Vol. 32 No. 6 December/January 2020

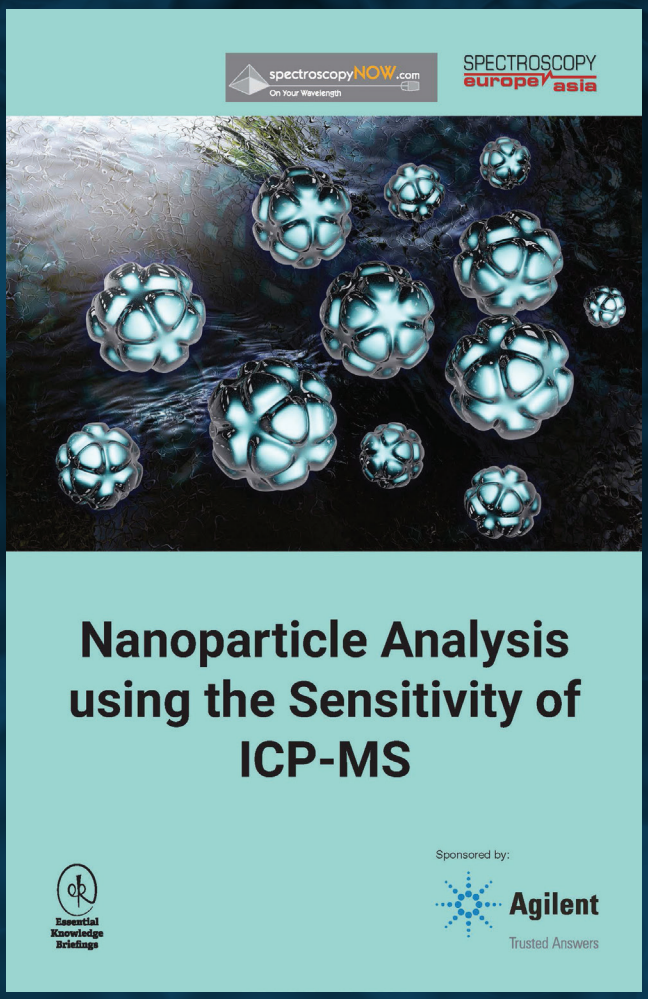
SPECTROSCOPY europe

Informing European spectroscopists for over 40 years

Raman in the nuclear field
Software advances for 4D-Proteomics
Gold IS special: and needs special sampling

WILEY

imp publications



New from Wiley

Nanoparticle Analysis using the Sensitivity of ICP-MS

This essential knowledge briefing provides an introduction to the use of ICP-MS for nanoparticle analysis and how the technique works for nanoparticles. The briefing advantages, as well as highlighting practical applications. The briefing goes on to outline the parameters that affect performance and how to get the best results and looks at some of the challenges of working with ICP-MS and how to address them.

Download for free today
at **www.essentialknowledgebriefings.com**

WILEY

19 - 534705

EDITORIAL

Important developments: please read

From 2021, *Spectroscopy Europe* will increase the frequency of publication to eight issues a year and each issue will contain more material. However, we will not be producing any more printed issues. Increases in international distribution costs, in particular, have made print copies unviable. I have also felt increasingly uncomfortable about the environmental impact of producing tons of printed paper and mailing it wrapped in plastic film. It seems that this view is shared by an increasing number of readers as well, who have already switched to digital-only subscriptions.

I am excited about *Spectroscopy Europe*'s future as a digital-only publication and to bringing you a greater range of articles and news. I have two requests.

First, we need to find out how best to deliver the new digital *Spectroscopy Europe* to you, and to find out what you like and, just as importantly, don't like about what we publish. I would be most grateful if you would spend a couple of minutes completing the online survey at spectroscopyeurope.com/2021-survey.

Second, whilst you are online, please log in and go to your profile and check it: particularly that your e-mail address is up to date. There is a password reminder option if you need that, but if you are unable to log in or have difficulties, feel free to e-mail katie@impopen.com who will sort you out. Naturally, any further communication with you will be via e-mail, so it is *vital* that we have your correct details.

There is one further change, but one that should not affect readers. For the past 28 years, *Spectroscopy Europe* has been published as a joint venture between Wiley and IM Publications. From 2021, IM Publications will be the sole publisher. If you are a company or organisation looking to advertise in *Spectroscopy Europe*, please contact me (ian@impopen.com) in future.

I am very excited about continuing the journey I started in 1982 when I took on the role of Editor of *European Spectroscopy News* (*ESN*). That ground-breaking publication has evolved through different titles and publishers to become the digital *Spectroscopy Europe* of 2021. That must be over 225 issues that I have produced; I'm looking forward to many more!

As the authors of the Sampling Column point out, Gold is always special. However, with its high value and the extremely heterogeneous nature of the rock it is found in, correct sampling is essential. Find out more on page 21. Recovered gold nuggets from Conglomerate deposits, Karratha region of Western Australia. Photograph courtesy of Artemis Resources Ltd.

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

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

Paid subscriptions
IM Publications Open, 6 Charlton Mill, Charlton, Chichester, West Sussex PO18 0HY, United Kingdom. Tel: +44-1243-811334, Fax: +44-1243-811711,
E-mail: katie@impopen.com

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. 32 No. 6
December/January 2020

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 2020. 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 Warners Midland

WILEY

imp
impublications

SHIMADZU
Excellence in Science

Maximum Performance – minimum footprint the AA-7000 family

Drive-in

The AA-7000 series of atomic absorption spectrophotometers covers the fully automatic multi-element analysis in flame and graphite furnace mode. The drive mechanism changing the atomizer units by software operation minimizes switching times between flame and furnace measurements. This is a truly efficient "drive-in" solution.

Family of three systems
featuring flame or graphite furnace analysis as well as both modes in one system

Most-sensitive detector
covering high concentrations as well as ultra-trace samples

Excellent system performance
brings world class sensitivity and long time stability

Numerous accessories
upgrade the functionalities to fully automated multi-element analysis

www.shimadzu.eu/aa-7000

NEWS

Non-invasive breath test for COVID-19 using GC-IMS

Working with partners at the IMSPEX Group as well as the Royal Infirmary of Edinburgh and Germany's Klinikum Dortmund Hospital, a research team led by Loughborough University has been able to identify candidate biomarkers present in the breath of someone affected by Covid-19. Utilising technologies developed by GAS GmbH as part of the TOXI-Triage project, the team has demonstrated how these markers can be used to rapidly distinguish Covid-19 from other respiratory conditions at point of need, such as an emergency department, a workplace or a care setting, with no laboratory support.

Ninety-eight patients were recruited for the feasibility study, of whom 31 had Covid-19. Other diagnoses included asthma, exacerbation of asthma and COPD, viral pneumonia, other respiratory tract infections and cardiac conditions. Participants gave a single breath-sample for volatile organic compounds analysis by gas chromatography/ion mobility spectrometry (GC-IMS). This analysis identified aldehydes (ethanal, octanal), ketones (acetone, butanone) and methanol that discriminated COVID-19 from other conditions.

Speaking about the feasibility study, Paul Thomas of Loughborough University, said: "We are hugely encouraged by these findings. Employing tried and tested techniques used during the TOXI-Triage project, suggests that Covid-19 may be rapidly distinguished from other respiratory conditions."

"To develop this technique further larger studies are required, together with complementary GC-MS studies, to build on the data collected so far. If shown to be reliable, it offers the possibility for rapid identification or exclusion of Covid-19 in emergency departments or primary care that will protect healthcare staff, improve the management of patients and reduce the spread of Covid-19."

Speaking about their involvement with the project, Emma Brodrick, Systems Application Manager at IMSPEX said: "Currently the two leading tests for Covid-19—antigen detection and PCR—both utilise invasive means of taking samples, which can be uncomfortable for the patient and may discourage some from going to get a test they desperately need. We are excited to be working with NHS Trusts in Scotland, Klinikum Dortmund in Germany and Loughborough University to develop a minimally invasive test, that produces results rapidly, indeed in TOXI-Triage our results were within one minute."

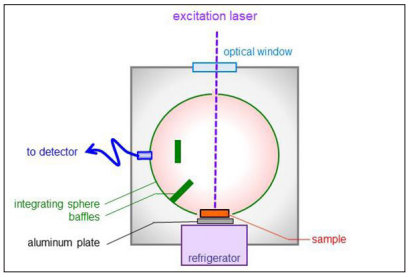
The research has been published in *EClinicalMedicine* (doi.org/ghgv52).

The BreathSpec device was used during the TOXI-Triage field trials last year in Finland for a CBRN exercise. Photo by Andrew Weekes

Photoluminescence spectroscopy of semiconducting crystals

Tohoku University researchers have improved a method for probing semiconducting crystals using omnidirectional photoluminescence (ODPL) spectroscopy to detect defects and impurities. "Our technique can test materials at very low temperatures and can find even small amounts of defects and impurities", says Tohoku University materials scientist Kazunobu Kojima.

Kojima and his colleagues demonstrated their approach using gallium nitride crystals. Gallium nitride is a semiconducting crystal that has been used in energy-saving light-emitting diodes (LEDs) since the 2000s. It has interesting optic and electronic properties, making it attractive for many applications, including power-switching devices in electric vehicles. But it can develop defects and impurities during its fabrication, which can affect performance. Currently available methods for testing these crystals are expensive or too invasive. ODPL spectroscopy, on the other hand, is a non-invasive technique that can test the crystals, but only at room temperature. Being able to change the crystal's temperature is important to properly test its properties.



The sample is placed outside the integrating sphere and onto an aluminium plate connected to a cooling device. © Tohoku University

Kojima and his colleagues found a way to set up an ODPL instrument so that the crystal can be cooled. The process involves placing a gallium nitride crystal on an aluminium plate connected to a cooling device. This is placed under an integrating sphere and external light is shone through the sphere onto the

crystal, exciting it. The crystal emits light back into the sphere in order to return to its initial unexcited state. The two lights, from the external source and the crystal, are integrated within the sphere and measured by a detector. The result reveals the crystal's "internal quantum efficiency", which is reduced if it contains defects and impurities, and can be measured even at very low temperatures.

The team's modification—placing the crystal outside the sphere and connecting it to something that cools it—means the temperature change crucially happens only within the crystal and not within the sphere. The scientists were able to measure the internal quantum efficiency of gallium nitride samples using this technique at temperatures ranging from –261 °C to about 27 °C.

The details of their new set-up were published in *Applied Physics Express* (doi.org/fcs6).

Fluorescence spectroscopy has an excellent palate

University of Adelaide wine researchers are developing a fast and simple method of authenticating wine. The team was able to identify the geographical origins of wines originating from three wine regions of Australia and from Bordeaux in France with 100 % accuracy with a novel technique of molecular fingerprinting using fluorescence spectroscopy.

"Wine authentication can help to avoid any uncertainty around wine labelling according to origin, variety or vintage. The application of a relatively simple technique like this could be adapted for use in the supply chain as a robust method for authentication or detection of adulterated wines" , says Ruchira Ranaweera, PhD student in the University's Waite Research Institute, who conducted the research.

The researchers looked at Cabernet Sauvignon—a globally important grape variety and the second most planted in Australia—from three different wine regions of Australia and Bordeaux in France, the birthplace of Cabernet Sauvignon. They compared an existing approach for authentication using inductively coupled plasma-mass spectrometry (ICP-MS), with the more simple, rapid and cost-effective fluorescence spectroscopy technique.

In every wine they tested using the novel combination of fluorescence spectroscopy with machine learning-driven data analysis, they were able to correctly allocate 100 % of the wine to the correct region with the fluorescence data; ICP-MS classified 97.7 %.

PhD student Ruchira Ranaweera loads a wine sample into a spectrofluorometer, with Associate Professor David Jeffery. Courtesy of University of Adelaide.

There are other useful applications of this technology for the wine industry that are available now or in the pipeline, such as phenolic and wine colour analysis, and smoke taint detection. Project leader Associate Professor David Jeffery, from the Waite Research Institute and the ARC Training Centre for Innovative Wine Production, says they hope ultimately to identify specific chemical markers that help discriminate between wine regions.

"Other than coming up with a robust method for authenticity testing, we are hoping to use the chemical information obtained from fluorescence data to identify the molecules that are differentiating the wines from the different regions", Associate Professor Jeffery says.

The research has been published in *Food Chemistry* (doi.org/fg5z) and was supported by Wine Australia and the Australian Government, the Waite Research Institute and industry partners through the ARC Training Centre for Innovative Wine Production.

Osteoarthritis biomarker found with MS imaging

Using mass spectrometry imaging (MSI) to identify signs of osteoarthritis (OA), University of South Australia scientists are learning more about changes at the molecular level which indicate the severity of cartilage damage. A study led by PhD student Olivia Lee and her supervisor Associate Professor Paul Anderson has mapped complex sugars on OA cartilage, showing different sugars are associated with damaged tissue compared to healthy tissue. The finding will potentially help overcome one of the main challenges of osteoarthritis research—identifying why cartilage degrades at different rates in the body.

