to do to break this viscous circle?

Clearly, innovation is possible, we discussed in this column the superb developments in the field of terahertz spectroscopy from the old days of poor weak sources and insensitive detectors in the far-infrared to the latest instruments based on innovative sources and detectors based on completely new technologies.[1]

Return on investment—for us end users and our financial controllers

Evidently, the age of our instrument parks can be one of the issues hindering ongoing innovation? So, what is stopping us from upgrading more frequently? Those of us with strong financial controller guidelines need to produce our own detailed Return on Investment calculations to justify these capital investment decisions. If we look at the full economic costs of the time our expert staff are spending with outdated equipment, it is not hard to find 10–20% improvements in the data acquisition speed. The latest Royal Society of Chemistry staff remuneration survey results will be available for all to see from 1 November, but even without the latest figures it is likely that you should easily be able start with at least 20–30k € in cost savings over the write-down period of the planned investment.[2]

You should next look at the issues brought to our laboratories from Microsoft's policies on stopping legacy operating system support. It is often preferably, in both industrial and academic organisations, to have expensive spectrometers supplying data to several users, and to do this efficiently it is best to carry out the analysis of this data off-spectrometer. The legitimate demands of the IT support police to keep ancient instrument control PCs off the internal networks as potential sources of security vulnerabilities usually means additional loss of efficiency. These demands can result in simply stopping off-spectrometer data analysis or resulting in scientists running backward and forward clutching USB sticks, or USB drives for the instruments delivering hyperspectral high-resolution data sets—that is, of course, if the old computers actually support USB! So, let us add another 10% to the ROI calculation for inefficiencies which have crept in to the data analysis workflows as the spectrometers have aged.

How about considering, for example, the time lost by your internal or external colleagues who may potentially be waiting for your delayed results on their samples before they can work out the most appropriate strategy to handle

TONY DAVIES COLUMN

a key customer's complaint. These may also provide further strong financial leverage for an instrument update policy. Finally, and probably most obviously, more modern instruments tend to be far more efficient in terms of energy consumed, expensive laboratory floor or bench space required and consumables devoured.

Return on Investment—for the instrument and software vendors

OK, so if we manage to revive demand by making it clear to our finance experts how an up-to-date instrument park can bring strong financial sense, let us look at some of the underlying issues hindering our vendor product managers from making the case for more innovation in their spectrometer offerings for "normal" laboratories. First, and quite annoyingly, it may again be worth looking longingly across at our brother and sister chromatographers. They are extremely well served by their vendors providing a range of chromatography data systems which deliver standardised data handling routines for chromatogram processing and results generation. They also provide multi-run and multi-vendor instrument control, greatly simplifying the development of the off-instrument data processing strategy. It also allows for better innovation deployment, as you can buy the best separation science hardware for the desired task without having to deploy another data processing solution for your scientists to have to learn. This capability can only be dreamt of at the moment for the spectroscopy laboratory. Current attempts to integrate hyphenated methods linking chromatography and spectrometry instrumentation into centralised CDS data processing workflows have only been partially successful. So, if our spectroscopy vendors can lower their guards a little and allow access to their control codes, maybe we can look to a future of reduced overall instrument development costs through the use of common CDS-like instrument control capabilities.

It is also worth looking outside our own industry for examples where others have faced similar price pressures. In the automobile industry, we live in an age of common chasses underpinning vehicles addressing vastly differing user needs from sports cars to people carriers. Such an approach could also reduce the cost of introducing innovation where the development can focus on the new ideas whilst relying on stable common components to keep development costs focused where they need to be deployed. This would also deliver further downstream cost savings for our vendors in the area of keeping their support engineers trained and up to speed on their diverse instrument parks.

Our survey

So, in our utopian world we have shown why and how you can justify financing the purchase of innovation from our vendors, and also some ideas as to how vendors can address the stifling ROI demands from corporate financial controllers to even allow innovation to be pursued. So, what do you want to be delivered? Please go to our survey page at https://www.spectroscopyeurope.com/survey and answer three simple questions:

- What field of spectroscopy do you work in?
- Name the top three instrumentation innovations you would like to see available to help in you daily work.
- Name the top three software innovations you would like to see available in coming years.

And let us see if we cannot persuade our suppliers to protect and nurture innovators and our financial controllers to realise the benefits of reinvigorating our instrument parks and working environments. Your staff are, of course, your best assets, but do not underestimate the benefits of targeting investments to ensure your best assets have access to the best tools to support their daily work.

Comments

Perhaps you disagree? Feel free to comment on the online version of this article at https://www.spectroscopyeurope.com/td-column/what-developments-do-you-need-spectroscopy.

References

1. A.N. Davies, "Terahertz spectroscopy", *Spectrosc. Europe* **26(1),** 23 (2014). http://bit.ly/tdthz
2. *The results are in....* Royal Society of Chemistry (2015) [Accessed 15 October 2017]. http://rsc.li/2giFcGc

thermoscientific

What did you do today?

Whether you're discovering new materials, solving analytical problems or assuring product quality, your spectrometer needs to deliver the definitive answers you're looking for — fast! Thermo Fisher Scientific goes beyond your expectations with a full line of FTIR, NIR and Raman spectroscopy systems to help you move from sample to answer, faster than ever before.

With the Thermo Scientific™ DXR™2xi Raman Imaging Microscope, you get an easy-to-use system that rapidly delivers accurate data – so you spend less time concentrating on measurement and more time on scientific discovery.

Discover. Solve. Assure.
thermofisher.com/discover-dxr

ThermoFisher SCIENTIFIC

SAMPLING COLUMN

Experimental validation of a primary sampling system for iron ore pellets


Karin Engström

Karin Engström[a] and Kim H. Esbensen[b]
[a]PhD Student & Method development engineer in sampling and analytical test methods
[b]KHE Consulting (www.kheconsult.com) & Guest professor (Denmark, Norway, Puerto Rico)

This feature forms an intermezzo in the current segment of process sampling columns. We have asked Karin Engström, Luossavaara Kiirunavaara AB (LKAB), Kiruna, Sweden to outline how industrial validation of a process sampling system takes place following ISO standards guidelines. These prescribe a rigorous procedure for comparison of a process sampling system with a "stopped belt" + manual sample extraction reference system as a means for checking for a sampling bias, as the reference sampling system is considered to be fully TOS-compliant, i.e. representative. This column forms a comprehensive backdrop for the on-line alternative of variographic characterisation of the same iron ore pellet stream (which will follow in the next installment).

Introduction: status of current ISO standards

Primary sampling of iron ore is well established and standardised through the International Organization for Standardization (ISO). In comparison to standardisation of other mineral commodities and particulate materials (e.g. food/feed, pharmaceuticals), iron ore sampling standards are in close compliance with the Theory of Sampling (TOS).[1–3] Iron ore mining and processing operations apply sampling and grade control in all parts of the production value chain, from diamond drill and blast holes all the way to process sampling of slurries, pellet feed, finished pellets and at ship loading. Sampling of iron ore is standardised through the international standard ISO 3082: "Iron ores—Sampling and sample preparation procedures".[4] The iron ore industry has improved its conformance to ISO 3082 over the last 10–20 years, especially regarding commercial purposes. However, there are still several areas where deviations from the standard and issues with sample representativity are common.[3] For newly constructed sampling systems or in-use systems that have been modified, ISO 3082 demands verification of the full sampling system in accordance to ISO 3086: "Iron ores—Experimental methods for checking the bias of sampling".[5] We here report on an experimental verification and validation experiment, as a base-line reference to be compared with an on-line variographic sampling system QC in the following column.

Fundamental Sampling Principle and basic requirements for iron ore sampling systems

The Fundamental Sampling Principle (FSP) for representative sampling states that all parts of the lot must have *equal probability* of being selected for the sample.[6–8] This principle is equally important for primary sampling extraction as for all subsequent sampling stages, i.e. during mass reduction/sample division. ISO 3082 describes the best place for primary sample extraction to be at a transfer point between conveyor belts, where a full cross-section of the stream can be intercepted, and extracted at regular intervals. Sampling from stationary lots such as ships or stockpiles is not permitted by ISO 3082, as it is impossible to drive a sampling device through the lot depth and extract a full column of ore. ISO 3082 therefore recommends only to extract samples as the ore is being transported to or from a ship, stockpile, bunker or silo.

The extraction of primary increments shall comply with the following regulations to ensure that no bias is generated (well-tested TOS principles):

- a complete cross-section of the ore stream shall be taken when sampling from a moving stream;
- the aperture of the sample cutter shall be at least three times the nominal top size of the ore, or 30 mm for the primary sampling and 10 mm for subsequent stages, whichever is the greater;
- the speed of the sample cutter shall not exceed 0.6 m s^{-1}, unless the cutter aperture is correspondingly increased;
- the sample cutter shall travel through the ore stream at uniform speed, both the leading and trailing edges of the cutter clearing the ore stream at the end of its traverse;
- the lips on the sample cutter shall be parallel for straight-path samplers and radial for rotary cutters; these conditions shall be maintained as the cutter lips wear;

- changes in moisture content, dust loss and sample contamination shall be avoided;
- free-fall drops shall be kept to a minimum to reduce size degradation of the ore pellets and hence minimise bias in size distribution;
- primary cutters shall be located as near as possible to the loading or discharging point to further minimise the effects of size degradation;
- a complete column of ore with nominal top size less than 1mm shall be extracted when sampling iron ore concentrate in a wagon.

Principles and general requirements for checking sampling bias

The method for checking for sampling bias is to compare the online stream sampling system (Figure 1) to a reference sampling method considered to produce true and unbiased results. For 1-D process sampling, the reference is the so-called "stopped belt" sampling using a sampling frame as outlined for manual increment/sample extraction, see Figure 2.

The number of *paired comparisons* between the reference method (method A) and the sampling method to be tested (method B) should be no less than ten. The samples A and B should be taken as close together as possible to ensure that the local variability in the ore does not affect the bias test. Quality characteristics important to the ore, such as iron content, size distribution or other metallurgical, chemical or physical properties can be used for bias testing (ISO 3082). But it is well-known that size distribution parameters offer the most powerful check—if the size distribution of method A and B is identical, so will the chemistry.

The paired measurements for the selected quality characteristics are compared using a 90% confidence interval, or an equivalent t-test, for checking if there is a bias present for the B sampling system.

The current edition of both the ISO standard for checking of bias (ISO 3082) and the ISO standard for estimation of sampling precision (ISO 3085) support elimination of *outliers* identified through a Grubbs outlier test, taking no account whether assignable causes can be identified or not. This method of outlier eliminating is likely to affect the bias test by favouring the tested sampling system (B) as well as underestimating the sampling system precision. Later publications have recommended that identified outliers should only be eliminated after bona fide assignable causes have been identified; in addition, a new data set should also be collected and processed to ensure correct calculations.[9] (N.B. these published suggestions are in the process of being incorporated in the upcoming revisions of the ISO standards for iron ore sampling.)