"Despite its prevalence in the community, there is a lot about osteoarthritis that we don't understand", Professor Anderson says. "It is one of the most common degenerative joint diseases, yet there are limited diagnostic tools, few treatment options and no cure."

Existing OA biomarkers are still largely focused on bodily fluids, which are neither reliable nor sensitive enough to map all the changes in cartilage damage. By understanding the biomolecular structure at the tissue level and how the joint tissues interact in the early stages of osteoarthritis, the researchers say any molecular changes could be targeted to help slow the progression of the disease with appropriate medication or treatment.

In a recent paper published in the *International Journal of Molecular Sciences* (doi.org/ghbvvg), Lee and her colleagues from the University of South Australia's Musculoskeletal Biology Research Laboratory and the Future Industries Institute explore how advances in MSI to detect OA are promising.

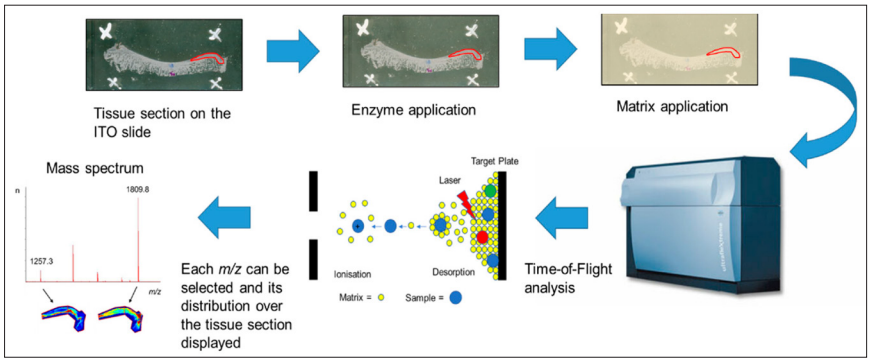


Image reproduced from https://doi.org/ghbvvg under a CC BY licence.

Smaller imaging spectrometer

Researchers led by Ronald Lockwood from MIT Lincoln Laboratory have developed a new Chrisp compact VNIR/SWIR imaging spectrometer (CCVIS). It has a volume about >10× smaller than most of today's devices. One version of the CCVIS is 8.3 cm in diameter and 7 cm long, about the size of a drinks can. The spectrometer has a wavelength range of 400–2500 nm.

"Our compact instrument facilitates the application of imaging spectroscopy for a variety of scientific and commercial problems, such as deployment on small satellites for planetary exploration or using unmanned aerial systems for agricultural purposes", said Lockwood. "We believe that our new spectrometer could also be used to study climate change, one of the most exciting applications of an imaging spectrometer."

Most of today's imaging spectrometers use an Offner–Chrisp optical configuration because it offers excellent control of optical aberrations. However, this design requires a relatively large optical setup. The new CCVIS developed by the researchers performs much like the Offner–Chrisp configuration, but with new optical components that create a more compact design.

To make the new CCVIS, the researchers used a catadioptric lens that combines reflective and refractive elements into one component. This created a more compact instrument while still controlling optical aberrations. The researchers also used a special flat reflection grating that is immersed in a refractive medium rather than air. This grating takes up less space than a traditional grating but with the same resolution.

To test their new design, the researchers demonstrated the spectrometer using a laboratory setup. Their experiments verified that the CCVIS had the expected performance over the full field of view.

"The CCVIS's compact size means that it can be made into modules that could be stacked to increase the field of view", said Lockwood. "It also makes it relatively easy to keep stable with no temperature changes so that optical alignment, and thus spectral performance, remains unchanged."

As a step toward the ultimate goal of a space-based demonstration, the researchers are seeking funding to develop a full prototype that could be thoroughly tested from an airborne vehicle.

The new instrument is reported in *Applied Optics* (doi.org/fhbg).

ARTICLE

Advances in the application of Raman spectroscopy in the nuclear field

Jean-Yves Colle,[a] Dario Manara,[b] Thorsten Geisler[c] and Rudy J.M. Konings[a]
[a]European Commission, Joint Research Center, PO Box 2340, D-76125 Karlsruhe, Germany.
[b]European Commission Joint Research Centre, Via Enrico Fermi, 2749, I-21027 Ispra (VA), Italy
[c]Institute of Geosciences, University of Bonn, Bonn, Germany

Contact
J.Y. Colle: jean-yves.colle@ec.europa.eu; https://orcid.org/0000-0003-4242-8083
D. Manara: https://orcid.org/0000-0002-0767-9859
T. Geisler: https://orcid.org/0000-0003-1923-2023
R.J.M. Konigs: https://orcid.org/0000-0001-6113-2128

Raman spectroscopy is a powerful technique to identify and structurally or chemically characterise chemical compounds in the condensed or gaseous states, including hazardous and highly radioactive materials. It is principally non-destructive, can be performed at distance (up to few tens of metres) and through transparent and semi-transparent shielding screens. It is thus not surprising that it is widely used in industry and research, but also by customs, the police, military, hazmat and in medicine. To meet the different demands, the size of the instruments range from handheld devices, dedicated to fast identification of materials or chemicals, up to high-end and high-resolution scientific instruments.

Raman spectroscopy is based on the indirect measurement of the energy of vibrational transitions of chemical bonds after they have been excited into a virtual vibrational state by monochromatic laser light (photons). The transition energy is thereby revealed through inelastic scattering of the incident photons after their interaction with the interatomic vibrations of the material. The inelastically scattered photons lose (Stokes Raman bands) or eventually gain (anti-Stokes) energy if the bond excited by the photon was originally in a high energy state and does not return to its original ground state. Each vibration transition is thus revealed by a relative shift of the wavelength of the scattered light relative to that of the incident light. Thereby only one photon out of 10^8 photons undergoes Stokes scattering and even fewer photons anti-Stokes scattering. The Raman signal is thus quite weak compared with the intensity of elastically scattered photons. On the other hand, those vibrational transitions are very specific to distinct chemical bonds, resulting in a unique Raman spectrum fingerprint for each material. Additionally, as chemical bond vibrations are sensitive to temperature, mechanical pressure and the molecular environment (for instance, crystal structure, lattice defects, impurities and crystallite size), the Raman spectrum is influenced by those parameters and can thus be used to deduce structural information from it. Measurements carried out with polarised light can even give information about the orientation of the bonds in a crystal.

Raman scattering is best observed with a high-power, monochromatic photon beam. For this reason, the rapid progress of laser technology in the last four decades has broadly improved the quality and the applicability of Raman spectroscopy to the most diverse range of solid, liquid, and gaseous materials and compounds. In modern instruments the measurement is performed by the illumination of the sample with a laser generally through a single objective, such as the one of a microscope and by the analysis of the scattered light with a spectrometer.

In the nuclear field, Raman spectroscopy has been used already for a few decades for the study and identification of actinide compounds.[1] The specific problem in this case is the radiation of the samples, which is hazardous to the operator, and can also deteriorate the instrumentation. When working with nuclear materials, one has to deal with several types of radiation. Actinides, such as uranium or plutonium, emit alpha radiation, which is easily shielded, but needs strict confinement to avoid incorporation during handling. Other actinides such as americium or used nuclear fuel emit gamma radiation that is highly penetrating and needs shielding and/or very small sample sizes. The traditional and obvious way to deal with this is to use a remote optical head connected via glass fibres to the spectrometer.[1] While the head is in the shielded confinement where the radioactive material is, the spectrometer is outside. This configuration, however, reduces the measurement flexibility as remote heads have specific requirements. An alternative is to place a full instrument in a glove box or hot cell, but this is only possible if the radiation

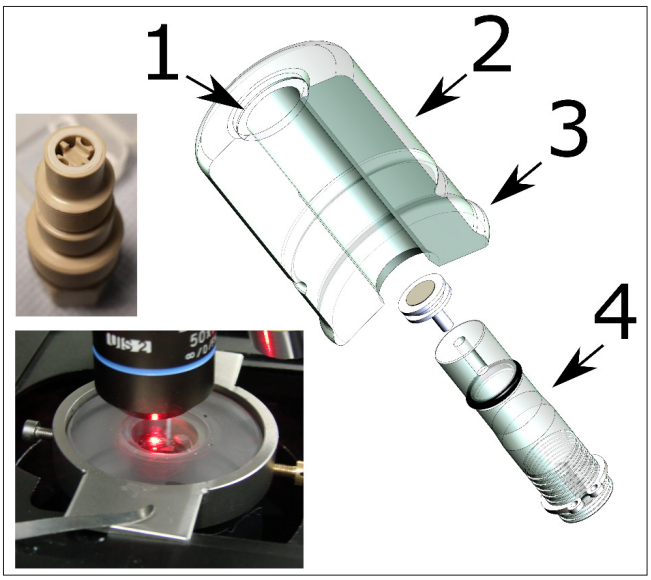


Figure 1. Compact confinement for Raman investigation of nuclear material. 1: optical window, 2: acrylic glass cylinder capsule. 3: flange for the fixation of the plastic bag tunnel. 4: movable sample support. Down left the confinement installed under a microscope. Up left: movable sample support for fluid-cell Raman spectroscopy.

dose is low so that no damage occurs to the instrument.[2]

A recent development is a compact confinement of radioactive material,[3,4] which allows use of a conventional Raman configuration with all its possibilities, resulting in innovative Raman applications on actinide compounds and other nuclear materials. The compact confinement (Figure 1) described in Reference 3 consists of an acrylic glass cylindrical capsule containing the sample just below an optical window. A system of plastic bag tunnels enables the transfer of the radioactive sample into the capsule without breaking the radioactivity confinement. The capsule easily fits onto a microscope stage (Figure 1) and the measurement can be carried out through a standard optical window. Note that this capsule is not reusable but could be designed to be. The only limitation is the need for a long focal objective (1 cm or more). This technique presents several advantages compared to custom nuclearised instruments. The first is the possibility to use the full capacity of the instrument. This includes for example the use of:

- a double subtractive system for the measurement of Stokes and anti-Stokes spectra down to very low wavelength (< 10 cm^{-1});
- a triple additive mode for very high-resolution measurements (spectral resolution down to about 0.3 cm^{-1})
- as many excitation wavelengths as needed, whereas each remote head is configured for only one wavelength;
- the polarisation features of the instrument; the different modes of the instrument (microscope, macro), the autofocus, the confocal microscope function, the mapping/imaging functions).

The second advantage is the easy maintenance of the instrument that will stay free of contamination. The encapsulation technique also provides the possibility to easily implement measurements of samples under vacuum, pressure, chosen atmosphere or in liquids. Finally, the use of a Raman microscope drastically reduces the amount of material needed for the analysis. A sample of about 0.1 mm^3 (~ 1 mg of actinide compounds) is largely sufficient for such a kind of Raman measurement. Handling such low quantities has the advantage that the radiation dose remains low.

The following paragraphs report some relevant and recent examples of scientific and technological applications of Raman spectroscopy in the nuclear field using the benefits of a flexible instrumentation.

Raman spectra of the actinide dioxides

The actinide dioxides are key materials in the nuclear fuel cycle. UO_2 is the most common fuel of nuclear reactors. ThO_2 is a well-known by-product of rare-earth mining, and is considered an alternative fuel material, although it is not fissile but fertile. PuO_2 is produced in nuclear reactors and separated in some countries for re-use in mixed oxide (MOX) fuel.

The actinide dioxides have the general composition AnO_2 and form a cubic fluorite-type crystal lattice. In this structure the most intense Raman signal is the T_{2g} band, which occurs for UO_2 at 445 cm^{-1}. T_{2g} corresponds to the asymmetrical O–U stretching vibration of the

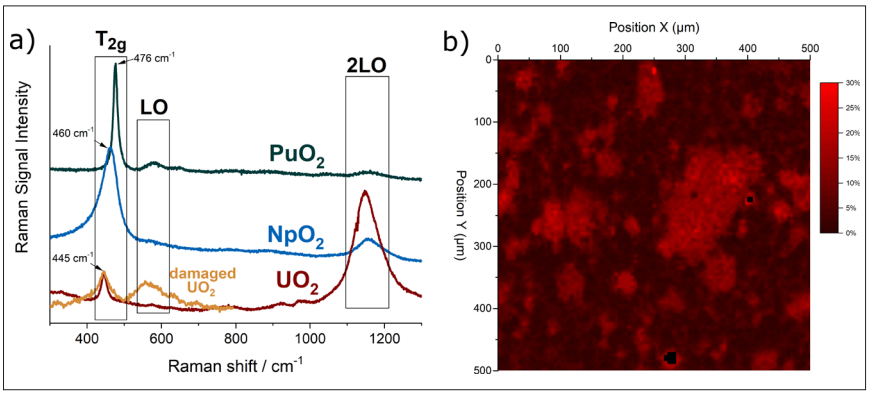


Figure 2. (a) Typical Raman spectra of UO_2, NpO_2 and PuO_2 with the characteristic bands T_{2g}, LO, 2LO. In orange, the spectrum of radiation-damaged UO_2 doped with ^{241}Am. (b) Raman map of the surface of the section of a MOX pellet indicating the concentration of Pu in U + Pu.

face-centred cubic crystal unit. Also, the other actinide dioxides have been measured and Figure 2a shows that the T_{2g} peaks vary regularly within the series, indicating clearly the dependence on the mass of the actinide and thus the energy of the An–O interatomic vibration.