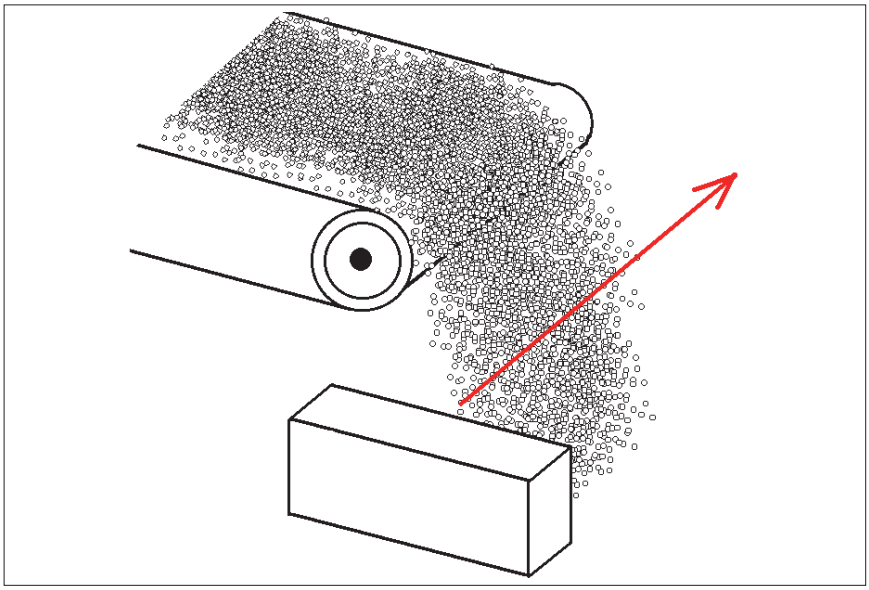


Figure 1. Illustration of the pinciple of the recommended cross-stream sampler. Image credit: ACABS research group.

Figure 2. Example of a "stopped belt" sampling frame allowing a complete cross-section of the ore stream to be extracted. This will serve as a reference to the online sampling system (Figure 1). Image credit: LKAB.

Validation experiment

The experimental validation reported here was executed at a sampling system collecting the final product from an iron ore pellet plant (i.e. process control samples—**not** commercial samples). The sampling system consists of a linear cross-stream sampler, Figure 1, collecting primary increments of iron ore pellets in accordance with the guidelines (ISO 3082). This system is based on a systematic time interval, collecting

SAMPLING COLUMN

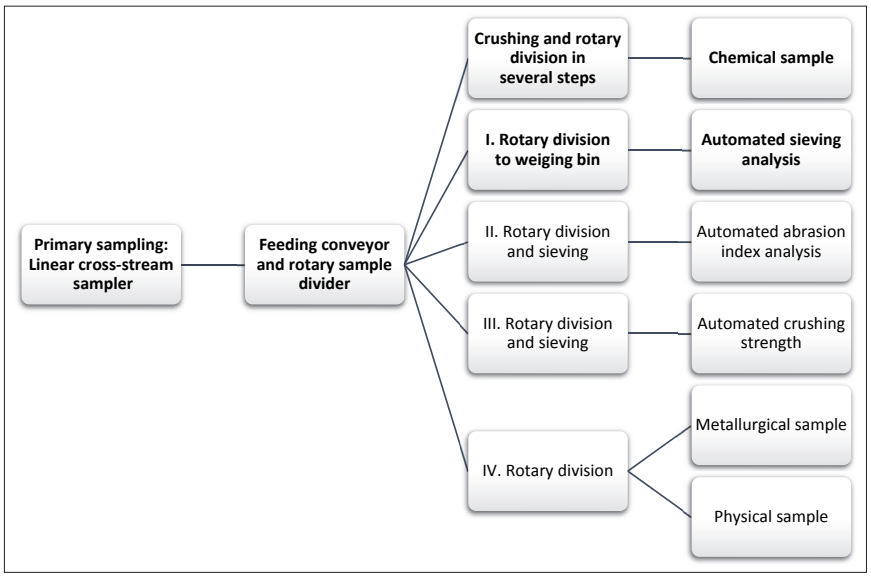


Figure 3. General schematics of complete automated sampling system, bold parts of the system were validated in the current study.

primary increments every five minutes. Apart from the primary sampling, the sampling system is fully automated with regard to sampling division, crushing for chemical analysis, sieving analysis, abrasion index analysis and crushing strength analysis.

In the present validation experiment, the primary sampling, sampling division and automated sieving analysis were validated. The schematics of the sampling system is presented in Figure 3. The ISO 3086 approach was used with the one exception that six (not ten) paired samples were extracted.

Experimental results

The validation results for the primary linear cross-stream sampler are presented in Table 1. The data were analysed using a two-sided t-test with a 95% confidence level. The t-test indicates that the primary sampler did not generate any bias for all parameters *except* for the particle size <5 mm, where a significant difference between the A and B sampling methods was identified: the linear cross-stream sampler overestimates the amount of fine material in the lot.

The reason for this discrepancy was investigated by thorough inspection of the complete sampling system, leading to the conclusion that the problem was not related to increment extraction, but rather due to *dust accumulation* on the conveyor belt during transportation of the primary increments to the first rotary sample divider. This problem was *counteracted* by improving ventilation to decrease the amount of "ambient dust" in the environment around the primary sampler, and by improving the physical shielding of the conveyor, ensuring no dust can reach the belt from outside sources.

To ensure TOS-correctness (un-biasness) of the automated sieve analysis connected to the primary sampling system, a bias test was also conducted in relation to a well-controlled reference laboratory sieving setup. The results of the t-test for the six compared samples are presented in Table 2. The t-test does not show any statistical difference between the two pieces of sieving equipment and the validation of the automated sieve is therefore approved.

Discussion

The result from the validation experiment of the primary linear cross-stream sampler illustrates the critical importance of validation experiments and bias testing. Even though the sampling system was constructed according to TOS principles and ISO 3082, a bias could be detected due to sample contamination after primary sample extraction, revealing an Incorrect Preparation Error (IPE) effect.

The decision to decrease the number of paired samples from ten to six in this validation experiment was due to cost considerations at the discretion of LKAB. Thus, the experiment had the possibility to be extended with four additional paired samples **if** the results were inconclusive.

Table 1. Validation data for linear cross-stream sampler bias test.

	Fe	SiO_2	12.5–16 mm	10–12.5 mm	<5 mm
Mean: linear cross stream samples	66.69%	2.143%	12.5%	59.6%	2.0%
Mean: stopped belt samples	66.70%	2.135%	12.6%	59.4%	1.2%
Mean difference	–0.01%	0.008%	–0.1%	0.2%	0.8%
Standard deviation for mean difference	0.043%	0.026%	1.6%	1.8%	0.4%
Critical t-value	2.57	2.57	2.57	2.57	2.57
Statistical t-value	–0.67	0.71	0.13	–0.32	–5.0
Significant difference?	No	No	No	No	Yes

Table 2. Validation data for the automated sieve analysis sampler.

	12.5–16 mm	9–12.5 mm	5–9 mm
Mean: automated sieve	18.7%	74. 9%	3.5%
Mean: reference sieve	18.5%	75.2%	3.9%
Mean difference	0.2%	–0.3%	–0.4%
Standard deviation for mean difference	4.7%	3.9%	0.9%
Critical t-value	2.57	2.57	2.57
Statistical t-value	0.09	–0.19	–1.07
Significant difference?	No	No	No

Even though a base-line validation of all new or modified sampling systems is required (ISO 3082), *continuous* monitoring and control is also mandatory—to ensure continuous representative sampling and analytical results.

Apart from regular maintenance and visual inspection, continuous variographic characterisation is an efficient way of monitoring and quality grading process sampling systems over time, ensuring that significant deviations in sampling or analytical variability has accidentally been introduced. The variographic approach is well described in previous columns, and will be applied to the same parameters used here to show how the present iron sampling procedure also can be monitored on-line (in a following column).

References

1. P. Gy, "Sampling of discrete materials—a new introduction to the theory of sampling I. Qualitative approach", *Chemometr. Intell. Lab. Syst.* **74,** 7–24 (2004). doi: https://doi.org/10.1016/S0169-7439(04)00167-4
2. R.J. Holmes and G.J. Robinson, "Codifying the principles of sampling into minerals standards", *Chemometr. Intell. Lab. Syst.* **74,** 231–236 (2004). doi: https://doi.org/10.1016/j.chemolab.2004.03.011
3. R.J. Holmes, "Common pitfalls in sampling iron ore", *Proceedings 8th World Conference on Sampling and Blending*, 9–11 May, Perth, Australia, pp. 261–264 (2017).
4. International Organization for Standardization, *ISO 3082: Iron Ores—Sampling and Sample Preparation Procedures* (2009).
5. International Organization for Standardization, *ISO 3086: Iron Ores—Experimental Methods for Checking the Bias of Sampling* (2006).
6. P. Gy, *Sampling of Particulate Materials—Theory and Practice, 2nd Edn*. Elsevier, Amsterdam (1979).
7. F.F. Pitard, *Pierre Gy's Sampling Theory and Sampling Practice, 2nd Edn*. CRC Press Inc., Florida (1993).
8. J.E. Everett, T.J. Howard and B.J. Beven, "Precision analysis of iron ore sampling preparation and measurement overcoming deficiencies in current standard ISO 3085", *Min. Technol.* **120,** 65–73 (2011). doi: https://doi.org/10.1179/1743286311Y.0000000002
9. K.H. Esbensen (Chairman Taskforce F-205 2010–2013), *DS 3077. Representative Sampling—Horizontal Standard*. Danish Standards (2013). http://www.ds.dk

NEWS

Paper spray MS for cocaine use test

A team, led by Dr Catia Costa and Dr Melanie Bailey from the University of Surrey, has developed a new technique to analyse the levels of cocaine detected in fingerprints. They used chromatography paper to take the sample for analysis by paper spray mass spectrometry. The study involved taking fingerprints from a group of patients seeking treatment at drug rehabilitation centres, as well as a larger group not known to be drug users. All of those taking part washed their hands before the test in a variety of ways, and then samples were collected on the prepared chromatography paper. The fingerprint is developed using chemicals, so that the ridges of the fingerprint (and therefore the identity of the donor) can be established prior to analysis. When someone has taken cocaine, they excrete traces of benzoylecgonine and methylecgonine as they metabolise the drug, and these chemical indicators are present in fingerprint residue. Importantly, the traces can still be detected even after handwashing.

It is anticipated that this technology could see the introduction of drug tests for law enforcement agencies to use within the next decade. Drug testing is used routinely by probation services, prisons, courts and other law enforcement agencies. However, traditional testing methods have limitations. Where bodily fluids are tested, there can be biological hazards and often a requirement for particular storage and disposal methods.

Their work is reported in *Clinical Chemistry* (doi: https://doi.org/10.1373/clinchem.2017.275578).

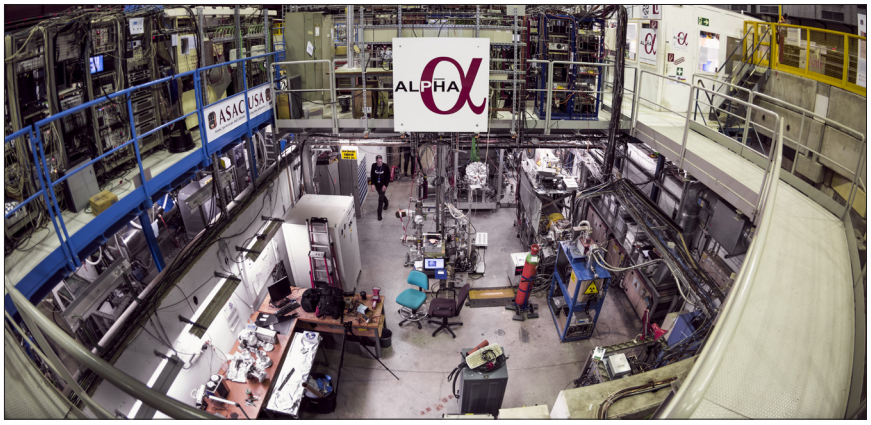
Metabolic studies using LC-MS determine if carrots are organic

In recent years, the organic food market in the EU, driven by steadily increasing demand, has developed significantly. The overall challenge faced by the organic sector is to ensure a steady growth of supply and demand, while maintaining consumers' trust. An element to be considered is the pressure of demand that also increases the risk of fraudulent behaviours or other intentional violations. Not only do they harm consumers' interest and cause economic damages distorting competition, but they can also negatively impact on reputation of organic operators that are complying with the rules.