In addition to the interatomic vibrations, characteristic electronic transitions can also be observed by Raman spectroscopy, as shown in recent publications by Naji[5] and Villa-Aleman.[4] In these studies, the Raman spectra of NpO_2 and PuO_2 were measured using multiple excitation wavelengths and laser excitation heating where temperature was measured by the Stokes/anti-Stokes ratio. Naji performed those measurements on a fragment of a nuclear fuel pellet. Villa-Aleman reported an original study on as-fabricated PuO_2, namely with a very different material morphology consisting of long-square stick-shaped crystallites. It was demonstrated that additional bands arise from the coupling of phonon and electronic transitions originating from the crystal field splitting of the degenerate ground states of the $5f^n$ electron configurations.

Matrix defects in crystalline materials, such as the one produced by stoichiometry deviation, impurities or radiation damage, also influence the spectrum. For example, Figure 2a shows in orange the spectrum of UO_2 doped with 5% americium (^{241}Am). The brightening of the T_{2g} is an indication of the presence of defects in the matrix. Alpha-particles produced by the alpha decay of americium have a high energy and deposit it in a small volume of the material leading to atomic displacements and oxygen and uranium Frenkel pairs, with slightly different interatomic distances and forces resulting in the observed brightening.

These detailed analyses give a sound basis for the identification and characterisation of actinide oxides materials in applications like fresh fuel characterisation, irradiation damages in fuel, nuclear forensic and nuclear safeguards.

Distribution of plutonium in MOX nuclear fuel

Worldwide about 5% of the nuclear fuel used in nuclear reactors is MOX fuel. It consists of plutonium-239 (^{239}Pu) as the fissile element instead of uranium-235 (^{235}U) and is mixed with natural or depleted uranium. It targets the re-use of separated plutonium from the recycling of standard UO_2 fuels and can be used for the burning of excess plutonium from dismantled nuclear weapons. It has advantages in terms of proliferation, as the resulting spent fuel has a Pu isotopic composition that cannot be used for fabrication of weapons. It has also the advantage of reducing the consumption of enriched uranium. For industrialisation process optimisation reasons, the standard MOX is made from a blend of about 70 wt% of UO_2 and 30 wt% of a $(U_{0.70}Pu_{0.30})O_2$ powder that is sintered to fuel pellets. This results in a fuel consisting of a UO_2 matrix containing islands of plutonium-rich areas. The fission and thus the heat production will occur particularly in those islands. Therefore, quality control is required to assure that the distribution of the plutonium-rich areas is homogeneous in the fuel.

The PuO_2 distribution in MOX fuel is generally measured by electron probe microanalysis (EPMA), which is an effective technique, although quite expensive and cumbersome. Samples must be carefully prepared for EPMA analysis, and dedicated radioactive hot cells or nuclearised instruments are needed.

Raman detection of the plutonium distribution in MOX fuel would present considerable advantages. Thanks to the possibility of remote measurements through a window, integration of this technique in the production line could be considered without major adaptations.[6] An experimental demonstration of the feasibility of such measurement has been undertaken at JRC Karlsruhe. The analysis is based on the fact that when some of uranium atoms in the UO_2 crystal are replaced by plutonium atoms, the T_{2g} band shifts in a regular way towards higher frequencies[7] up to those of pure PuO_2 near 476 cm^{-1}. This permits unknown PuO_2 concentrations to be obtained from a measured Raman spectrum after correlating concentration and band position in a calibration curve.

A two-dimensional Raman map of the surface of a MOX pellet is shown in Figure 2b. The sample was measured in an alpha-confinement capsule with a standard Raman spectrometer. The false colour scale of the map corresponds to the position of the T_{2g} band at a given position. A T_{2g} band at 445 cm^{-1} corresponds to zero % of Pu in U + Pu (in black), and a band at 455 cm^{-1} corresponds to 30 % of Pu in U + Pu (in red). The map clearly reveals the areas enriched in plutonium.

In principle, this example demonstrates the feasibility of using Raman spectroscopy for the purpose of checking the conformity of the distribution of the plutonium in the fuel. Measurements can be performed with minimal sample preparation and at lower cost than EPMA. In principle, this technology enables increasing the throughput of the measured samples and thus a more comprehensive quality control.

PuO_2 in nuclear waste glasses

Borosilicate glasses are broadly used for the immobilisation of high-level waste from reprocessing of used nuclear fuel. The glass immobilises the waste stream containing unfissioned and undissolved fuel residues, fission products and minor actinides (Np, Am, Cm). It can also be used to immobilise low-quality plutonium. However, the precipitation of crystalline secondary phases from the glass matrix containing fissile isotopes should be avoided, as it may present issues related to the stability of the material in a nuclear repository.

Raman spectroscopy has been demonstrated[8] to be suited for the detection of crystalline plutonium dioxide in sodium borosilicate glasses. Glasses produced at JRC Karlsruhe to simulate the behaviour of high-Pu nuclear waste vitrification matrices were analysed. The precipitation of PuO_2 crystallites from the vitreous glass matrix was observed when the plutonium concentration exceeded a certain value (Figure 3), indicating that the solubility limit of plutonium dioxide in the glass had been exceeded. In this case, Raman spectroscopy helped to efficiently, quickly and non-invasively detect the precipitation of PuO_2 microcrystallites when the solubility limit was reached. Moreover, the results from a

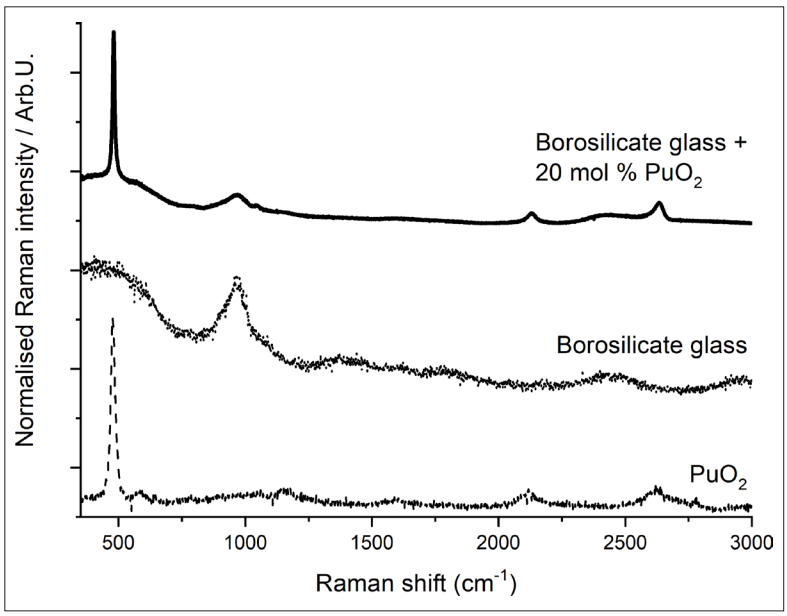


Figure 3. Plot of Raman spectra of borosilicate glass, PuO_2 and borosilicate glass with PuO_2 inclusions (after Reference 8).

recent Raman spectroscopy study of PuO_2 nano-crystals[9] can further be used to detect the early nucleation of crystalline plutonium dioxide nano-crystallites.

Fluid-cell Raman spectroscopy for the *in situ* and real-time study of the aqueous alteration behaviour of Chernobyl "lava"

During the nuclear accident at the Fourth Unit of the Chernobyl Nuclear Power Plant in 1986, the interaction between heated UO_2 fuel rods and fuel cladding with silicate materials of the reactor (concrete and serpentine) led to the formation of a highly radioactive silicate melt, the so-called Chernobyl "lava" that penetrated into different reactor compartments and solidified.[10] In 1990, scientists of the V.G. Khlopin Radium Institute personally collected samples inside the sarcophagus, that was built to cover the nuclear reactor, and observed the formation of secondary uranium phases [e.g., $UO_2 \cdot H_2O$; $UO_3 \cdot 2H_2O$; $Na_4(UO_2)(CO_3)$] on the surface of Chernobyl "lava", providing evidence for the ongoing chemical alteration of Chernobyl "lava".[10]

In order to study the effects of aqueous corrosion on this highly radioactive material by infiltrating water, the capsule for radioactive samples developed at JRC was adapted by replacing the sample holder by a fluid-cell containing a black Chernobyl "lava" sample immerged in an alkaline sodium carbonate solution (Figure 4).

Before the experiment, the black Chernobyl "lava" was investigated by EPMA and Raman spectroscopy. These analyses revealed that the Chernobyl "lava" consists of various inclusions of uranium and zirconium phases (UO_{2+x}, UO_x + Zr, Zr–U–O, $(Zr,U)SiO_4$ and $(Zr,U)O_2$), quartz sand, as well as Fe-bearing steel spheres, which are embedded by in a metaluminous silicate glass.

For the very first *in situ* Raman corrosion experiment,[11] a sample of black "lava" was mounted in the fluid-cell and immersed in a sodium carbonate solution (Na_2CO_3; pH ≈ 11.8). This solution was chosen to simulate the alkaline conditions inside the sarcophagus. The Raman capsule with the integrated fluid-cell was then placed on an automated x,y,z stage, which allowed the acquisition of 2D Raman maps of the area of interest. This area involved an inclusion of uranium oxide and smaller metallic steel spheres, which were surrounded by the glassy matrix.

Raman mapping of the area of interest was repeated approximately every 48 h for a total duration of two months. Within the first 28 days, significant changes in

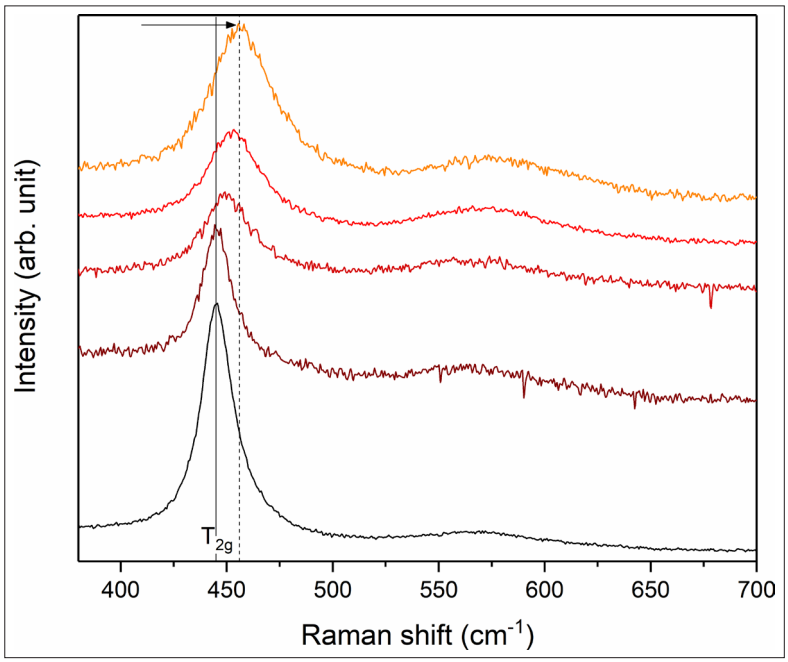


Figure 4. Stacked plot of Raman spectra between 350 cm^{-1} and 700 cm^{-1} from different UO_{2+x} inclusions of the black Chernobyl "lava". Note the frequency shift and simultaneous band broadening of the T_{2g} mode from 445 cm^{-1} (marked by grey solid line) up to 456 cm^{-1} (grey dashed line) indicating the existence of hyper-stoichiometric UO_{2+x}.

the Raman spectra were observed. Although some new bands remain unidentified, the appearance of two intensive bands point to the formation of a yet unidentified secondary phase that precipitated and grew from solution. The results of this work demonstrate the feasibility of extended kinetic analysis of reactions between radioactive materials and aqueous solutions. As recently been shown for non-radioactive glasses,[12] this technique opens up new avenues to study the interaction of nuclear materials and aqueous solutions by Raman spectroscopy with the ability to study specific sub-processes *in situ* and in real time.