In such context, JRC scientists carried out a comprehensive biochemical analysis based on untargeted liquid chromatography–mass spectrometry (LC–MS) metabolomics of carrots coming from different agronomic environments over a period of four years. Carrot samples of Nerac and Namur varieties were collected directly from paired fields from the Walloon region of Belgium, one with a conventional growing system and one with organic husbandry. The extracts (organic solvents) from the carrot samples were analysed using high performance liquid chromatography coupled with mass spectrometry. Using a strict quality control scheme, the data acquired were subsequently exported for multivariate statistical analysis.

Compounds were identified following guidelines of the Metabolomics Standards Initiative. With the use of chemometrics it was possible to classify samples according to agricultural practices and predict the origin of unknown samples. Several markers related to carbohydrate metabolism and plant defence mechanism were identified as responsible for the differences between organic and conventional agricultural systems.

This is the first time that a metabolomics approach has been used for organic food authentication purposes in a long-term (four years) field study and by using external validation sample sets to predict the origin of unknown samples. The work was published in *Food Chemistry* (doi: https://doi.org/10.1016/j.foodchem.2017.06.161).



The Alpha experiment at CERN (image © CERN).

Spectroscopy of antimatter

The ALPHA experiment at CERN's Antiproton Decelerator has reported the first observation of the hyperfine structure of antihydrogen, the antimatter counterpart of hydrogen, using microwave spectroscopy. These findings (published in *Nature*, doi: http://doi.org/10.1038/nature23446) point the way to ever more detailed analyses of the structure of antihydrogen and could help understand any differences between matter and antimatter.

The researchers conducted the microwave spectroscopy measurements on homemade antihydrogen atoms, which drive transitions between different energy states of the anti-atoms. They could in this way improve previous measurements by identifying and measuring two spectral lines of antihydrogen.

In 2012, the ALPHA experiment demonstrated for the first time the technical ability to measure the internal structure of atoms of antimatter. In 2016, the team reported the first observation of an optical transition of antihydrogen. By exposing antihydrogen atoms to microwaves at a precise frequency, they have now induced hyperfine transitions and refined their measurements. The team were able to measure two spectral lines for antihydrogen, and observe no difference compared to the equivalent spectral lines for hydrogen, within experimental limits.

"Spectroscopy is a very important tool in all areas of physics. We are now entering a new era as we extend spectroscopy to antimatter", said Jeffrey Hangst, Spokesperson for the ALPHA experiment. "With our unique techniques, we are now able to observe the detailed structure of antimatter atoms in hours rather than weeks, something we could not even imagine a few years ago."

With their trapping techniques, ALPHA are now able to trap a significant number of antiatoms—up to 74 at a time—thereby facilitating precision measurements. With this new result, the ALPHA collaboration has clearly demonstrated the maturity of its techniques for probing the properties of antimatter atoms.

The rapid progress of CERN's experiments at the unique Antiproton Decelerator facility is very promising for ever more precise measurements to be carried out in the near future.

New technology enables standard cameras to produce hyperspectral images

Researchers at Ben-Gurion University of the Negev (BGU) have developed miniaturised hyperspectral technology as an add-on for a standard camera that will generate superior quality images and video faster and at a lower cost than currently available commercial devices.

The device, developed by four BGU researchers, can re-purpose a standard camera for many applications. The device is only a few tens of μm wide and is easy

to produce, using commonly available materials such as liquid crystal.

Professor Adrian Stern, head of BGU's Unit of Electro-Optical Engineering says, "The technology uses our software based on 'compressive sampling', which minimises collection of redundant data during image capture, making the camera at least 10 times faster and creates spectral images of a markedly superior quality." Compressive sampling captures signals and images from much fewer samples or measurements than the traditional Nyquist–Shannon sampling theorem on which cameras and sensing devices have been based for 100 years.

"Instead of using a large and heavy prism inside the camera, we developed a very small, tunable filter and sensor that are activated by electrical current", says Professor Dan Blumberg, BGU vice president and dean for research and development, who is also part of the research team. "Every time the current changes, a photo is taken."

The development team also includes Professor Stanley Rotman of the Department of Electrical and Computer Engineering and Dr Yitzhak August, a former PhD student in the Electro-Optical Engineering Unit.

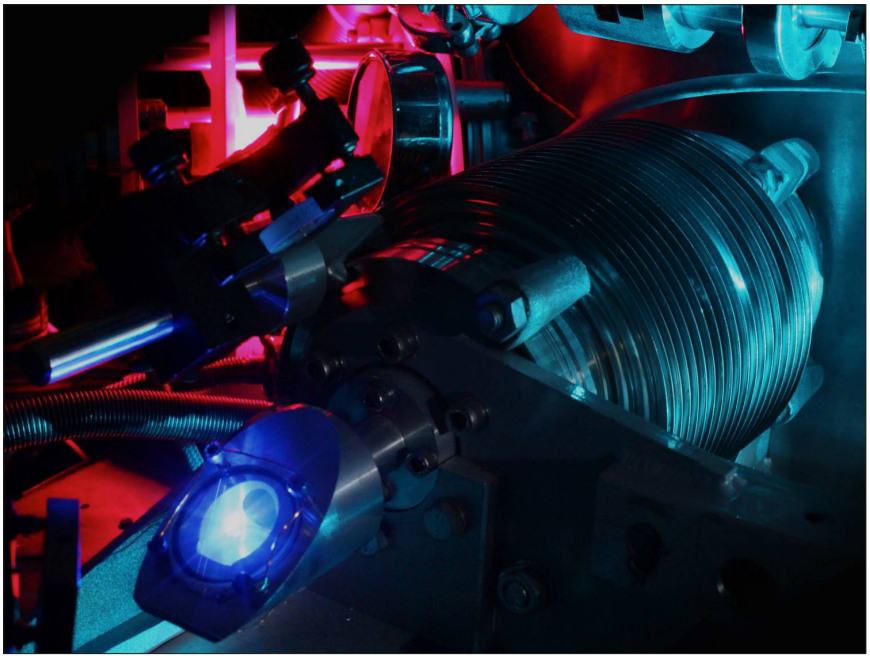
Laser spectroscopy confirms small radius of hydrogen proton

It was one of the breakthroughs of the year 2010: laser spectroscopy of muonic hydrogen resulted in a value for the proton charge radius that was significantly smaller, by four standard deviations, than previous determinations using regular hydrogen. This discrepancy and its origin have attracted much attention in the scientific community, with even extensions of the so-called standard model of physics being discussed. Now, a team of scientists from the Laser Spectroscopy Division of Professor Theodor W. Hänsch at the Max Planck Institute of Quantum Optics in Garching has made a new spectroscopic measurement of regular hydrogen. The resulting values for the Rydberg constant and the proton radius are in excellent agreement with the muonic results (*Nature*, doi: https://doi.org/10.1038/nature09250), but disagree by 3.3 standard deviations with the average of the previous determinations from regular hydrogen.

Hydrogen is the simplest of all chemical elements. According to the model proposed by Niels Bohr in 1913, it consists of a single proton and an electron orbiting around it. The theory of quantum electrodynamics predicts the energy levels of this system with 12 digits of precision. Because of this, hydrogen plays a key role in our understanding of nature. Its study allows the determination of fundamental constants such as the Rydberg constant and the proton charge radius.

Hydrogen is thus the ideal subject for testing the laws of nature. This is why a measurement on muonic hydrogen, resulting in a surprisingly small value for the proton charge radius, made big waves in 2010. In that experiment, done at the Paul Scherrer Institute in Villingen, Switzerland, the electron of the hydrogen atom is replaced with its sister particle, the 200-times heavier and short-lived muon. Laser spectroscopy of this muonic hydrogen resulted in a value of the proton radius that was extremely precise, but four percent smaller than previous measurements on regular hydrogen. "Since the muon is 200-times heavier than the electron, it orbits much closer to the proton and 'feels' its size", explains Professor Randolf Pohl (now at Johannes Gutenberg-Universität Mainz), a member of the MPQ team. "Because of this, the proton radius has a seven orders of magnitude larger influence on the spectral lines than in regular hydrogen. This allows us to determine the proton radius with such a high precision."

The large discrepancy between the measurements of regular hydrogen and its exotic cousin has sparked many debates about its origin. "However, some of the previous measurements in fact agree with the muonic value. The influence of the proton radius on the energy levels in regular hydrogen is tiny, and even very high precision measurements struggle to resolve it. The discrepancy only becomes significant when all measurements are averaged", explains Lothar Maisenbacher, one of the graduate students working on the project. "This is why, to solve this 'proton radius puzzle', new individual measurements



This photo shows the vacuum chamber used to measure the 2*S*–4*P* transition frequency in atomic hydrogen. The purple glow in the back stems from the microwave discharge that dissociates hydrogen molecules into hydrogen atoms. The blue light in the front is fluorescence from the ultraviolet laser that excites the atoms to the 2*S* state. The turquoise blue glow is stray light from the laser system used to measure the frequency of the 2*S*-4*P* transition. (Photo: MPQ)

with high precision, and, if possible, using different experimental approaches are necessary."

In order to determine both the Rydberg constant and the proton charge radius from spectroscopy of regular hydrogen, two different transition frequencies need to be measured. The by far sharpest resonance, the so-called 1*S*–2*S* transition, serves as a corner stone in this determination. Its frequency has been measured, in 2011, to 15 digits by the MPQ team (*Phys. Rev. Lett.* doi: https://doi.org/10.1103/PhysRevLett.107.203001). This high precision was made possible not least by the invention of the frequency comb, for which Professor Hänsch was awarded the Nobel Prize in Physics in 2005. For the second frequency measurement needed, the MPQ team chose the so-called 2*S*–4*P* transition, which connects the metastable 2*S* state with the much shorter lived 4*P* state.

In the experiment, this transition is excited by a laser with a wavelength of 486 nm and the collected fluorescence from the decay of the 4*P* state serves as a signal. The apparatus used previously for the 1*S*–2*S* measurement serves as a source of atoms in the 2*S* state. Compared to previous experiments, which used room temperature atoms, the atoms probed here have a substantially lower temperature of 5.8 Kelvin and, consequently, a much lower velocity. This, together with especially developed techniques, strongly suppresses the Doppler shift, which constitutes the largest source of uncertainty for this measurement.

"Another source of uncertainty in this experiment is the so-called quantum interference", explains Lothar Maisenbacher. "If we could probe a single, isolated transition, the shape of the resulting spectral line would be symmetric. However, in our case there are two other upper states that can be excited by the laser, called $4P_{1/2}$ and $4P_{3/2}$. This results in a slightly asymmetric shape of the spectral lines, making the determination of the line centre more challenging. While this is a very small effect, it plays a big role for us because we determine the line centre with such a high precision of almost one part in 10,000 of the line width."