Outlook

Thanks to the high flexibility of the Raman spectroscopy technique, the number of studies performed on nuclear materials of many different types and applications has increased impressively in the last fifteen years and moved into applied fields such as nuclear safeguards, decommissioning and process control. Its wider technological application requires a fundamental understanding of the spectra of the materials employed in the nuclear fuel cycle, but also in non-electric applications, such as space power or radioisotope production. In addition to establishing reference spectra for these radioactive materials, one also needs to understand the effects of radiation damage on the spectra and peculiar effects resulting from the unique electronic configuration of the actinides.

References

1. G.M. Begun, R.G. Haire, W.R. Wilmarth and J.R. Peterson, "Raman spectra of some actinide dioxides and of EuF_2", *J. Less-Common Metals* **162,** 129 (1990). https://doi.org/10.1016/0022-5088(90)90465-V
2. G. Guimbretière, L. Desgranges, C. Jegou, A. Canizarès, P. Simon, R. Caraballo, N. Raimboux, M. Barthe, M. Ammar, O.A. Maslova, F. Duval and R. Omnée, "Characterization of nuclear materials in extreme conditions: The Raman spectroscopy approach", *2013 3rd International Conference on Advancements in Nuclear Instrumentation, Measurement Methods and their Applications (ANIMMA)*, pp. 1–8 (2013). https://doi.org/10.1109/ANIMMA.2013.6727957
3. J.-Y. Colle, M. Naji, M. Sierig and D. Manara, "A novel technique for Raman analysis of highly radioactive samples using any standard micro-Raman spectrometer", *JoVE* **122,** e54889 (2017). https://doi.org/10.3791/54889
4. E. Villa-Aleman, N.J. Bridges, T.C. Shehee and A.L. Houk, "Raman microspectroscopy of PuO_2 particulate aggregates", *J. Nucl. Mater.* **515,** 140–149 (2019). https://doi.org/10.1016/j.jnucmat.2018.12.022
5. M. Naji, N. Magnani, L.J. Bonales, S. Mastromarino, J.Y. Colle, J. Cobos and D. Manara, "Raman spectrum of plutonium dioxide: vibrational and crystal field modes", *Phys. Rev. B* **95(10),** 104307 (2017). https://doi.org/10.1103/PhysRevB.95.104307
6. Z. Talip, S. Peuget, M. Magnin, M. Tribet, C. Valot, R. Vauchy and C. Jégou, "Characterization of un-irradiated MIMAS MOX fuel by Raman spectroscopy and EPMA", *J. Nucl. Mater.* **499,** 88–97 (2018). https://doi.org/10.1016/j.jnucmat.2017.11.014
7. R. Böhler, M.J. Welland, D. Prieur, P. Cakir, T. Vitova, T. Pruessmann, I. Pidchenko, C. Hennig, C. Guéneau, R.J.M. Konings and D. Manara, "Recent advances in the study of the UO_2–PuO_2 phase diagram at high temperatures", *J. Nucl. Mater.* **448(1),** 330–339 (2014). https://doi.org/10.1016/j.jnucmat.2014.02.029
8. D. Manara, M. Naji, S. Mastromarino, J.M. Elorrieta, N. Magnani, L. Martel and J.Y. Colle, "The Raman fingerprint of plutonium dioxide: Some example applications for the detection of PuO_2 in host matrices", *J. Nucl. Mater.* **499,** 268–271 (2018). https://doi.org/10.1016/j.jnucmat.2017.11.042
9. D. Hudry, C. Apostolidis, O. Walter, A. Janßen, D. Manara, J.-C. Griveau, E. Colineau, T. Vitova, T. Prüßmann, D. Wang, C. Kübel and D. Meyer, "Ultrasmall plutonium oxide nanocrystals: an innovative material in plutonium science", *Chem.-Eur. J.* **20(33),** 10431–10438 (2014). https://doi.org/10.1002/chem.201402008
10. B.E. Burakov, E.B. Anderson, S.I. Shabalev, E.E. Strykanova, S.V. Ushakov, M. Trotabas, J.Y. Blanc, P. Winter and J. Duco, "The behavior of nuclear fuel in first days of the Chernobyl accident", *MRS Proceedings* **465,** 1297 (2012). https://doi.org/10.1557/PROC-465-1297
11. M.I. Lönartz, *First* in situ *Observations of the Alteration Behaviour of Chernobyl Lava by Fluid-Cell Raman Spectroscopy*. Steinmann-Institut für Geowissenschaften der Rheinischen Friedrich-Wilhelms-Universität Bonn (2019).
12. T. Geisler, L. Dohmen, C. Lenting and M.B.K. Fritzsche, "Real-time *in situ* observations of reaction and transport phenomena during silicate glass corrosion by fluid-cell Raman spectroscopy", *Nat. Materials* **18(4),** 342–348 (2019). https://doi.org/10.1038/s41563-019-0293-8

ARTICLE

Software innovations in four-dimensional mass spectrometric data analysis

Jürgen Cox[a] and Gary Kruppa[b]
[a]Research Group Lead of Computational Systems Biochemistry, Max Planck Institute of Biochemistry, Am Klopferspitz 18, 82152 Martinsried, Germany. cox@biochem.mpg.de; https://orcid.org/0000-0001-8597-205X
[b]Vice-President, Proteomics, Bruker Daltonics

Over the last two decades, significant advances in technology and new methodologies have made proteomics an extremely powerful tool for protein scientists, biologists, and clinical researchers.[1] As analytical instrumentation continues to evolve, more data is produced with each technical advancement in proteomics research. That, of course, also creates new challenges for bioinformatics software development.

The modern high-throughput mass spectrometry (MS)-based proteomics methods that are required to gain deeper insights into biological processes produce enormous amounts of data. This raw data necessitates powerful, automated computer-based methods that provide reliable identification and quantification of proteins.

Quantitative proteomics software

Freely available for academic and non-academic researchers, many laboratories across the globe benefit from the MaxQuant quantitative proteomics software package's precise protein and peptide quantification algorithms. The software was developed by the Computational Systems Biochemistry group at the Max Planck Institute of Biochemistry (MPIB) in Martinsried, Munich, Germany. The group also developed Perseus, a software platform that supports researchers in the interpretation of protein quantification and interaction data as well as data on post-translational modifications (PTMs).

MaxQuant possesses a large ecosystem of algorithms for comprehensive data analysis. It incorporates the peptide search engine Andromeda and, coupled with Perseus, was developed to offer a complete solution for downstream bioinformatics analysis.[2] MaxQuant performs quantification with labels and via the MaxLFQ algorithm on label-free data, and achieves high peptide mass accuracies thanks to its advanced non-linear recalibration algorithms.

MaxQuant for four-dimensional proteomics

MaxQuant is often used for liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS) shotgun proteomics—a method of identifying proteins in complex mixtures to provide a wider dynamic range and coverage of proteins.

Shotgun (or bottom-up) proteomics is the most commonly used MS-based approach to study proteins by digesting them into peptides prior to MS analysis.

Ion mobility can add a further dimension to LC-MS based shotgun proteomics that has the potential to boost proteome coverage, quantification accuracy and dynamic range. However, this additional information requires suitable software that extracts the information contained in the four-dimensional (4D) data space spanned by *m/z*, retention time, ion mobility and signal intensity.

The impact of the addition of the ion mobility dimension on the proteomics field can be seen, for example, in the widespread adoption of Bruker's timsTOF Pro instrument, which was designed to deliver greater sensitivity, selectivity and MS/MS acquisition speeds for proteomics research. The novel design allows for ions to be accumulated in the front section, while ions in the rear section are sequentially released depending on their ion mobility, and in subsequent scans selected precursors can be targeted for MS/MS. This process is called Parallel Accumulation Serial Fragmentation, or PASEF®.[3]

The unique trapped ion mobility spectrometry (TIMS) design allows researchers to reproducibly measure the collisional cross-section (CCS) values for all detected ions, and those can be used to further increase the system's selectivity, enabling more and more reliable relative quantitation information from complex samples and short gradient analyses.

The addition of TIMS to LC-MS based shotgun proteomics, using PASEF, has the potential to boost proteome coverage, quantification accuracy and dynamic range, resulting in fast ultra-sensitive analysis. The increase in speed resulting from PASEF technology allows more samples to be analysed in a shorter time frame, but also generates vast amounts of spectral data, creating challenges when dealing with large sample cohorts.

MPIB software developers adapted the MaxQuant shotgun proteomics workflow to extract this abundance of information from the timsTOF Pro

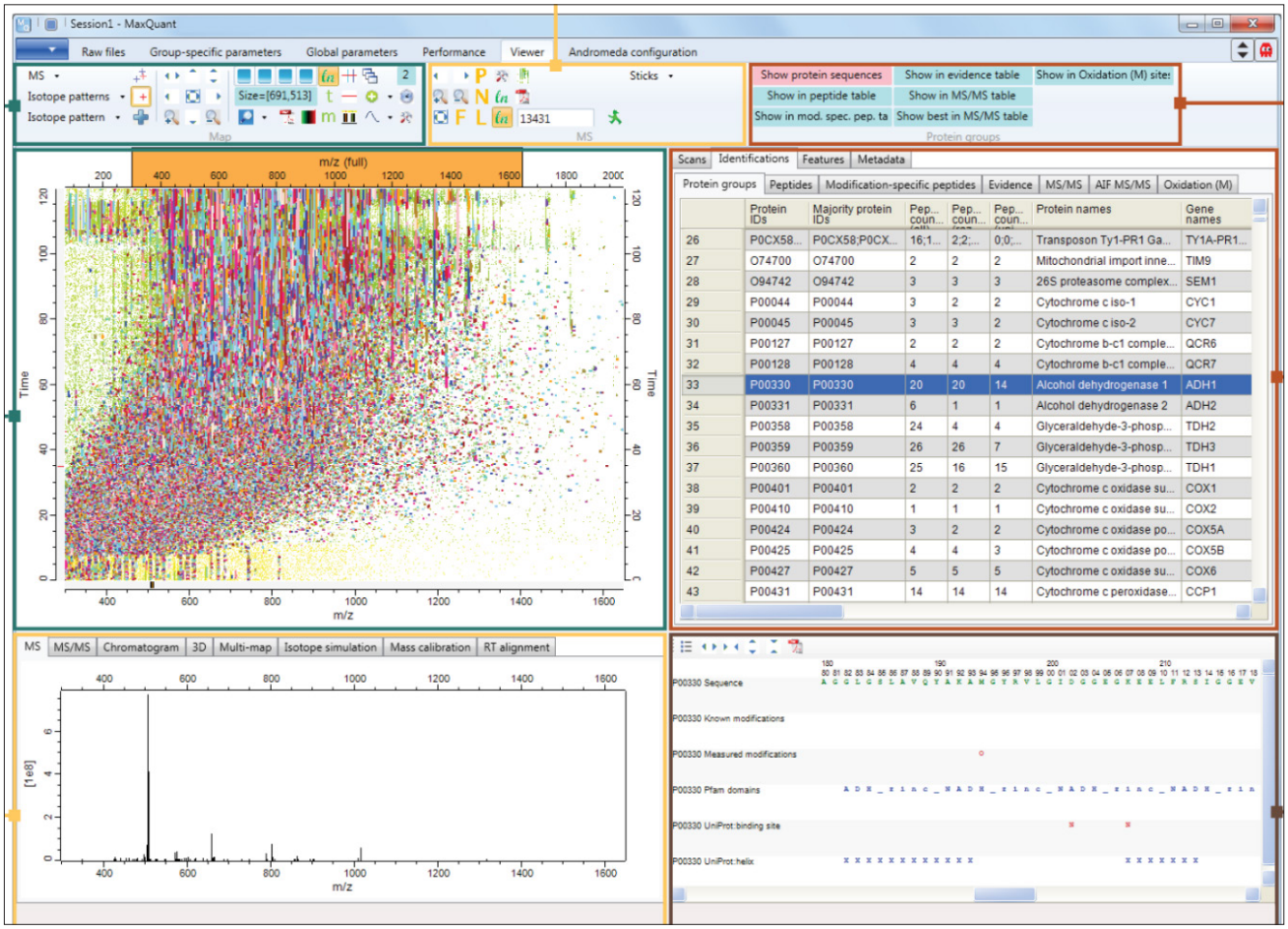


Figure 1. Screenshot of MaxQuant quantitative proteomics software package designed for analysing large mass spectrometric data sets. Source: Maxquant.org

data, making it possible to manage 4D features in the space spanned by retention time, ion mobility, mass and signal intensity that benefit the identification and quantification of peptides, proteins and PTMs.

TIMS is challenging for software developers because it is not just one piece of new information—it adds an additional dimension. The updated MaxQuant 4D-Proteomics workflows can process data produced via PASEF, data-independent acquisition (dia)-PASEF and Mobility Offset Mass Aligned (MOMA).

Adding another dimension can lengthen algorithm processing times, creating a significant challenge for software development with 4D-Proteomics. As a result, the team optimised computation time in MaxQuant to overcome this, so users can achieve good results in a reasonable time frame.

Emerging applications for 4D-Proteomics

Powerful developments in MS technology have led to the expansion of MaxQuant and the software's ability to meet future needs in the proteomics field. These improvements are helping researchers develop new capabilities and applications by delivering more sensitivity and selectivity for the identification and quantification of peptides, proteins and PTMs. Instrumentation advances have also bolstered the ongoing development of MaxQuant. The MPIB software development team's goal is always to expand and improve MaxQuant to meet the complexity of biological processes and novel MS instruments.

Clinical research proteomics

The MPIB Computational Systems Biochemistry team believes clinical research proteomics will be one of the main applications of 4D-Proteomics in the future, and they are working with several clinical groups to bring MS-based proteomics into clinical practice. However, the analysis of proteomics data from samples derived from patients requires special computational strategies. The problems that need to be addressed include: how to extract meaningful protein expression signatures from data with high individual variability, how to integrate the genomic background of the patients into the analysis of proteomics data, and how to determine biomarkers and properly estimate their predictive power.

To answer these questions, the MPIB software developers are working to make use of machine learning algorithms to classify patients and employ feature selection algorithms to extract predictive

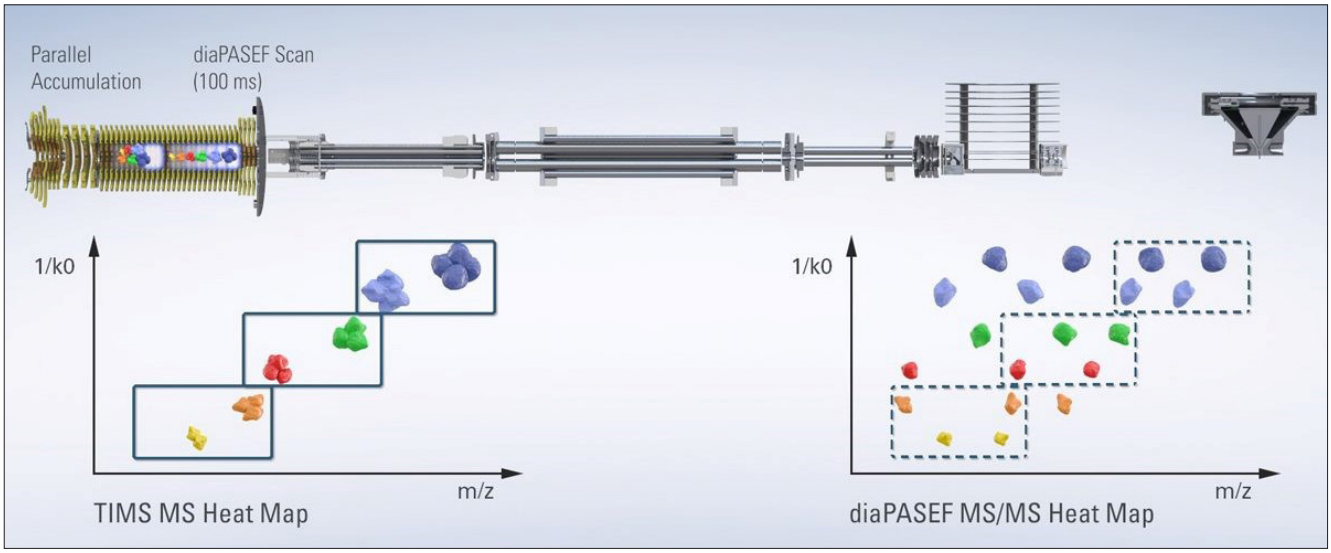


Figure 2. Design of the timsTOF Pro instrument. Source: www.Bruker.com

protein signatures. The extra clinical test engine provides a challenge for the software. It is not clear yet if proteomics will be a guide to which molecules to look at, or if it will be a major component of clinical diagnostics.

Single-cell proteomics

While today's laboratories generate large data sets from single-cell genomic and single-cell transcriptomic research, single cell proteomics (sc-proteomics) is a nascent field. It holds the potential to enable researchers to detect and quantitate the proteins in single cells, avoiding the need to infer proteins from cellular messenger-RNA levels.[4] That also creates new challenges for computational analysis.

MPIB researchers are looking ahead to future needs as sc-proteomics develops, closely examining emerging technologies to establish quantification standards. The software developers believe sc-proteomics will be achievable in the next few years. The two main advances enabling this will be improved sensitivity in the instrumentation and the software to work with it.

Data-independent acquisition

Recent advances in data-independent acquisition (DIA) sensitivity have encouraged the MPIB Computational Systems

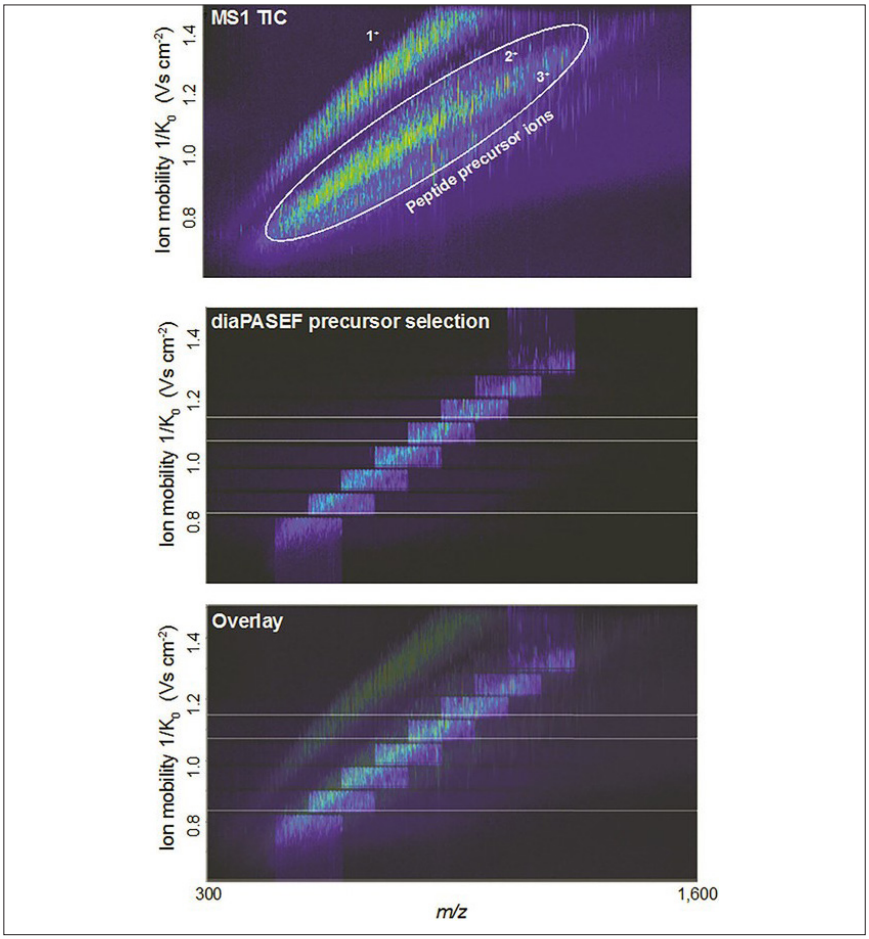


Figure 3. Example showing how dia-PASEF can efficiently fragment nearly all peptide ions eluting at a given retention time. Figures courtesy of: F. Meier, A.-D. Brumer, M. Frank, A. Ha, I. Bludau, E. Voytik, S. Kaspar-Schoenefeld, M. Lubeck, O. Raether, R. Aebersold, B. Collins, H.-L. Rost and M. Mann, "Parallel accumulation – serial fragmentation combined with data-independent acquisition (diaPASEF): Bottom-up proteomics with near optimal usage", *bioRxiv* 656207 (2020). https://doi.org/10.1101/656207

Biochemistry team to integrate DIA workflows into MaxQuant using machine learning algorithms. The success of DIA relies on key instrumental capabilities—namely resolution, sensitivity, accuracy and dynamic range uncompromised by a fast-spectral acquisition rate.

DIA has been implemented in the timsTOF Pro in a way that takes advantage of the speed and sensitivity of TIMS and PASEF, in a method called dia-PASEF®. The 4D nature of the dia-PASEF data is an advantage for DIA software developers, who can make use of the additional ion mobility dimension for alignment and extraction of features. Such recent technological advances, as well as developments in DIA methods, have provided new opportunities for the MPIB Computational Systems Biochemistry research group.

The MaxQuant developers are enthusiastic about the platform's upcoming DIA capability. DDA and DIA are becoming comparable because of improved sensitivity in instrumentation. The hardware is becoming simpler for users, but data has been much more challenging because the software must find which fragments belong to which molecule. Addressing these challenges will provide a deeper coverage of proteomics, which could extend the feasibility of applying 4D-Proteomics for clinical research applications, as mentioned previously.

Conclusion

Bioinformatics software development is a constantly changing field that will see more technological advancement in the future. As analytical instrumentation advances, the MPIB Computational Systems Biochemistry team must deal with even larger amounts of information on the software side because these instruments continue to expand their dynamic range and capabilities.

These improvements to the MaxQuant software platform are possible due to important collaborations between the MPIB Computational Systems Biochemistry research team and other institutions and industry leaders. These collaborations include projects designed to improve proteomics technologies, with MaxQuant providing the computational tools necessary to analyse data acquired on new and emerging hardware platforms. The results of these collaborations will be used to develop future versions of the software to optimise 4D-Proteomics workflows.

References

1. J. Cox and M. Mann, "Quantitative, high-resolution proteomics for data-driven systems biology", *Ann. Rev. Biochem.* **80,** 273–299 (2011). https://doi.org/10.1146/annurev-biochem-061308-093216
2. J. Cox, N. Neuhauser, A. Michalski, R.A. Scheltema, J.V. Olsen and M. Mann, "Andromeda: a peptide search engine integrated into the MaxQuant environment", *J. Proteome Res.* **10(4),** 1794–1805 (2011). https://doi.org/10.1021/pr101065j
3. F. Meier, A.D. Brunner, S. Koch, H. Koch, M. Lubeck, M. Krause, N. Goedecke, J. Decker, T. Kosinski, M.A. Park, N. Bache, O. Hoerning, J. Cox, O. Räther and M. Mann, "Online Parallel Accumulation-Serial Fragmentation (PASEF) with a novel trapped ion mobility mass spectrometer", *Mol. Cell. Proteomics* **17(12),** 2534–2545 (2018). https://doi.org/10.1074/mcp.TIR118.000900
4. V. Marx, "A dream of single-cell proteomics", *Nat. Methods* **16,** 809–812 (2019). https://doi.org/10.1038/s41592-019-0540-6

TONY DAVIES COLUMN

Bill George

Antony N. Davies[a] and Peter McIntyre[b]
[a]SERC, Sustainable Environment Research Centre, Faculty of Computing, Engineering and Science, University of South Wales, UK
[b]Laugharne, Carmarthenshire, Wales, UK

The last 18 months has been a really sad time with the passing of several of the founding fathers of British spectroscopy. Peter McIntyre and I thought it would be good to celebrate briefly the life of a real Welsh character and educator whose style and charisma influenced many to go on and not only stay in science but to rise to leading positions either in industry or academia.

Award winning Fellow—in more ways than one

Professor William (Bill) George BSc (Hons), PhD, DSc, CChem. FRSC, FLSW was educated in South Wales. He attended the same grammar school as Sir Harry Secombe, but I have no indication that this early training was the root of Bill's humour. I don't think Bill's time at Dynevor Grammar School overlapped with Harry Secombe, but Bill went on to Swansea Technical College and University College, Swansea before spending periods in industry at the United Kingdom Chemicals Company, Swansea and moving out of Wales at the Distillers Company Research Laboratory in Epsom.

Bill moved into academia, spending 13 years at what is now Kingston University in London where he became its first Reader and led the first MSc Course.

It is, however, his time spent having returned as an educator and researcher to Wales where most of the anecdotes from his life arise. Bill saw huge change during his career, his laboratories in Pontypridd were located in the original buildings of the revolutionary South Wales and Monmouthshire School of Mines, in Treforest. By 1958 the School had expanded its courses in science, technology and commerce and been renamed Glamorgan College of

Bill George

Technology. As the expansion continued it evolved into Glamorgan Polytechnic and then the Polytechnic of Wales in 1975 when Bill was appointed Head of the Department of Science.