To describe the influence of the quantum interference, the scientists performed sophisticated numerical simulations, which are in very good agreement with the experimental results. "In our case, however, a specially derived, simple fit function is sufficient to remove the effects of quantum interference", emphasises Vitaly Andreev, also a graduate student on the project. "We use this fit function for our data evaluation. In this way, the simulation is only needed for small corrections on the order of 1 kHz."

With this, the MPQ team managed to determine the frequency of the 2*S*–4*P* transition with an uncertainty of 2.3 kHz. This corresponds to a fractional uncertainty of 4 parts in 10^{12}, making this the second-best spectroscopy measurement of hydrogen after the aforementioned 1*S*–2*S* transition measurement. Combining these results, the Rydberg constant and the proton size are determined to be $R_\infty = 10973731.568076(96)\ m^{-1}$ and $r_p = 0.8335(95)$ fm, respectively.

"Our measurement is almost as precise as all previous measurements on regular hydrogen combined," summarises Professor Thomas Udem, the project leader. "We are in good agreement with the values from muonic hydrogen, but disagree by 3.3 standard deviations with the hydrogen world data, for both the Rydberg constant and the proton radius. To find the causes of these discrepancies, additional measurements with perhaps even higher precision are needed. After all, one should keep in mind that many new discoveries first showed up as discrepancies."

This work is reported in *Science* (doi: https://doi.org/10.1126/science.aah6677).

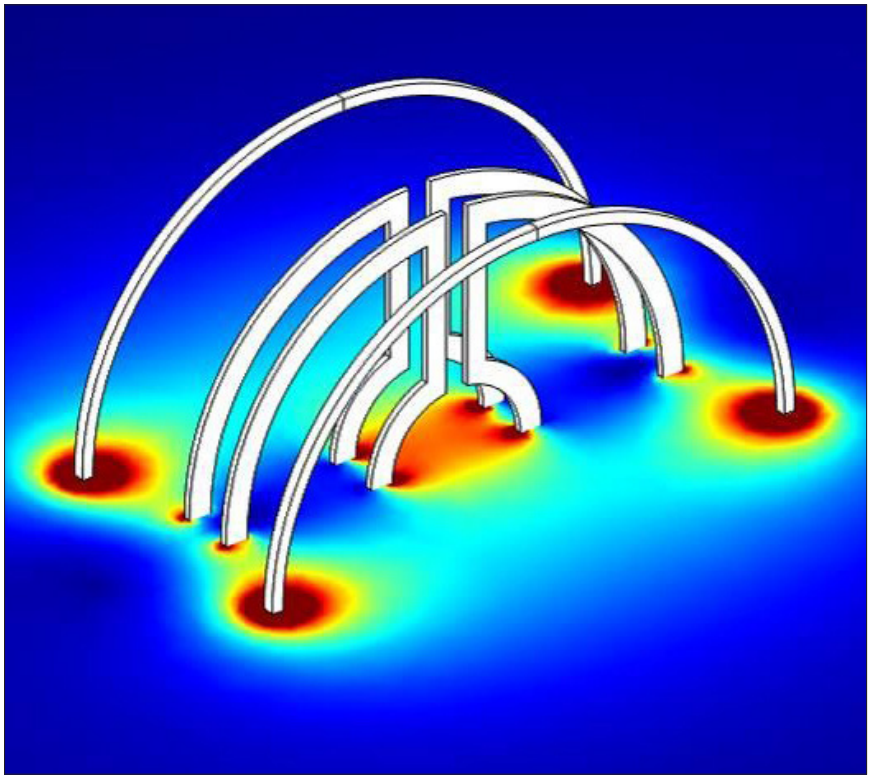
High sensitivity NMR on very small scales

The physical effect of nuclear magnetic resonance (NMR) is used to study molecular properties of matter. The specimen is located in a high constant magnetic field and irradiated with a high-frequency alternating magnetic field. Both magnetic resonance tomography (MRT)

and NMR spectroscopy, are based on nuclear magnetic resonance. However, research has to cope with the challenge of constantly improving the unfavorable signal-noise ratio and, thus, increasing the sensitivity of NMR measurements. "A high sensitivity is indispensable in particular when applying mass- and volume-limited methods or when a high spatial resolution is required", Professor Jan Gerrit Korvink, Director of the Institute of Microstructure Technology (IMT) of Karlsruhe Institute of Technology (KIT) explains.

In NMR measurements on small samples, miniaturised high-frequency coils proved effective for the generation and reception of the alternating magnetic field. For mobile applications and further miniaturisation, an international team of researchers has now developed a new method to enhance sensitivity in very small spaces. They use magnetic lenses, so-called Lenz lenses, to focus the magnetic flux of a macroscopic high-frequency coil on a smaller volume and to locally enhance sensitivity. With these lenses named after physicist Emil Lenz, who published the law on changing magnetic flux, magnetic flux of the alternating field cannot only be focused, but also diverted or transformed. In this respect, the effect of these lenses can be compared with that of optical lenses on light beams. Change of the magnetic field induces current in the Lenz lenses made of metal plates or wires in symmetric or asymmetric arrangement. The shape of the lenses guides the induced currents such that the magnetic field is focused.

With Lenz lenses, sensitivity of measurements in very small spaces, into which conventional NMR systems do not fit, can be increased significantly. Moreover, the lenses work at any field strength. Among others, medical applications can benefit from the use of Lenz lenses, Korvink says: "As the Lenz lenses are not wired, they are particularly suited for implants". Application might be feasible in brain implants to observe healing of the tissue over longer terms with high resolution or on plasters for the observation of skin cancer. Currently, scientists are exploring further applications, among others in electrical engineering.



Two Lenz lenses are arranged in a Helmholtz coil pair. Simulation shows how the Lenz lenses focus magnetic flux. (Illustration: Nils Spengler/KIT)

Professor Jan Gerrit Korvink, together with Dr Nils Spengler, Professor Ulrike Wallrabe, Dr Peter T. While and Markus V. Meissner, experimentally demonstrated the use of Lenz lenses in NMR measurements and mathematically formulated the underlying principle. The results of the scientists' work has been reported in the *PLOS ONE* (doi: https://doi.org/10.1371/journal.pone.0182779).

Finding life: search for spectral biomarkers in planetary atmospheres

The physicist Dr Andreas Elsaesser from the Freie Universität Berlin has been granted a Freigeist Fellowship from the Volkswagen Foundation for his research project "Finding Life: Spectral Biomarkers in Planetary Atmospheres". With the Freigeist grant of roughly one million euros, he aims to investigate molecules to determine whether they offer signs of possible life on other planets. A major focus will be on the stability and spectral detectability of organic molecules that can potentially be detected as so-called biomarkers or biosignatures in the atmosphere of a planet.

One of Dr Elsaesser's main areas of research is the interaction between radiation and matter, in particular, the effects of radiation on organic molecules and biological systems. He investigates the stability of biomolecules both by means of the most advanced spectroscopic methods in ultraviolet and visible wavelengths up to the infrared wavelength range as well as by means of mass spectrometric techniques. Elsaesser is currently involved in several projects on the International Space Station (ISS), and together with colleagues from the German Aerospace Center, the European Space Agency and NASA, with the goal of investigating the effect of solar and cosmic radiation on organic molecules and potential biomarkers. The knowledge gained will help identify potential organic molecules and their decay products and possibly detect them on other planets.

continued on page 25

Wiley Spectra Lab
Spectroscopy Solved

WILEY-VCH WILEY

The world's largest spectral database, with over 2.2 million spectra

Wiley Spectra Lab provides researchers with access to 2.2 million MS, NMR, and IR spectra – the broadest and largest collection in the world. This expert spectral data system that uses empirical data and advanced software to help chemists, toxicologists, and life scientists confidently identify chemical substances.

To arrange a demo, email dbinquiry@wiley.com
www.wileyspectralab.com

Powered by KnowItAll®

BIO-RAD

PRODUCT FOCUS

Product Focus on Mass Spectrometry

Spectroscopy Europe Product Focuses highlight currently available instrumentation in a particular area of spectroscopy. This Product Focus is on Mass Spectrometry, and some companies have provided information on their key products, their applications and features.

See our media information (https://www.spectroscopyeurope.com/advertise) for details of future Product Focuses.

Advanced Chromatography Technologies Limited

Tel: +44 1224 841301
info@ace-hplc.com
www.ace-hplc.com

PRODUCT: ACE Method Development Kits for UHPLC / HPLC
APPLICATIONS: HPLC and UHPLC method development ▪ LC-MS
KEY FEATURES: Columns based on ultra-inert high efficiency silica ▪ Kits enable chromatographers to maximise selectivity ▪ Fully porous, solid-core and Bioanalytical 300A kits ▪ Wide range of particle sizes and column dimensions ▪ Convenient and cost effective

PRODUCT: ACE UltraCore SuperC18
APPLICATIONS: Applications with solid-core particles ▪ Ideal for LC-MS ▪ Compatible with MS compatible buffers with extended pH range of 1.5 to 11
KEY FEATURES: Phase based on high efficiency, low back pressure solid-core particles ▪ Unique Encapsulated Bonding Technology ▪ SuperC18 phase shows minimal background bleed and increased MS response ▪ Available in microbore and analytical column dimensions

Bio-Rad Laboratories, Ltd

Tel: +44 (0) 20 8328 2555
informatics.europe@bio-rad.com
www.knowitall.com

PRODUCT: MS Databases
APPLICATIONS: Pharmaceutical ▪ Industrial ▪ Environmental ▪ Medicinal etc.
KEY FEATURES: Spectral identification ▪ Verification ▪ Classification

PRODUCT: MS Software
APPLICATIONS: Pharmaceutical ▪ Industrial ▪ Environmental ▪ Medicinal etc.
KEY FEATURES: Spectral analysis ▪ Search ▪ Data management

Hiden Analytical Limited

Tel: +44-1925 445 225
info@hiden.co.uk
www.hidenanalytical.com

PRODUCT: QGA Atmospheric Gas Analysis System
APPLICATIONS: Gas reaction studies ▪ Environmental gas analysis ▪ Fermentation analysis ▪ Thermal analysis ▪ Catalysis studies
KEY FEATURES: Real time multiple species gas and vapour analysis ▪ Wide concentration range: 0.1 ppm to 100% ▪ Continuous sampling at atmospheric pressure ▪ Low sample consumption requirements ▪ Integrated CO analyser option for ppm analysis

PRODUCT: HPR-40 DSA for Analysis of Gases, Vapours & VOCs in Liquids
APPLICATIONS: Soil core analysis ▪ Fermentation process analysis ▪ Environmental monitoring ▪ Methane production control ▪ Contamination studies ▪ Electrochemistry
KEY FEATURES: High sensitivity (to 5 ppb) ▪ Mass range options 200, 300 or 510 amu ▪ Compact bench-top/mobile cart/console rack construction ▪ Membrane re-circulation cell option ▪ Fast valve dissolved species probe ▪ New DEMS cells available for electrochemistry

Kore Technology Ltd

Tel: 44 (0)1353653030
info@kore.co.uk
www.kore.co.uk

PRODUCT: PTR-TOF-MS
APPLICATIONS: VOCs ▪ Environment ▪ Breath ▪ Process
KEY FEATURES: Fast ▪ Sensitive ▪ On-line ▪ High mass resolution

PRODUCT: INFORMS-Compact EI-TOFMS
APPLICATIONS: Process ▪ GC-TOFMS
KEY FEATURES: Fast ▪ Sensitive

SepSolve Analytical Ltd

Tel: 44 (0)1733 669222
hello@sepsolve.com
www.sepsolve.com

PRODUCT: BenchTOF-Select™
APPLICATIONS: Fragrance profiling ▪ Flavours and aromas ▪ Petrochemical ▪ Environmental monitoring ▪ Defence and forensics
KEY FEATURES: Unique Tandem Ionisation® for hard & soft ionisation in a single run ▪ Soft EI enables identification of isomers indistinguishable at 70 eV ▪ No inherent loss in sensitivity ▪ Reduced chemical noise improves detection limits for target compounds ▪ Speed up analysis of complex samples with fast GC and GC×GC.