He progressed up the career ladder in the years prior to the Polytechnic of Wales achieving university status. He became first Dean of the faculty of Science in 1984, adding Assistant Director for Research & Consultancy to his roles in 1988. The Polytechnic of Wales became the University of Glamorgan in 1992 and a year later found Bill taking on the increased burden of Pro Vice Chancellor. He was appointed Professor of Molecular Spectroscopy in 1997 and Emeritus Professor in 2000, although this later status change famously did little to hinder his research activities and, as he continued to obtain research grants—often for supercomputing time for *ab initio* and DFT studies—and to publish his results with his colleagues and former students! On April 2012 he was honoured to be elected as a Fellow of the Learned Society of Wales.

- 1959/60: awarded a University of Wales Fellowship
- 1974: awarded a DSc by the University of Wales
- 1975: appointed Head of the Department of Science at the then Polytechnic of Wales
- 1984: also became Dean of Faculty
- 1988: Dean of Faculty and Assistant Director for Research and Consultancy
- 1993: Pro Vice-Chancellor for three years

Figure 1. The original School of Mines Building.

- 1997: Professor of Molecular Spectroscopy
- 2000: Emeritus Professor
- 2008: Awarded Hon. DSc by University of Glamorgan
- April 2012: Elected Fellow of the Learned Society of Wales

His interest in improving teaching in molecular spectroscopy and, later, computational methods brought him to my attention when he and Peter McIntyre published the first interesting chapter on the use of the JCAMP-DX data standard shortly after it had become available across most infrared spectrometer software packages! Really early adopters! His series of textbooks published with different co-authors through John Wiley & Sons became the standard teaching texts in their field.[1–5]

He did not restrict his educational teaching to University level students, but also toured his fascination with spectroscopy around schools and other centres under the title of "What can the rainbow tell us?"

He even published a poem in an edition of this magazine in 2011 sticking to the rainbow theme entitled "The Rainbow Road from Genesis to Nemesis"![5]

Working for the spectroscopy community

Bill worked hard for the community of spectroscopists as a stalwart of the Infrared and Raman Discussion Group and the Association of British Spectroscopists Trust. Long-term ABS Trust treasurer, Terry Threlfall remembers Bill being genial, unperturbable and always full of good humour. Geoff Dent reminded me that Bill put on training courses which became IRDG courses and he was (using his love of computing!) the designer of the first ever IRDG website.

He served on Committees of the Science and Engineering Research Council (SERC), Council for National Academic Awards (CNAA) and the Royal Society of Chemistry (RSC). Locally he chaired the RSC Local Section Committees for both S. East Wales and S. West Wales Sections.

His industry background helped his work as Chair of the Academic Industry Links Organisation and Chair of the Deans of Science Committee, contributing to the widening of the role and membership of both organisations following the expansion in the number of UK universities in 1992. For all his activities promoting spectroscopy, he was awarded the prestigious Norman Sheppard Award in 2017 (Figure 2).

But most of all, Bill will be remembered as a unique character

During a visit to me at the Institute of Spectrochemistry and Applied Spectroscopy I was very keen to show off all the excellent work being carried out by our Molecular Spectroscopy group and the other teams in Dortmund. Bill showed polite interest in all I was so proud to show him… and then spent the next hour explaining the wonders of Microsoft Excel as a super tool to keep track of student grades!. This wasn't exactly what I had expected and, as was Bill's way, brought me right back down to ground. Pride does come before a fall.

There is also a story I haven't really been able to verify around a visit to Norway where Bill was travelling in company and they had to stop at a service station where Bill left the car to stretch his legs. On returning to the car he got in and seemed completely unperturbed that he was now sitting next to a complete stranger. She was somewhat more surprised, especially when he continued the conversation. Bill took it completely in his stride when his travelling companions came to get him and escort him back to the car he had really been travelling in upon arrival!

Figure 2. Bill George receiving the prestigious Norman Sheppard Award from Professor Karen Faulds in 2017.

The strength of the grounding in spectroscopy that Bill gave his students can be seen by the careers that they achieved after completing their PhDs. Prof. Alan Guwy, who heads up the Sustainable Environment Research Centre here in South Wales, can remember his first memorable experience on arriving at university as a fresh-faced undergraduate, knowing nobody, but heading down to the famous Otley public house next to the Treforest Campus where he got into conversation with a local character called Bill… it was only later in the week he discovered he had spent a pleasant evening over a few pints putting the world to rights with the head of the faculty he was just joining!

A shining example is Neil Lewis who completed his PhD with Bill before going on to work in the USA at the National Institute of Health. Neil founded and was president of Spectral Dimensions Inc. before becoming Chief Technical Officer and Technical Director of Malvern Instruments when they acquired Spectral Dimensions. Finally, he became the CTO and Head of R&D at Mettler Toledo Autochem. Neil wrote the excellent nomination for Bill for his Norman Sheppard award (see text box below). Neil became very good friends with Bill and explains in his own words some odd goings on at the famous Pittsburgh conference in New Orleans…

"Many years ago, there was a Pittsburgh Conference (Pittcon) in New Orleans. This was during the days when the attendance was enormous, and hotel space was always at a premium. At the time I was a post-doc at the NIH in the USA and Bill in his usual fashion had not planned ahead and was unable to secure a hotel room.

He called and asked if he could share with me. I informed him that I was already sharing with two other 'twenty-something' post-doc colleagues of mine and, even though he was welcome, it would be very 'cramped'. He accepted and in the evening he was supposed to

TONY DAVIES COLUMN

arrive, I told the hotel clerk that I was expecting him and asked them to provide him with an additional key.

My colleagues and I, being young, thirsty and in New Orleans, went out for an evening on the town! Well it must have been somewhere between 3am and 4am when we returned and burst into the room. Lights came on, Bill gave out several expletives, and sat bolt upright in his pyjamas buttoned to the top and on the edge of a tiny fold-out bed in the corner. It was a sight I will never forget! We were still in a raucous party mood and offered Bill a beer. Bill being the tremendous, good sport that he was he joined us. I remember him complaining that there was not a clean glass to be found for his beer and uttering a string of other sarcastic remarks that always came naturally to him.

After we had all managed to down a couple more beers, and to take a short nap, it was time to get up. I was personally awakened by loud and somewhat angry words coming from one of my colleagues complaining that the bathroom was locked!! As you can imagine, he was very keen to do what most people want to do first thing in the morning after a night consuming uncounted beers. Bill had locked the door and was taking a long hot bath making the next 30 minutes or so extremely tense! Eventually we all managed to get going for the day and I said to Bill that I would see him later. I never did, and I didn't catch another single glimpse of him for the rest of the week... I presume he decided that he would be better off looking for other sleeping arrangements even if it meant just laying down in the hotel lobby."

Bill's memorable exploits continued right up to the final seminar in his honour when, having finished the presentations, he turned down the optics of the overhead projector into the base without turning the very strong light source off. He continued answering questions from the audience completely impervious to the smoke rising from the projector he was doing a good job of setting on fire. Bill was always a great promoter of the power of light! I think we will all miss him.

The IRDG Norman Sheppard Award Committee:

It is with a strong sense of personal pride and honor that I nominate Professor WC "Bill" George for the prestigious IRDG Norman Sheppard Award.

As can be seen from his resume, Bill has had a long and prestigious academic track record, authoring or co-authoring more than 100 peer-reviewed publications focused almost exclusively on infrared and Raman spectroscopy. Later in his career Bill's work began to focus on the demographics, political landscape and the state of funding of scientific research in the UK; a cause that he still feels very strongly about.

Bill has been a long-standing supporter of education, and in particular higher education in Wales. He was one of the early driving forces that made the IRDG what it is today, and his enthusiasm for his work, and his passion for educating the next generation of scientists is exemplified by the excellent work he did organizing, developing and teaching a series of IRDG short-courses over a period of more than 20 years.

Bill was also an active volunteer in local high schools teaching 'basic spectroscopy' using the rainbow and other natural phenomena as a means of describing and explaining the interaction of light with matter. Bill was also quick to recognize the potential of the internet and web-sites for education, historical archiving and promotion. He played an active role promoting these developments at the University of Glamorgan, and in fact developed the first IRDG web-site.

Today the University of South Wales is the home to approximately 30,000 students going through a series of significant growth spurts since its early, and more humble, days as the South Wales and Monmouthshire School of Mines founded in 1913. Much of that growth occurred during Bill's tenure as first the Dean of the Faculty of Science and then Pro Vice Chancellor. Bill's position and commitment to higher education gave him a forceful and influential voice, he always spoke his mind and was active in promoting those values that he believed to be in the best interest of the students, and the university at large. Bill's philosophy to education also extends beyond the formal teaching years at school or university. As one of Bill's PhD students I can attest to the personal and professional support he gave to all his students both during and after their graduation. Indeed, his active support and interest in my professional development lasts to this very day more than 30 years after my graduation. I had the pleasure of meeting up with Bill again a couple of weeks ago and nothing changes, at the age of 84 he just wanted to talk about science and my career. He actively rummaged through his archives and supplied me with a series of reprints for me to take away with me to read!

I believe Bill is without doubt deserving of consideration for this award and I hope you will give my nomination your most serious consideration.

Yours, sincerely,

E. Neil Lewis

References

1. W.O. George and P. McIntyre, *Infrared Spectroscopy (Analytical Chemistry by Open Learning)*. Wiley (1987). ISBN: 978-0471913832
2. W.O. George and H. Willis (Eds), *Computer Methods in UV, Visible and IR Spectroscopy*. Wiley (1990). ISBN: 978-0851863238
3. W.O. George and D. Steele (Eds), *Computing Applications in Molecular Spectroscopy*. Wiley (1995). ISBN: 978-3540787648
4. B.H. Stuart, W.O. George and P. McIntyre, *Modern Infrared Spectroscopy (Analytical Chemistry by Open Learning)*. Wiley (1995). ISBN: 978-0471959175
5. W.O. George, "Poem: The Rainbow Road from Genesis to Nemesis", *Spectrosc. Europe* **23(3),** 27 (2011). https://www.spectroscopyeurope.com/article/poem-rainbow-road-genesis-nemesis

SAMPLING COLUMN

Quality and sampling error quantification for gold mineral resource estimation


Simon Dominy


Saranchimeg Purevgerel


Kim Esbensen

Simon C. Dominy,[a] Saranchimeg Purevgerel[b] and Kim H. Esbensen[c]
[a]Camborne School of Mines, Cornwall, UK and Novo Resources Corporation, Perth, Western Australia. s.dominy@e3geomet.com
[b]MSA Global LLC, Ulaanbaatar, Mongolia. p.saranchimeg@msaglobal.net
[c]KHE Consulting, Copenhagen, Denmark. khe.consult@gmail.com

Sampling is a vital component during all stages of the mine value chain. It includes the sampling of *in situ* material and broken rock for geological, metallurgical and geoenvironmental purposes. Sampling errors are defined in the context of the Theory of Sampling (TOS), where incorrect actions may lead to uncertainty and create a significant overall sampling + measurement error.[1–3] The TOS breaks down this error into a series of contributions along the full value chain (the planning to assay-measurement process). Errors are additive throughout this pathway, unavoidably exacerbating risk.[2,4–6] After collection, sampling errors also occur throughout all subsequent downstream processes contributing to uncertainty in test work and any decisions made thereon. Across the full mine value chain, the sum of these errors generate both financial and intangible losses. In essence, poor-quality, non-representative sampling increases project risk and may consequently often lead to incorrect project valuation. There is hardly any other application field where this is as critically important than for Gold mineral resource estimation, because of the very low grades and the extremely irregular mineralisation heterogeneities encountered (Figure 1).

Sampling—the first critical success factor in the mine value chain

The data produced must be fit-for-purpose to contribute to mineral resources/ore reserves reported in accordance with the 2017 PERC[7] or other international codes. Quality assurance/quality control (QA/QC) is critical to maintaining data integrity through documented procedures, sample security, and monitoring of precision, accuracy and contamination. Samples and their associated assays are key inputs into important decisions throughout the mine value chain.

The TOS was first developed in the 1950s by Dr Pierre Gy to deal with sampling challenges in the mining industry, though it has far wider applications

Figure 1. For optimal sampling error quantification for Gold mineral resource estimation no efforts are spared: Reverse Circulation grade control drilling at the Novo Resources Corporation Beatons Creek project in Western Australia.

today.[1–3] The TOS provides critical guidelines for reducing sampling errors, Table 1.

Quality assurance and quality control

Quality assurance and quality control are the key components of a quality management system.[8–10] Quality assurance is the collation of all actions *necessary* to provide adequate *confidence* that a process (e.g. sampling, test work and assaying) will satisfy the pertinent quality requirements. While QA deals with prevention of problems, QC aims to detect these—in time. Quality control procedures monitor both precision and accuracy of samples and data, as well as possible sample contamination during preparation and assaying. Throughout any mineral resource sampling programme, QA/QC is a key activity to determine the imperative of fit-for-purpose samples.