PRODUCT: BenchTOF-HD™
APPLICATIONS: Flavours and aromas ▪ Petrochemical ▪ Environmental monitoring ▪ Defence and forensics ▪ Fragrance profiling
KEY FEATURES: Powerful TOF-DS software for instrument control and data processing ▪ Robust ion source design eliminates the need for regular maintenance ▪ Real-time processing displays results while your sample is acquiring ▪ Sub-unit selectivity for easy elimination of matrix interferences ▪ SIM-like sensitivity for both targets and unknowns

MSP Kofel

Tel: +41 31 972 31 52
MassLib@msp.ch
www.masslib.com

PRODUCT: MassLib® Software Package for Processing and Interpretation of Single and Series of Mass Spectra
APPLICATIONS: MS library search ▪ GC-MS data evaluation ▪ LC-MS data evaluation ▪ $MSMS/MS^n$ data evaluation ▪ Mass spectra archiving
KEY FEATURES: Unique mass spectral search for structurally related similarities ▪ Handling of chromatograms, ion traces, spectra and structures ▪ Import capabilities for most MS vendor data formats ▪ Commercial and user mass spectral libraries with retention index data ▪ Reporting, structure drawing and automation tools

PRODUCT FOCUS

continued from page 22

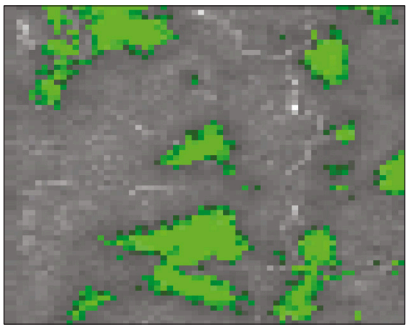
Vibrational microspectroscopy reveal diabetes progression

Researchers from Umeå University in Sweden have described a new method based on molecular spectroscopy to study biochemical changes that occur in the pancreas during the development of diabetes. The method, recently published in *Scientific Reports* (doi: https://doi.org/10.1038/s41598-017-07015-z), can be used to extract biochemical profiles containing information about disease progression. The method could facilitate improved understanding of the mechanistic processes on molecular and cellular levels that are key to the development of diabetes.

The method uses vibrational microspectroscopic technology, including Fourier transform infrared (FT-IR) and Raman microspectroscopy. By using advanced statistical methods, researchers can filter out "noise" such as, for example, natural variations. This results in a better overview and allows researchers to focus on the important factors.

"This method is well-suited for studying biological samples, since it does not damage the sample, does not require external markers such as antibody labels, and can be used in microscopy settings. The method can for example be used to determine which cell types are affected in a certain tissue, where and how", says András Gorzsás, researcher at the Department of Chemistry and co-author of the article.

The researchers also describe how orthogonal projections to latent structures discriminant analysis (OPLS-DA) enables them to handle multiple variables simultaneously and thus analyse complex data from vibrational microspectroscopy of the pancreas. Using this method, which until now has been used primarily to study plant tissues, the researchers show that it is possible to discover previously unknown biochemical changes in the pancreas during disease development. In addition, previously known changes in the tissue may also be detected, but at even earlier stages of disease progression compared to what has been described by other techniques.



The digitally produced image illustrates how biochemical changes in the pancreas may be studied with the described method, without the need for labelling with external markers. The green structures correspond to an accumulation of beta-sheet proteins which indicate the development of type 2 diabetes. Image: Ulf Ahlgren

"By using this method we can create biochemical fingerprints of all changes occurring in the pancreas. The fingerprints inform us of what cell type we are looking at, which animal model it comes from and how far the disease has progressed. These fingerprints are so precise that even unknown samples can be classified if there is available reference material", says Ulf Ahlgren, Professor of Molecular Medicine and co-author of the article.

The method can be used to analyse both mice and human pancreas from the outside the organ, i.e. without the need to obtain tissue samples. Moreover, the researchers demonstrate in a transplantation experiment that pancreatic tissue (the so-called Islets of Langerhans) may be studied *in vivo*. In addition to studying mechanistic aspects of diabetes development and manifestation, the researchers hope that the method can be used to develop better prognostic and diagnostic tools for diabetes.

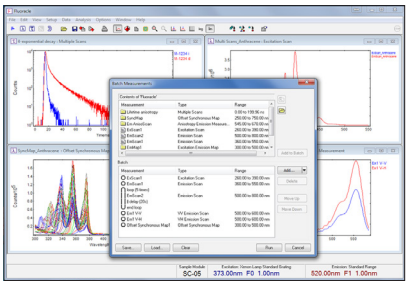
The researchers are also hopeful that their findings can lay the foundations for developing better tools for identifying cancer tissue to be surgically removed as part of pancreatic cancer treatment. The study was a collaborative effort with researchers at NTNU in Trondheim, Norway, and Karolinska institutet. The research was made possible by funding from the Swedish Research Council and the Kempe Foundations.

NEW PRODUCTS

LUMINESCENCE

Batch measurement for Fluoracle software

Edinburgh Instruments have added a new batch measurement option to their Fluoracle software for the FS5 spectrofluorometer and FLS1000 fluorescence



Batch acquisition software from Edinbugh Instruments.

spectrometer. Batch acquisition enables a sequence of multiple different measurements to be designed, including repetition, delays and loops. Batch routines can be stored and edited, saving time in long measurements and screening assays.

Edinburgh Instruments

link.spectroscopyeurope.com/29-W-053

A-TEEM combines absorbance and transmission for water analysis

Horiba Scientific has patented a new technique for molecular fingerprinting. A-TEEM™ (Absorbance and Transmission Excitation Emission Matrix) simultaneously measures absorbance and fluorescence transmission. A-TEEM fingerprints molecules with high specificity and ultra-high-sensitivity enabling researches to identify, quantify and understand dynamics of fluorescing and absorbing molecular states, and mixtures.

A-TEEM is included on Horiba's Aqualog compact, benchtop spectrometer. Aqualog is the only instrument to simultaneously measure both absorbance spectra and fluorescence excitation–emission matrices, and these EEMs are acquired up to 100× faster than with other instruments.

Horiba Scientific

link.spectroscopyeurope.com/29-W-056

IMAGING

Imaging and spectroscopic detectors for scanning electron microscopes

Horiba Scientific has introduced the Horiba CLUE Series detectors for scanning electron microscopes. This provides a scalable platform for imaging and spectroscopic analysis of nano-objects with SEM and dual SEM/FIB (focused ion beam) microscopes.

i-CLUE is a fast Cathodo-Luminescence (CL) imaging system featuring a large field of view ellipsoidal collection mirror. i-CLUE is field upgradeable to a complete spectroscopy solution, offering a first step into high sensitivity CL detection at an affordable price.

The F-CLUE rugged imaging and hyperspectral CL solution is an easy upgrade on existing configurations. It has a compact design inside (fully retractable mirror collection) and outside the specimen chamber, and its fibre-coupled spectrometer fits any microscope with a free horizontal port.

H-CLUE imaging and hyperspectral CL solution which combines a high quality parabolic mirror for DUV-VIS-NIR, direct optical coupling and a high-resolution spectrometer.

R-CLUE combines cathodoluminescence, Raman spectroscopy and photoluminescence within one compact fibre-coupled solution.

Horiba Scientific

link.spectroscopyeurope.com/29-W-066

4-megapixel, back-illuminated, scientific CMOS camera

Princeton Instruments has introduced the KURO 2048B, the newest member of the KURO family of back-illuminated, scientific CMOS (sCMOS) cameras. The KURO 2048B will be used for hyperspectral imaging, astronomy and quantum imaging. The KURO 2048B incorporates a large format 2048 × 2048 sensor with 11-µm pixels. Back-side illumination enables 100% pixel fill factor and delivers CCD-like sensitivity.

The KURO 2048B back-illuminated scientific CMOS camera from Princeton Instruments.

The frame rate for KURO 2048B at full resolution of 2048 × 2048 is 47 fps at 12 bits and 23 fps at 16 bits, with the ability to reach over 3000 fps in reduced resolution. It also has 95% quantum efficiency (QE) in the mid-range visible, 80% peak QE in the ultraviolet, a full well specification of 80,000 electrons and read noise of 1.3 e^- rms median. Additionally, programmable trigger modes synchronise camera operation with external events or light sources.

Designed for operation within the Princeton Instruments LightField® software system, the KURO 2048B camera is easy to control and can be integrated quickly in myriad imaging experiments. The 64-bit LightField imaging and spectroscopy software package provides hundreds of user enhancements, includes a powerful built-in math engine to perform live data analysis, and permits direct control from third-party packages such as LabVIEW and MATLAB. A full suite of input–output TTL signals is provided as well, making it easy to synchronise camera operation with external events or light sources.

Princeton Instruments

link.spectroscopyeurope.com/29-W-063

INFRARED

New compact FT-IR spectrometer

Bruker has launched the Alpha II, the latest in the Alpha series of compact FT-IR

spectrometers. The Alpha II features fully-automated test routines for operational qualification (OQ) and performance validation (PQ), as well as developments in its temperature-stabilised detector and high-quality IR source technology. The Alpha II has an optional, industrial grade touch panel PC and OPUS-TOUCH touch-screen software for easy and intuitive use which features built-in routine application workflows.

The ALPHA II is equipped with an IR source that utilises Bruker's CenterGlow™ technology guaranteeing a constantly high intensity and a life-time of at least five years. CenterGlow™ optimises the location of the glowing area within the source to maximise the light flux. The detector of the ALPHA II is temperature-stabilised. A wide range of plug and play QuickSnap™ sampling modules are available for the ALPHA II.

Bruker's new ALPHA II compact, FT-IR spectrometer.

Performance readiness is automatically guaranteed through permanent checks of the involved components and periodically performed test measurements verifying the specification of the ALPHA II system. For regulated pharmaceutical laboratories the ALPHA II has fully-automated test routines for operational qualification (OQ) and performance validation (PQ). Its software is compliant with 21CFRp11, and validation according to the US, European and Japanese Pharmacopeia is possible with optional, integrated NIST traceable standards. Its compact footprint, that of a lab book, makes it suitable for class rooms and teaching laboratories.

Bruker

link.spectroscopyeurope.com/29-W-062

MASS SPEC

timsTOF Pro mass spectrometer enables the PASEF method for proteomics

Bruker has introduced the timsTOF Pro system for parallel accumulation and serial fragmentation (PASEF) mass spectrometry, using proprietary trapped ion mobility spectrometry (TIMS) technology for higher-speed, higher-sensitivity and robust shotgun proteomics. The timsTOF Pro also has advantages for quantitative proteomics workflows.

The front-end TIMS analyser is optimised for higher-speed shotgun proteomics from smaller sample amounts. Its dual TIMS geometry allows ions to be accumulated in parallel in the first TIMS section, and after an additional TIMS separation step in real time, the ions are released from the second TIMS section

Bruker's timsTOF Pro for parallel accumulation and serial fragmentation.

NEW PRODUCTS

for MS/MS fragmentation. The PASEF capability delivers higher-sensitivity and higher-speed shotgun proteomics without loss of mass resolution. Higher scan speeds result in lower mass resolution in FT-based MS technology commonly used for shotgun proteomics. These limitations are eliminated by PASEF, allowing for a duty cycle near 100% with high sensitivity while maintaining ultra-high mass resolution for both the precursor and the product ions.

Quantitative proteomics is a key area of research in proteomics. The timsTOF Pro delivers over four orders of magnitude of dynamic range with low peptide loads (100–200 ng), which makes it suitable for proteomics on the small cell populations and low sample amounts often encountered in biological and clinical research.

Bruker

link.spectroscopyeurope.com/29-W-057

VACUUM

Air-cooled pumps for LC-MS and ICP-MS

Edwards has introduced the air-cooled nXLi dry vacuum pump range, which provides a small footprint with quiet operation. The intelligent drive enables the system to achieve an enhanced performance plateau from single-phase power supply, with consistent worldwide operation. There is a choice of control options, with either manual, simple remote or serial communications control as standard. The pump mechanism is

nXLi air-cooled dry vacuum pumps.

non-contacting and oil-free to provide extended operation with maximum up time, up to five years' service life.

During the initial chamber pump down the intelligent transient overload protection allows a higher power draw for a short period of time, this enables the pump to evacuate the chamber from atmospheric pressure to operating pressure. This higher initial power boost enables the pump to overcome initial gas loads and once operating pressure is achieved, less power is required.

This new single-phase pump is available in two options, nXL110i and nXL200i, both optimised for LC-MS and ICP-MS, and capable of handling gas loads of up to 25 slm.

Edwards

link.spectroscopyeurope.com/29-W-054

RMS

Wide range wavelength reference

A new Certified Reference Material from Starna gives 14 well-defined, evenly spaced and proven interference-free peaks for spectrophotometer wavelength qualification covering a wavelength range from 240 nm to 795 nm. Rare earth oxide solutions have been used for wavelength calibration for many years and are

Starna's new CRM for wavelength qualification.

accepted for this purpose by all the pharmacopoeias. This reference combines holmium and didymium (neodymium and praseodymium) and covers the most popular wavelengths in UV and visible spectrophotometry. Because peak values measured on sharp peaks are wavelength dependent, the Calibration Certificate accompanying each reference gives actual values measured at nine different bandwidths from 0.1 nm to 5 nm, covering most operating situations. Certified values are all fully traceable to NIST, and Starna is accredited to both ISO Guide 34 as a Reference Material producer and ISO 17025 (0659) as a Calibration Laboratory. Like all Starna reference materials, this reference carries a lifetime guarantee.

Starna Scientific

link.spectroscopyeurope.com/29-W-065

SOFTWARE

Updated data management software

Sensologic have introduced version 2.0 of their SL Data Manager software for spectroscopy. Several new functions have been introduced which will be helpful in management, visualisation and handling of data for quantitative and qualitative modelling with the SL Calibration Wizard and the soon-to-be-released SL Classification Wizard.

The new features include:

Advanced graphics options, e.g. for improved visual evaluation of spectra series or libraries—including direct application of transformations—and for graphical spectra selection. Also available is the ability to view all spectra assigned to a sample in a sample spectra graph, or display a selected single spectrum.

Easy removal of database entries which are no longer in use, e.g. applications, methods, models, samples or series etc.

Simple preparation of transformation batches, using additional transformation algorithms, e.g. Savitzky–Golay, Multiplicative Scatter Correction or Kubelka–Munk. New transformations can be applied immediately to series and library graphics.

Helpful additional information in the info pane, e.g. for series, the available properties are shown, including the number of assigned samples and a property value histogram; for calibration models, the used series and wavelength range are shown.

Extended data management of libraries (Create, Edit, Copy, Delete) for development of qualitative models with the new SL Classification Wizard.

Improved usability, for example by easy availability of model reports, a new

"Save as" function for duplicating models or methods, and simple access to the validation status of methods.

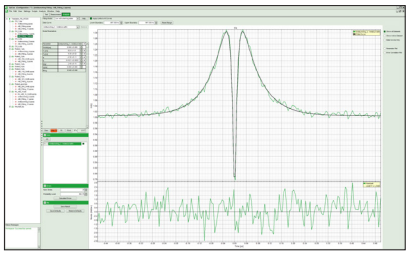
Sensologic

link.spectroscopyeurope.com/29-W-061

Software for the analysis of quantum correlations

PicoQuant has released the QuCoa software package, an integrated solution for data acquisition and analysis using the unique T2 time-tagging mode of PicoQuant's TCSPC electronics. QuCoa is aimed at research areas relying on coincidence detection such as Hanbury–Brown–Twiss setups to study single photon sources ($g^{(2)}$ / antibunching), quantum key distributions (QKD), general quantum optics or the study of entanglement using Hong–Ou Mandel setups (HOM). The software offers special analysis routines for coincidence correlations as well as for coincidence counting applications.

The QuCoa software's correlator permits the correlation of the absolute arrival times of photons in real time. This allows, for example, the quality of an antibunching curve to be assessed during the measurement. The $g^{(2)}(0)$ value and count rates on all detectors are continuously calculated and displayed. Several established models such as



PicoQuant's QuCoa is an integrated solution for data acquisition and analysis.

single emitter with or without shelved state or pulsed excitation based on exponential decays, including the influence of the limited detector resolution as well as correlated and uncorrelated background can be fitted to the measured data. The fitting models support both pulsed and cw excitation.

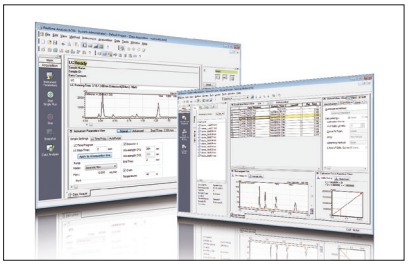
The QuCoa software also provides an easy to use event filter feature for coincidence counting applications. Filters can be constructed via a simple and intuitive graphical user interface which permits combining detection channels and marker signals in a user-defined time window using logical operations (AND, OR, NOT). The filters can be applied both during data acquisition and offline analysis mode. By defining time gates, the user can restrict coincidence detection to specific time ranges.

PicoQuant

link.spectroscopyeurope.com/29-W-060

Shimadzu have updated their LabSolutions software

Shimadzu has released a new version of its LabSolutions analytical data system. This incorporates supplementary functions to comply with data integrity regulations and to support development and quality inspection procedures in pharmaceutical companies. The LabSolutions software family features an operating environment providing complete data management to ensure secure information in networked laboratories. In the pharmaceutical industry, compliance with regulations and guidelines as well as the proper, efficient management of instruments and analytical data is



New version of Shimadzu's LabSolutions analytical data system.

required. The LabSolutions analysis data system meets these needs.

Amongst the new features is peak separation based on differences in the UV spectral pattern. An improved detector function (i-PDeA II) uses differences in the UV absorption spectral patterns of target analytes to reliably separate multiple components that elute at more or less the same time. Hard-to-separate peaks can be shown in a single trace, simplifying detection and at the same time enabling analysis and quantitative calculations with respect to the resulting data. In addition to pharmaceuticals, this revolutionary solution can be used for the analysis of clinical specimens, chemical products, natural products, functional foods and residual pesticides.

Shimadzu

link.spectroscopyeurope.com/29-W-058

NEW PRODUCTS

SURFACE

Multi-technique surface analysis system

The new Thermo Scientific Nexsa surface analysis system is designed easily to integrate multiple analytical techniques in a single compact, fully-automated surface analysis instrument. The Nexsa system combines the high throughput and sensitivity of the Thermo Scientific K-Alpha$^+$ XPS system with the multi-technique capabilities of the Thermo Scientific ESCALAB Xi$^+$ XPS microprobe. Users of the Nexsa system can add complementary techniques, such as Raman spectroscopy, ion scattering spectroscopy (ISS), reflected electron energy loss spectroscopy (REELS) and UV photoelectron spectroscopy (UPS), to generate multiple measurements from the same point on the sample, without repositioning. Integration of multiple analytical techniques is designed to allow users to conduct true correlative analysis, unlocking the potential for further advances in microelectronics, ultra-thin films, nanomaterials development and many other fields.

Additional features of the Thermo Scientific Nexsa surface analysis system include: a new small spot x-ray source, designed to improve the imaging capabilities compared to previous instruments; the ability to transfer air-sensitive materials, such as lithium-ion battery materials, into the instrument without exposure to the atmosphere either via an optional vacuum transfer module or integration with an external glove box; and optional integration with the patented Thermo Scientific MAGCIS dual mode ion source to enable depth profiling of soft materials, such as polymers.

Thermo Fisher Scientific

link.spectroscopyeurope.com/29-W-059

The Thermo Scientific Nexsa surface analysis system.

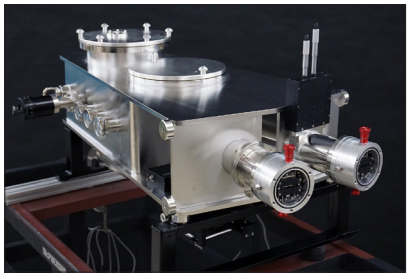
UV/VIS

Imaging spectrometer for VUV

Certain McPherson spectrometers can now measure vertical spatial profile in addition to dispersed wavelengths. They operate in the vacuum ultraviolet (VUV) to view plasma impurity emission lines from 300 Å to 3200 Å.

McPherson have announced that their one- and three-metre normal incidence spectrometers are now capable of imaging. The ability to measure both spatial and wavelength resolution is important in many applications. One example, diagnostics of impurity profile in edge plasma of large magnetically confined tokomak devices used in fusion research.

With the addition of a carefully adjusted horizontal aperture in the optical path, these spectrometers can now measure vertical spatial profile in addition to dispersed wavelengths. They operate in the vacuum ultraviolet (VUV) and view plasma impurity emission lines in the wavelength range of 300–3200 Å. The new spectrometers can be built for high vacuum (10^{-6} Torr) and also ultra-high vacuum (10^{-10} Torr). They can be equipped with microchannel plate intensifiers or CCD detectors. Other solar blind and single channel detectors are available for scanning applications. The instruments are delivered with precise wavelength calibration and repeatable means to set the observed vertical range. Users may choose to calibrate the sensitivity of VUV spectrometer systems using calibrated light sources and/or detectors too.



Imaging comes to certain McPherson VUV spectrometers.