Protocols should be set up to cover: field collection, laboratory preparation and analysis. During grade control, QA/QC should include field duplicates and certified reference material (CRM) submission, e.g. a minimum of three CRMs at a range of grades, including blanks. Laboratory QA/QC shall include internal CRMs, pulp duplicates, umpire sample submission, pulp screen tests and contamination tests. In particular, duplicate field samples provide a measure of variability of the entire sampling and analysis process. Best practice QA/QC is a very comprehensive framework, Table 2.

Documentation of sample collection and laboratory activities is an important part of QA/QC, as is appropriate staff training and monitoring. It is the opinion of the present authors that quality samples only follow from well-trained and experienced personnel. Companies should ensure that all staff involved in sampling activities are appropriately trained in sampling and, during their first few months, have adequate mentoring (sampling QA). This will be additional to other standard operational and safety training. Proper training shall be facilitated by well-written and illustrated documentation, see examples in Reference 3.

Table 1. Definition of TOS sampling errors.

Sampling error	Acronym	Error type	Effect on sampling	Source of error	Error definition
Fundamental	FSE	Correct Sampling Error (CSE)	Random errors—Precision generator	Characteristics of the ore type. Relates to constitution and distribution heterogeneity	Results from grade heterogeneity of the broken lot. Of all sampling errors, the FSE does not cancel out and remains even if a sampling operation is "correct". Experience shows that the total nugget effect in variographic modelling can also be artificially high because sample masses are not optimal.
Grouping and Segregation	GSE				Relates to the error due to the combination of grouping and segregation of rock fragments in the lot. Once rock is broken, there will be segregation of particles at all scales, e.g. surface stockpile or laboratory pulp.
Delimitation	IDE	Incorrect Sampling Error (ISE)	Uncontrollable inconstant errors—Bias generators	Sampling equipment and materials handling	Results from an incorrect shape of the volume delineation of an increment or a sample.
Extraction	IEE				Results from the incorrect extraction of a sample. Extraction is only "correct" when all fragments within the delineated volume are fully extracted.
Weighting	IWE				Samples should represent a consistent mass per unit (e.g. $kg\,m^{-1}$).
Preparation	IPE				Refers to issues during sample transport and storage, e.g. mix-up, damage, loss and alteration, preparation (contamination and/or losses), and intentional actions (sabotage and salting), as well as unintentional actions (carelessness and non-adherence to protocols).
Analytical	TAE	—		Analytical process	Relates to all errors during assay and analytical processes, including issues related to rock matrix effects and debilitating analytical equipment maintenance, faulty calibration etc.

Table 2. Best practice QA/QC for a Gold grade control sampling programme for sound resource estimation.

QA/QC action	Rate[k]	Instigator	Key Performance Indicators (KPI) Fine Gold/[Coarse Gold]	
Field duplicates[a]	1 in 20	Operator	90%±10–25% HARD[j] [90%±25–50% HARD]	90%±13–35% RSV [90%±35–70% RSV]
Sample quality index[b]	All	Operator	Depends upon sample type; for saw-cut channels >80%; for diamond drill core >85% recovery	
Coarse reject duplicates[c]	1 in 20	Laboratory	90%±10–20% HARD [90%±20–50% HARD]	90%±13–28% RSV [90%±26–70% RSV]
Pulp duplicates[d]	1 in 20	Laboratory	90%±10% HARD [90%±10–20% HARD]	90%±13% RSV [90%±13–28% RSV]
Certified Reference Materials[e]	1 in 20	Operator and laboratory	<2δ ("safe zone") no action required 2δ–3δ ("warning") investigate (re-assay 25% of batch if required) >3δ ("action") re-assay 100% of batch	
Blanks[f]	1 in 20	Operator and laboratory	Less than 0.05 g/t Au	
Pulp quality[g]	1 in 20	Laboratory	95% to be P95 –75 µm	
Barren flush[h]	1 in 20–50	Laboratory	≪0.5% gold loss	
Umpire assays[i]	1 in 20	Operator	90%±10% HARD	90%±13% RSV
Laboratory audit	Quarterly	Operator	Full adherence to agreed practices and performance levels	
QAQC review	Monthly	Operator and laboratory	Compliance across all metrics	

[a]Applies to any sample type collected.
[b]Applied to linear and drill samples; KPIs are based on sample type and expected mass.
[c]Laboratory crusher or reverse circulation (RC) rig rejects.
[d]Dependent upon nature of ore and assay method. For samples assayed via screen fire assay (SFA), a high precision would be expected for undersize fraction.
[e]Recommendation to have a minimum of three CRMs at grades ranging from cut-off, ROM and high-grade. For any batch of (say) 20–30 samples, three key CRMs should be added. Note that by their very need to be homogeneous, CRMs do not bear coarse "nuggety" gold, but they can be matrix matched by being quartz-dominated, sulphide-bearing a.o. The laboratory will also insert its own CRMs. CRMs used for SFA process will just be fire assayed. Action is required if 3δ breached, usually re-assay of the entire batch if possible.
[f]Blanks provide a measure of contamination. They should be inserted after expected high-grade and/or visible gold-bearing samples. If substantial visible gold is present, two separate blanks should be placed after the sample. One blank should be added together with the three CRMs per batch. Laboratory will also place blanks into the sample stream.
[g]Test involves screening or use of an autosizer of the pulp to ensure 95% passing. All samples should pass or the entire batch should be reground.
[h]Barren flush may be inserted after each and every sample for coarse gold samples. Assaying of the barren flush; for fine gold ores, a rate of 1 in 50 is appropriate increasing to 1 in 20 for coarse gold ores. Careful management of coarse gold ores is required. It is suggested that laboratories include a "wash" after visibly high-grade (e.g. visible gold-bearing) samples. However, if the ore bears notable coarse gold, then cleaning is best after each sample given that even low-grade samples can bear coarse gold particles.
[i]Monthly submission of samples (typically pulps), including standards and duplicates is sufficient to provide a check of primary laboratory results. This is especially important where an on-site laboratory is being used as it provides independent confirmation of the results. Where SFA, LW or PAL is used, there may be no pulp residues to submit. In this case, coarse rejects can be used. Umpire samples (e.g. pulps or coarse duplicates) should be supplied to the mine and submitted by mine staff to the umpire laboratory. In some cases, the laboratory (mine or off-site) may submit umpire samples as part of their internal QA/QC.
[j]HARD is half the absolute difference of the pair divided by the pair mean; HARD value for fine versus coarse gold; HARD can be expressed as RSV, where HARD = $\sqrt{2}/2 \cdot$ RSV, e.g. ±10% HARD is ±13% RSV.
[k]It is important to ensure that enough QA data is collected, particularly during a small sampling programme. The rate of insertion of CRMs, blanks etc. may need to be increased beyond the nominal 1 in 20 to achieve a minimum of 10 results.

Training and mentoring should be linked to continuous quality improvement programmes, where protocols are internally and externally audited at least annually. On-going supervision and periodic re-training are strongly recommended, and should always in part be based on practice at the rock face/in the core shed, not only in the classroom.

Quantifying errors along the full sampling value chain

The results of *duplicate sampling* programmes document the magnitude of errors across the *full* sampling value chain, Table 3. These generally show that a large component of the total error is introduced during sample collection, especially during primary sampling. As a result, undertaking excessive efforts to reduce errors during preparation and analysis will not necessarily result in a substantive uncertainty reduction. In contrast, the collection of larger, high-quality field samples (for examples using a higher number of increments in composite samples) will result in significant error reduction provided that other protocols are optimised appropriately.

Test work from a Gold vein deposit exemplifies the impact of sampling error through comparison of chip vs channel samples, Table 4.[13]

Seventy-five sample *triplicates* (chip, hand-cut channel and saw-cut channel) were collected from around a 40 m × 20 m stope block (Figure 2). The mineralisation was known to have a moderate variability, containing visible gold up to 1.5 mm in size. The test block was sampled from faces located every 1.5 m along its upper and lower drives and two raises. After cleaning, a reference line was drawn across each face centre and the different types of samples were collected systematically from the bottom up: chip sample, hand-cut and saw-cut channels. The sample delimitation dimensions were estimated and designed to achieve a theoretical sample support of 3 kg m^{-1}. All samples were subsequently prepared and assayed in identical fashion, via a total sample preparation and screen fire assay route. The FSE for this highly optimised protocol, was effectively zero. A QA/QC programme was applied, with all CRMs and blanks within expectation.

These results show a marked reduction in RSV and nugget effect between the three sample sets. The rigorous laboratory protocol and QA/QC indicate that errors within the laboratory were at a minimum. Therefore, the remaining variability relates to the *in situ* nugget effect and sample collection. The dominant error for the channel samples relates to the *in situ* nugget effect, given that sampling error was minimal. The dominant difference between the chip and saw-cut channel samples relates to sampling error. These results corroborate many previous findings, showing that saw-cut channel samples provide the best sample quality. Most importantly, this experiment substantiates the critical role of *empirical* total sampling/preparation/analysis error quantification.

Stage-wise error evaluation

More detailed error evaluations can be undertaken including each key stage along the sampling value chain. Thus Table 5 shows the results of such an analysis for two contrasting Gold ore types (termed mesothermal and epithermal). In both cases the highest stage error again turned out to be the field RSV, at 42% and 34% respectively.

For the epithermal system (no coarse gold), all stage errors were found to be

Table 3. Distribution of errors across stages of a gold sampling programme.

Error	Stage	Sample type/activity	TOS errors	Duplicate component error range[a]	FSE[b]	Other TOS errors[c]
Sampling	Collect and transport	*In situ* sampling (e.g. core and linear samples)	INE, IDE, IEE, IWE, IPE	±20–70%	INE	±23%
		Broken rock sampling (e.g. core and RC samples etc.)	FSE, GSE, IDE, IEE, IWE, IPE		±16%	
Preparation	Preparation	Drying	IPE	±5–20%	±11%	
		Crushing/grinding	IPE			
		Splitting	FSE, GSE, IDE, IEE, IPE			
Analytical	Assay	Splitting	FSE, GSE, IDE, IEE, IPE	±1–15%	±8%	
		Analysis	TAE			
Total error				±20–70%	±21%	±23%
Target error (fine-gold)/coarse-gold				(±20%) ±40%	±32%	

[a]Potential component error range as determined from duplicate sample (pair) analysis;[11] [b]Maximum recommended FSE distribution across the sampling stages;[12] [c]Maximum recommended *other* TOS error proportions across the sampling stages;[12] RC: Reverse Circulation.

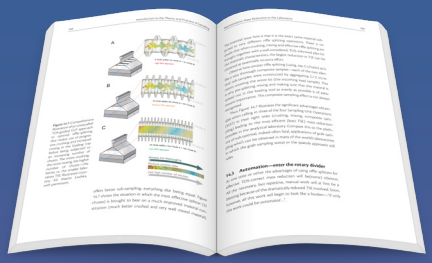
Introduction to the Theory and Practice of Sampling

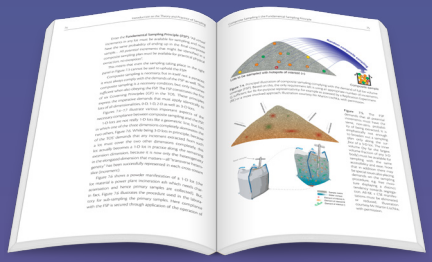
Kim H. Esbensen

with contributions from Claas Wagner, Pentti Minkkinen, Claudia Paoletti, Karin Engström, Martin Lischka and Jørgen Riis Pedersen

“Sampling is not gambling”. Analytical results forming the basis for decision making in science, technology, industry and society must be relevant, valid and reliable. However, analytical results cannot be detached from the specific conditions under which they originated. Sampling comes to the fore as a critical success factor before analysis, which should only be made on documented representative samples. There is a complex and challenging pathway from heterogeneous materials in “lots” such as satchels, bags, drums, vessels, truck loads, railroad cars, shiploads, stockpiles (in the kg–ton range) to the miniscule laboratory aliquot (in the g–µg range), which is what is actually analysed.

This book presents the Theory and Practice of Sampling (TOS) starting from level zero in a novel didactic framework without excessive mathematics and statistics. The book covers sampling from stationary lots, from moving, dynamic lots (process sampling) and has a vital focus on sampling in the analytical laboratory.





“I recommend this book to all newcomers to TOS”

“This book may well end up being the standard introduction sourcebook for representative sampling.”

“One of the book’s major advantages is the lavish use of carefully designed didactic diagrams”

impopen.com/sampling

IMPublicationsOpen

Table 4. Empirical example: comparison between chip and channel sample replicates. Mean grades cut and diluted to stope width. Reconciled stope head grade 13.7 g/t Au.