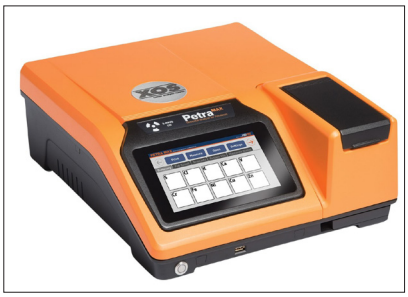
McPherson

link.spectroscopyeurope.com/29-W-064

X-RAY

ASTM D4294 analyser

XOS were showing their Petra MAX D4294 analyser at Pittcon and we reported on this in issue 29/2. The production version has now been released with some significant performance improvements. Petra MAX offers combined analysis of 13 elements from P to Zn, with high-precision (5.7 ppm) sulphur analysis in addition to rapid monitoring of elements like Ca, Fe, K, Ni and V at sub-ppm levels. Test methods for measuring sulphur content, like ASTM D4294 and ISO 8754, have become critical for assessing the value of crude oil. Petra MAX is based on XOS' patented high definition X-ray fluorescence, which uses doubly curved crystal optics coupled with a high-performance silicon drift detector and an intense monochromatic excitation beam. This reduces background noise and increases signal-to-noise output, enabling low detection limits and high precision without the need for consumable helium gas, a vacuum pump or extensive sample preparation.



The Petra MAX XRF analyser from XOS.

XOS

link.spectroscopyeurope.com/29-02-091

DIARY

Conferences

2017

7–10 November, Prague, Czech Republic. **8th International Symposium on Recent Advances in Food Analysis (RAFA2017).** jana.hajslova@vscht.cz, http://www.rafa2017.eu.

8–10 November, Singapore, Singapore. **9th Electronic Structure and Processes at Molecular-Based Interfaces (ESPMI 9).** June Chan, junechan@nus.edu.sg, http://www.physics.nus.edu.sg/espmi9/.

12–17 November, Matsue-City, Shimane, Japan. **7th Asia-Pacific Winter Conference on Plasma Spectrochemistry (APWC).** Takafumi Hirata, Koyto University, Kitashirakawa, Oiwakecho, Sakyo-ku, Kyoto 606-8502, Japan. hrt1@kueps.kyoto-u.ac.jp, http://www2.es.titech.ac.jp/okino/pdf/17APWCposter.pdf.

12–16 November, Minneapolis, MN, United States. **SETAC North America 38th Annual Meeting.** SETAC North America, setac@setac.org, https://msp.setac.org/.

12–16 November, San Diego, California, United States. **American Association of Pharmaceutical Scientists (AAPS) 2017 Annual Meeting.** AAPS, 2107 Wilson Blvd, Suite 700, Arlington, Virginia 22201-3042, USA, aaps@aaps.org, http://www.aaps.org/annualmeeting/.

13–15 November, Plainsboro, New Jersey, United States. **Eastern Analytical Symposium and Exposition (EAS 2017).** askEAS@eas.org, http://www.eas.org.

26 November–1 December, Boston, MA, United States. **Materials Research Society 2017 Fall Meeting.** Materials Research Society, 506 Keystone Drive, Warrendale, PA 15086-7573, USA, info@mrs.org, http://www.mrs.org/fall2017.

10–13 December, Águas de Lindóia, Brazil. **5th Brazilian Meeting on Chemical Speciation (EspeQBrasil 2017).** espeqbrasil2017@rc.unesp.br, http://www.unesp.br/portal#!/cea/home/espeqbrasil2017/espeqen/the-meeting/.

11–15 December, New Orleans, Louisiana, United States. **American Geophysical Union AGU 2017 Fall Meeting.** meetinginfo@agu.org, http://www.agu.org/meetings.

21 December, London, United Kingdom. **IRDG Christmas Meeting and Poster Session 2017.** http://www.irdg.org/meetings/future-meetings/.

2018

8–13 January, Amelia Island, Florida, United States. **2018 Winter Conference on Plasma Spectrochemistry.** Ramon Barnes, wc2018@chem.umass.edu, http://icpinformation.org.

10–12 January, Cesena, Italy. **5th International Conference on Foodomics: from Data to Knowledge (ICF2018).** foodomics2018@foodomics.org, http://www.foodomics.eu/.

24–26 January, Cardiff, United Kingdom. **15th International Symposium on Hyphenated Techniques in**

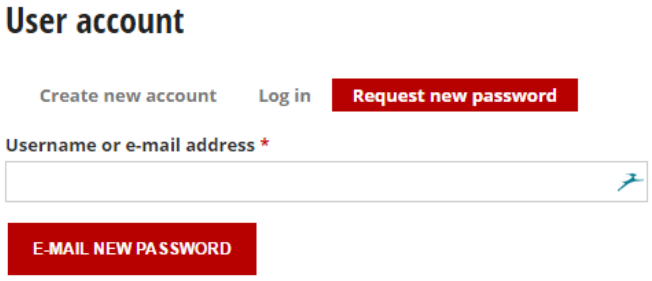
A New Website for *Spectroscopy Europe*

We have just launched a new website which works well on all devices from large screens to smartphones. The URL remains www.spectroscopyeurope.com.

We have migrated all users/readers from the old website but it was impossible, due to built-in security, to transfer users' passwords. I hope you have received an e-mail with a link to log in and reset your password. If you have not or are having any difficulty, here is how to log into the new site.

1) Use the Lost Password facility

From any page, click LOGIN in the main menu, and then "Request new password" to the right of the white-on-red "Log in". Enter your e-mail address and you will receive an e-mail with a "one-time" link that you can use to log in and then change the password to one you want to use. Please also check your details whilst you are in your Profile.

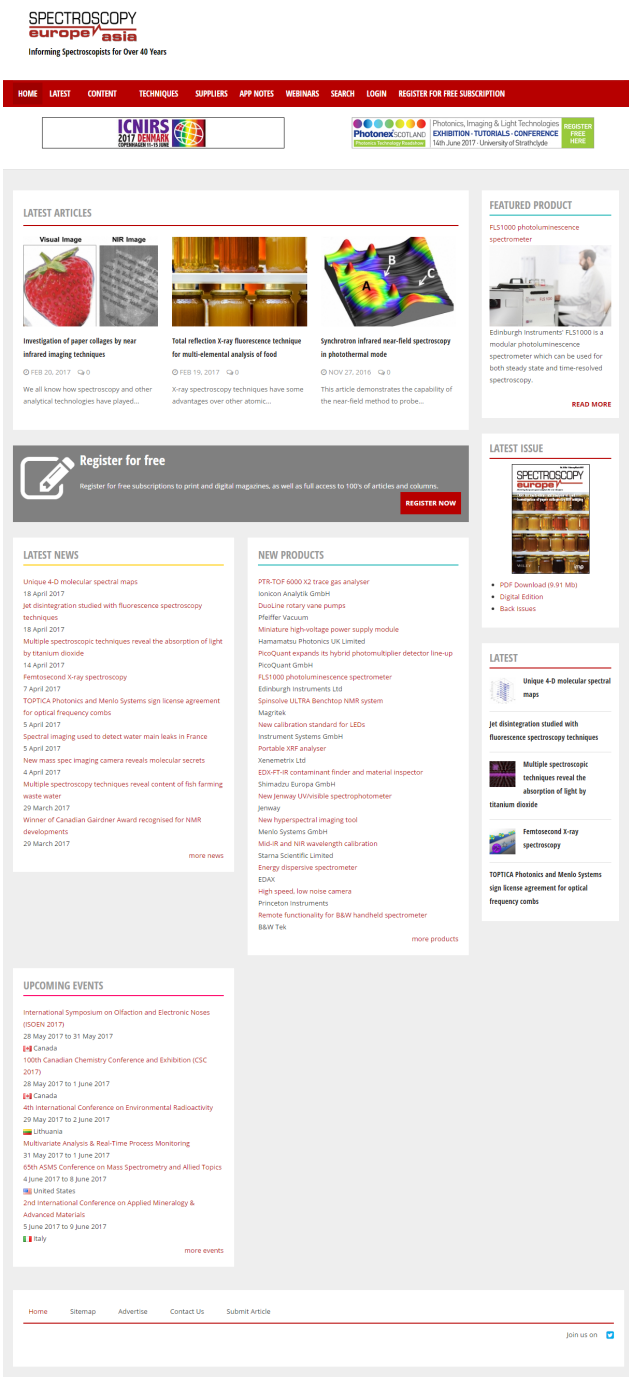


The e-mail usually arrives within seconds; if you do not see it, check your spam folder(s): these types of e-mails are often mistaken for spam.

If this does not work, perhaps because your e-mail address has changed:

2) Ask for help

Just e-mail katie@impublications.com who will check if you have an account and help you log in.



Of course, if you or a colleague don't have an account, you can quickly create one and ensure your continued access to the print version of *Spectroscopy Europe* as well as online access.

www.spectroscopyeurope.com

Chromatography and Separation Technology HTC-15. lucinda@ilmexhibitions.com, http://htc-conference.co.uk/.

31 January, Jealott's Hill, United Kingdom. **4th BMSS Ambient Ionisation SIG Meeting.** Lisa Sage, bmssadmin@btinternet.com, http://www.bmss.org.uk/aims2018/aims2018.shtml.

19–24 February, Seattle, Washington, United States. **American Academy of Forensic Sciences (AAFS) 70th Annual Scientific Meeting.** www.aafs.org.

26 February–2 March, Saint Petersburg, Russia. **Winter Symposium on Chemometrics (WSC-11).** Irina Yaroshenko, wsc11@chemometrics.ru, http://wsc.chemometrics.ru/wsc11/.

18–22 March, New Orleans, United States. **255th American Chemical Society National Meeting.** natlmtgs@acs.org, www.chemistry.org.

22–23 March, London, United Kingdom. **11th Edition of International Conference on Proteomics.** proteomics@eurosciconmeetings.com, http://proteomics.euroscicon.com/.

26–29 March, Santa Fe, New Mexico, United States. **International High Power Laser Ablation Symposium (HPLA 2018).** Amy Walker, awalker@blue-52productions.com, www.usasymposium.com/hpla.

8–13 April, Vienna, Austria. **European Geosciences Union (EGU) General Assembly 2018.** EGU Executive Office, secretariat@egu.eu, www.egu.eu.

14–18 April, Chicago, IL, United States. **Annual Meeting American Association for Cancer Research.** AACR, aacr@aacr.org, www.aacr.org.

15–19 April, Estepona (Málaga), Spain. **6th International Congress on Operando Spectroscopy (Operando VI).** Secretary, info@operandoconference.com, http://www.operandoconference.com/index.

16–18 April, Berlin, Germany. **Fourth International Glow Discharge Symposium (IGDSS2018).** Peter Robinson, pete@masscare.co.uk, www.ew-gds.com.

17–20 April, Glasgow, United Kingdom. **IRDG Martin & Willis Prize Meeting 2018.** http://www.irdg.org/meetings/future-meetings/.

17–20 April, Glasgow, United Kingdom. **Spring SciX2018.** Duncan Graham, duncan.graham@strath.ac.uk, https://www.scixconference.org/scix-home/future-conferences.

21–25 April, San Diego, CA, United States. **Experimental Biology 2018.** Experimental Biology, eb@faseb.org, http://experimentalbiology.org.

28 April–2 May, Prague, Czech Republic. **33rd Congress International Society for Advancement of Cytometry.** infor@cytoconference.org, http://cytoconference.org.

27–30 May, Lecce, Puglia, Italy. **CMA4CH Meeting 7th edition Multivariate Analysis and Chemometry: an essential support for Environment and Cultural Heritage.** infocma4ch@uniroma1.it, http://www.cma4ch.org/index2.html.

27–31 May, Edmonton, Canada. **101st Canadian Chemistry Conference.** www.csc2018.ca.

3–7 June, San Diego, CA, United States. **66th ASMS Conference on Mass Spectrometry.** office@asms.org, www.asms.org.

10–15 June, Glasgow, Scotland, United Kingdom. **10th International Conference on Clinical Vibrational Spectroscopy (SPEC-2018).** http://spec2018.com/.

10–13 June, Leon, Norway. **9th Nordic Conference on Plasma Spectrochemistry.** Yngvar Thomassen, yngvar.thomassen@stami.no, www.nordicplasma.com.

17–20 June, Seatlle, WA, United States. **International Association for Spectral Imaging (IASIM) Conference 2018.** http://www.iasim.net.

26–29 June, Pau, France. **14th European Workshop on Laser Ablation (EWLA 2018).** Christophe Pecheyran, https://ewla2018.sciencesconf.org/.

23–25 July, Milan, Italy. **2nd World Congress on Pharmaceutical and Chemical Sciences.** pharma@colossalfacet.com, http://colossalfacet.com/pharma-conference/.

19–23 August, Boston, MA, United States. **256th American Chemical Society National Meeting.** natlmtgs@asc.org, www.chemistry.org.

26–30 August, Liverpool, United Kingdom. **7th EuCheMS Chemistry Congress.** www.euchems.eu.

26–29 August, Toronto, Ontario, Canada. **132nd Association of Official Agricultural Chemists (AOAC) International Annual Meeting and Exposition.** meetings@aoac.org, www.aoac.org.

10–13 September, Cambridge, United Kingdom. **39th BMSS Annual Meeting.** Lisa Sage, bmssadmin@btinternet.com, http://www.bmss.org.uk/bmss2018/bmss2018.shtml.

21–26 October, Atlanta, GA, United States. **45th Annual Conference of Federation of Analytical Chemistry and Spectroscopy Societies (SciX2018).** facss@facss.org, http://www.scixconference.org.

4–8 November, Washington, DC, United States. **American Association of Pharmaceutical Scientists (AAPS) 2018 Annual Meeting.** aaps@aaps.org, www.aaps.org/annualmeeting/.

4–8 November, Indianapolis, Indiana, United States. **2018 Geological Society of America (GSA) Annual Meeting.** meetings@geosociety.org, www.geosociety.org/meetings/.

4–8 November, Sacramento, CA, United States. **SETAC North American 39th Annual Meeting.** setac@setac.org, www.setac.org/.

2019

8–12 July, Auckland, New Zealand. **International Conference on Advanced Vibrational Spectroscopy (ICAVS10).** ICAVS Secretariat, Podium Conference Specialists, 2661 Queenswood Drive, Victoria, BC, Canada, V8N 1X6. http://

SPECTROSCOPY europe READER SERVICE

Use this page to register for a free subscription to *Spectroscopy Europe* You can also register online at the address below.

Online version at http://www.spectroscopyeurope.com/register

Your Details

Title (Professor/Dr etc.):	City:
First Name:	State/Region:
Last Name:	Postal Code:
Job Title:	Country:
Organisation Name:	E-mail:
Address:	

☐ Please send me/continue to send me a free copy of *Spectroscopy Europe*. Signed: _____

Field of Work:
(please tick ALL that apply)

- ☐ Agriculture
- ☐ Analytical Chemistry
- ☐ Biotechnology
- ☐ Chemicals
- ☐ Electronics/Semiconductors
- ☐ Energy and Fuels
- ☐ Environmental
- ☐ Food
- ☐ Genomics and other -omics
- ☐ Instrumentation
- ☐ Life Science
- ☐ Materials Science
- ☐ Medical Sciences
- ☐ Metals and Minerals
- ☐ Nanotechnology
- ☐ Pharmaceuticals
- ☐ Polymers and Peptides
- ☐ Security and Forensics
- ☐ Water

Techniques:
(please tick ALL that apply)

- ☐ Atomic Absorption
- ☐ Atomic Emission
- ☐ Chemometrics
- ☐ Computers/Automation
- ☐ ICP/MS
- ☐ Infrared
- ☐ Laser Spectroscopy
- ☐ Luminescence/Fluorescence
- ☐ Mass Spectrometry
- ☐ Microscopy and Imaging
- ☐ MRI
- ☐ Near Infrared
- ☐ NMR, ESR, EPR
- ☐ Photonics and Optics
- ☐ Raman
- ☐ Separation Science
- ☐ Surface Analysis
- ☐ UV/Vis
- ☐ X-Ray Diffraction
- ☐ X-Ray Spectrometry
- ☐ None

Area of employment:
(please tick ONE only)

- ☐ Industry
- ☐ Independent Lab
- ☐ University/Institute
- ☐ Hospital
- ☐ Government
- ☐ Research Inst/Foundation
- ☐ Other_____

Job function: *(please tick ONE only)*

- ☐ Analyst
- ☐ Quality Control/Assurance
- ☐ Research Scientist
- ☐ Lab Manager
- ☐ University Professor
- ☐ Engineering/Design
- ☐ Manufacturing/Processing
- ☐ Teaching
- ☐ Marketing/Sales
- ☐ Other_____

Do you recommend, specify or authorise the purchase of spectroscopic instrumentation or services: ☐ Yes ☐ No

Your Personal Data (we will never pass your personal details to any third party)

John Wiley & Sons Ltd and IM Publications LLP will use the information you have provided to fulfil your request. In addition, we would like to:

1. Contact you by post with information and offers from relevant companies (your details are never disclosed to them). Tick here if you do NOT want to receive this: ☐
2. Contact you by e-mail with information and offers from relevant companies (your e-mail address and details are never disclosed to them). Tick if you WANT to receive this: ☐
3. Your registration to this website includes a regular e-mail newsletter alerting you to the latest content. Tick if you do NOT want to receive this: ☐

Now fax this page to +44-1243-811711, or return by mail to: *Spectroscopy Europe*, 6 Charlton Mill, Charlton, Chichester, West Sussex PO18 0HY, UK.

DIARY

www.icavs.org/contact/, http://www.icavs.org/2019-conference/.

15–20 September, Gold Coast, Australia. **NIR-2019.** www.nir2019.com.

13–18 October, Palm Springs, United States. **46th Annual Conference of Federation of Analytical Chemistry and Spectroscopy Societies (SciX2019).** facss@facss.org, http://www.scixconference.org.

12–18 January, Tucson, Arizona, United States. **2020 Winter Conference on Plasma Spectrochemistry.** wc2020@chem.umass.edu, http://icpinformation.org.

Courses

2017

6–9 November, Berlin, Germany. **15th European Short Course on Principles and Applications of Time-resolved Fluorescence Spectroscopy.** Nicola Kasse, trfcourse@picoquant.com, http://www.picoquant.com/trfcourse.

7–8 November, Birmingham, United Kingdom. **Metabolomics with the Q Exactive.** David Epps, d.epps@bham.ac.uk, http://www.birmingham.ac.uk/facilities/metabolomics-training-centre/courses/q-exactive.aspx.

7–8 November, London, United Kingdom. **Introduction to Multivariate Data Analysis (London).** Joseph McCurley, http://camo.com/contact-form.html, http://camo.com/training/more/en/mva.html?id=764&tid=7&po=1.

9 November, Breda, Netherlands. **Free Seminar: An Introduction to Infra-Red, Raman Spectroscopy and Microscopy.** Dlangir Cordero, dlangir.cordero@thermofisher.com, www.thermofisher.com/MeetTheExpert.

13–14 November, Berlin, Germany. **Introduction to Multivariate Data Analysis (Berlin).** Jens Oestreich, http://camo.com/contact-form.html, http://camo.com/de/training/multivariate-datenanalyse.html?id=778&tid=7&po=1.

22–24 November, Norderstedt, Germany. **Chemometric Spectroscopy: Basics and Multivariate Analysis for Quantitative and Qualitative Applications.** info@sensologic.de, www.sensologic.de/download/SL-Training-Registration_Nov-2017.pdf.

22–24 November, Utrecht, Netherlands. **Multivariate Analysis of Spectroscopic Data (Utrecht).** Joseph McCurley, http://camo.com/contact-form.html, http://camo.com/training/more/en/spectroscopy.html?id=793&tid=20&po=1.

6–8 December, Birmingham, United Kingdom. **Multiple Biofluid and Tissue Types, from Sample Preparation to Analysis Strategies for Metabolomics.** David Epps, d.epps@bham.ac.uk, http://www.birmingham.ac.uk/facilities/metabolomics-training-centre/courses/sample-analysis.aspx.

14–15 December, Birmingham, United Kingdom. **Metabolite Identification with the Q Exactive and LTQ Orbitrap.** David Epps, d.epps@bham.ac.uk, http://www.birmingham.ac.uk/facilities/metabolomics-training-centre/courses/metabolite-identification.aspx.

17–19 January, Heidelberg, Germany. **Brillouin Microscopy: Emerging Tool for Probing Mechanical Properties of Living Cells.** Adela Valceanu, adela.valceanu@embl.de, https://www.embl.de//training/events/2018/BRI18-01/index.html.

Exhibitions

2018

25 February–1 March, Orlando, FL, United States. **68th Pittcon 2018.** pittconinfo@pittcon.org, http://pittcon.org.

10–13 April, Munich, Germany. **Analytica 2018.** info@analytica.de, http://www.analytica.de/.

11–15 June, Frankfurt on the Main, Germany. **ACHEMA 2018.** achema@dechema.de, http://www.achema.de.

31 October–2 November, Shanghai, China. **Analytical China 2018.** Barbara Kals, barbara.kals@messe-muenchen.de, https://tinyurl.com/yd99eqox.

2019

7–9 May, Beijing, China. **AchemAsia 2019.** China National Convention Center, Tianchen East Road, Chaoyan District, Beijing 100105, China, http://www.cnccchina.com/.

ADVERTISERS INDEX

Have You Heard?

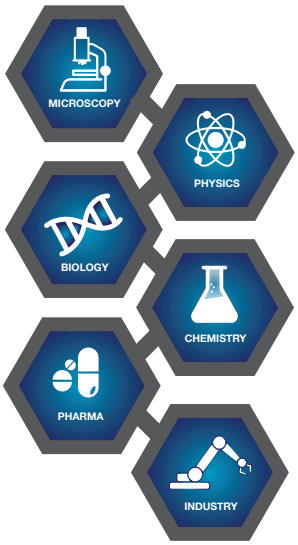
"Such ingenious CUBES!
That cooled CCD is impressive!
Look at the aberration-free spectra!
So small and easy to use!"

FERGIE's footprint is smaller than the page you're reading now!

High-performance spectroscopy can be easy!

How? Simply plug in the new Princeton Instruments FERGIE™ and start your work right away…
No calibrating. No aligning. No guessing.

Best of all, true novices and seasoned spectroscopists alike can easily configure and operate FERGIE for popular applications such as **Raman, absorption, microspectroscopy,** and **fluorescence.**



Join the many **scientists, educators,** and **engineers** worldwide who are embracing this revolution in spectroscopy!

"I can honestly say FERGIE is changing the way we do spectroscopy in our lab!"

– Dr. Mark Waterland, Massey University, New Zealand

Princeton Instruments

Global Sales & Support
www.princetoninstruments.com
info@princetoninstruments.com
tel: +1 609.587.9797