Type	Mean support (kg m^{-1}) [range]	Target support (%) [within ±5%]	Mean grade (g/t Au)	Percent of mined grade (%)	RSV (%)	Nugget effect (%)	Sample collection errors[a]	Preparation and assay errors[b]
Chip-channel	2.1 [1.4–3.4]	22	21.3	+55	198	68	High: IDE, IEE and IWE	Low: entire sample crushed and pulverised prior to total sample screen fire assay with triplicate fine-fraction fire assay. All equipment cleaned between samples.
Channel (hand cut)	2.6 [2.1–3.3]	63	16.7	+22	135	54	Low-moderate: IDE, IEE and IWE	
Channel (saw-cut)	2.8 [2.3–3.1]	77	16.3	+19	81	41	Low: IDE, IEE and IWE	

[a]Indicative sample collection error definition: Red: high (>±50%); Orange: moderate (±20–50%); Green: low (<±20%). [b]Indicative preparation and assay error: Red: high (>±20%); Orange: moderate (±10–20%); Green: low (<±10%).

reasonable and did not require further action (Figure 3).

For the mesothermal system (coarse gold, i.e. "nugget" gold) both the field and analytical RSVs were deemed *high*. In order to improve on this situation, the field RSV was attempted to be reduced by taking a *larger* split at the rig (up from 2 kg to 4 kg) and assaying the entire 4 kg by a more precise analytical method (LeachWELL). Based on initial duplicates from the revised protocol, the field RSV was now reduced to 36% and the analytical RSV 4%, now acceptable for a coarse gold mineralisation.

There is a need, and a clear advantage, in moving towards full quantification of errors for objective QC assessment, where a first step is the application of the RSV sampling + analysis variability characteristic as defined in DS3077.[3,14] Resolution of individual relative errors across the complete sampling, preparation and analysis stages can be gained from simple duplicate sample pairs, as evidenced by Table 5.

Gold—always special

For Gold resource estimation, special issues are about, compared to many other materials and commodities. Thus deliberately strenuous practical measures are recommended to reduce the risk of *tampering* of samples. These could include: maintaining increased security between the sample site (e.g. mine face and drill rig) and sample transport and

Figure 2. Underground sampling. Collection of optimal saw-cut channel sample. Left: cutting channel "delimitation" slots; right: sampling ("extraction") of the channel material.

Figure 3. Left: logging and marking diamond drilling cores for sampling (at former Castlemaine Goldfields Ltd Wattle Gully project in Victoria, Australia). Right: sample preparation: diamond drilling (DD) core ready for cutting.

Table 5. Stage-wise error estimation for two contrasting Gold ore types (RC = Reverse Circulation drilling; DD = Diamond Drilling).

RC mesothermal deposit with minor coarse Gold			
	Field RSV[a]	Crush RSV	Analytical RSV
Duplicate RSV	47%	20%	13%
Stage RSV	42%	15%	13%
Relative proportion	83%	10%	7%
DD epithermal deposit with no coarse Gold			
	Field RSV[b]	Crush RSV	Analytical RSV
Duplicate RSV	35%	7%	2%
Stage RSV	34%	7%	2%
Relative proportion	95%	4%	1%

All figures rounded to the nearest whole %. [a]Rig duplicate; [b]Core half duplicate.

careful recording of who has access to samples between collection and shipping, and maintaining a copy of that record.

Conclusions

Geologists and analytical chemists must acknowledge the systematic *rigour* of the TOS framework and should readily be able to appreciate the help from proper management of all associated errors.

Empirical error estimations of all stages involved in the complete "from-lot-to-aliquot" pathway demonstrated above and the value of the critical information gained has been laid out in no uncertain way. Where samples are analysed to support any resource estimate, a QA/QC programme must be introduced to ensure continuous quality information of both sampling and assaying. Written protocols and procedures, staff training, periodic auditing of protocols and people, and re-training are all required. DS3077[14] provides a framework on how to produce transparent protocols regarding the specific sampling pathway. There are many QA/QC frameworks that can be applied—more on this latter issue in later Sampling Columns.

References

1. P.M. Gy, *Sampling of Particulate Materials: Theory and Practice*. Elsevier (1982).
2. F.F. Pitard, *Theory of Sampling and Sampling Practice*. CRC Press (2019). https://doi.org/10.1201/9781351105934
3. K.H. Esbensen, *Introduction to the Theory and Practice of Sampling*. IM Publications Open (2020). https://doi.org/10.1255/978-1-906715-29-8
4. R.C.A. Minnitt, "Sampling: The impact on costs and decision making", *J. South Afr. Inst. Min. Metall.* **107,** 451–462 (2007).
5. S.C. Dominy, "Importance of good sampling practice throughout the gold mine value chain", *Min. Tech.* **125,** 129–141 (2016). https://doi.org/10.1179/1743286315Y.0000000028
6. J.-M. Rendu, *Risk Management in Evaluating Mineral Deposits*. Society of Mining, Metallurgy and Exploration (2017).
7. PERC, *Pan-European Standard for Reporting of Exploration Results, Mineral Resources and Reserves*. The Pan-European Reserves and Resources Reporting Committee (PERC) (2017).
8. M.A. Vallée, "Sampling quality control", *Explor. Min. Geol.* **7,** 107–116 (1998).
9. M.Z. Abzalov, "Quality control of assay data: a review of procedures for measuring and monitoring precision and accuracy", *Explor. Min. Geol.* **3–4,** 131–144 (2008). https://doi.org/10.2113/gsemg.17.3-4.131
10. A. Simon and G. Gosson, "Considerations on quality assurance/quality control and sample security", in *Proceedings of the Sampling Conference, Perth, Australia, 27–29 May 2008*. Australasian Institute of Mining and Metallurgy, pp. 135–140 (2008).
11. C.R. Stanley and B.W. Smee, "Strategies for reducing sampling errors in exploration and resource definition drilling programmes for gold deposits", *Geochem. Explor. Environ. Anal.* **7,** 329–340 (2007). https://doi.org/10.1144/1467-7873/07-128
12. F.F. Pitard, "Guidelines for acceptable allotted sampling uncertainty", in *Proceedings of the World Conference on Sampling and Blending*. Gecamin, Santiago, pp. 89–98 (2013).
13. S.C. Dominy, H.J. Glass, L. O'Connor, C.K. Lam, S. Purevgerel and R.C.A. Minnitt, "Integrating the Theory of Sampling into underground mine grade control strategies", *Minerals* **8(6),** 232 (2018). https://doi.org/10.3390/min8060232
14. *DS3077. Representative Sampling – Horizontal Standard*. Danish Standards Foundation (2013). www.ds.dk

NEW PRODUCTS

ATOMIC

Software for mobile OES analysers

Hitachi High-Tech Analytical Science has introduced SpArcfire to its mobile optical emission spectrometry (OES) analyser range; this operating software is already available across Hitachi's stationary spark OES range. The new interface has been optimised for touchscreens, and designed to maximise speed and efficiency for both simple and complex metals analysis tasks, removing the need for extensive training or highly experienced operators. SpArcfire software can help Hitachi OES instruments to complete all metals analysis tasks, including measuring unknown materials, identifying and verifying grades, creating customisable report templates, and performing and evaluating accuracy tests with control samples. Advanced users can edit and modify regression data to extend calibration ranges. SpArcfire software can also verify the status of the instrument, providing real-time monitoring and diagnostics on all systems parameters such as temperature, pressure and voltage inputs and outputs. This ensures essential analysis equipment can be kept operating at peak performance.

Hitachi High-Tech Analytical Science

http://link.spectroscopyeurope.com/32-094

IMAGING

Multisensor plant phenotyping system

The new HyperAixpert multisensor plant phenotyping system from Analytik is configurable with a wide range of sensors, and uses machine-learning-based analytical software to ensure that data acquisition is both standardised and repeatable. Suitable for a range of sample types, a variety of high-resolution sensors enable rapid digital analysis of phenotyping traits for experiments in genetics, plant research and breeding, plant cultivation products as well as detecting plant stress and diseases.

The HyperAixpert may be configured to operate hyperspectral cameras in scanning mode and, therefore, is suitable for physiological phenotyping. Options for PAM chlorophyll fluorescence imaging and NIR imaging also enable the HyperAixpert to deliver physiologically relevant data. Dimensions and morphology of plants under study can be determined using RGB-imaging and laser scanning sensors. Comprehensive data sets originating from the broad range of cameras/sensors deliver phenotypic data on both morphological and physiological levels.

Broad ranges of typical laboratory samples can be measured, including seedlings up to 20 cm height, samples in MTPs or petri dishes, or detached plant parts. Samples on trays can be loaded into the measuring cabinet by the user, or via an optional, automated TrayProvider unit. Using an optional recessed sample stage, plants up to 40 cm can be measured in manual loading mode. The HyperAixpert is designed for easy integration into climate control chambers.

Though the HyperAixpert was designed to work with model species such as *Arabidopsis*, it equally handles early-stage seedlings of most crop species. Further, the multisensor plant phenotyping of the system is not restricted to just plants, several other sample types match with the system. For instance, assessment of fungal growth on plates or insects feeding on leaf discs are applications that have been investigated by the HyperAIxpert.

Analytik

http://link.spectroscopyeurope.com/32-093

Spectral imaging platform for industrial sorting

Specim has released the SpecimONE spectral imaging platform for the industrial sorting market. SpecimONE is compatible with major industrial standards and allows seamless integration with machine vision systems, e.g. Halcon and Sherlock. SpecimONE consists of three parts: the Specim FX series hyperspectral camera, SpecimCUBE processing hardware and SpecimINSIGHT off-line software. Specim FX series hyperspectral cameras are designed specifically for industrial use, with high frame rate, robust construction and small size. SpecimCUBE is a processing platform to run classification models created by SpecimINSIGHT in real-time, and includes optimised software to meet industry requirements for throughput, latency and jitter. SpecimINSIGHT

allows users to browse and explore data, create and validate classification models. The current feature set focuses on sorting industry requirements, namely, a robust, yet fast classification PLS-DA algorithm, support for MROI functionality to benefit from the FX series camera's speed and support for all Specim dataset format cameras.

Specim

http://link.spectroscopyeurope.com/32-100

INFRARED

Palm-sized FT-IR platform

Si-Ware Systems has introduced the NeoSpectra-MIR, a highly compact FT-IR spectral sensing platform that offers wide wavelength coverage (2125–7400 cm^{-1}). The customisable platform, which can fit in the palm of your hand, can be used on-site as a test station or installed directly onto a pipe or into a production line, reducing operational costs and improving efficiency and safety. Due to the MEMS construction of the interferometer, it is cost-effective, with volume pricing significantly less than benchtop equipment, has low power consumption, robustness to shock/vibration and low maintenance.

Si-Ware has developed a series of product demonstrators that can be used off-the-shelf, and customised models can be configured and optimised with an easy-to-use toolkit. The modular components include the optical core module, the electronics, sampling interfaces, software and connectivity tools, chemometrics and system-level packaging. The core technology

could even be incorporated into handheld or drone mounted devices.

Si-Ware Systems

http://link.spectroscopyeurope.com/32-096

MASS SPEC

New PTR3-TOF 10K mass spectrometer

IONICON have introduced their new generation PTR-TOF for quantification of volatile organic compounds (VOC), which is optimised for the detection of their oxidation products typically, present in the parts-per-quadrillion (ppq) range. The key to ultra-high sensitivities is the novel patented 3D tripole reaction chamber where long reaction times are combined with precise control of the ion chemistry via electric fields. A dual-stage core sampling inlet system, which enables analyte transfer with virtually no wall interactions, allows organics ranging from volatile to even extremely low volatility (ELVOC) to be measured, even at ambient sample inlet temperature. In addition, the PTR3 has the unique ability to detect and quantify RO_2 radicals.

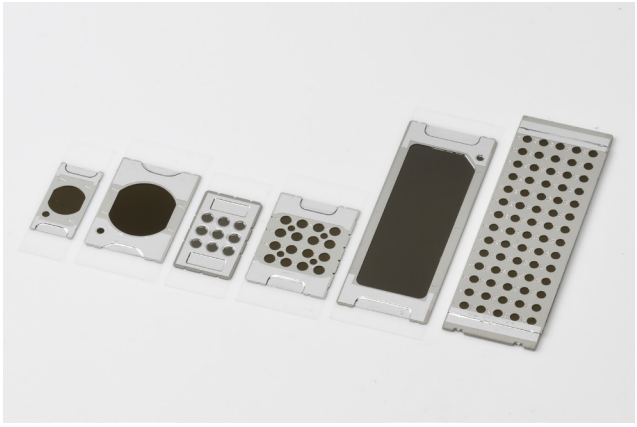
The latest instrument can achieve sensitivities of more than 50,000 cps/ppbv, e.g. for ketones. Ions are analysed with the novel high-resolution ioniTOF 10K, achieving mass resolving powers of typically 10,000–15,000 $m/\Delta m$.

IONICON

http://link.spectroscopyeurope.com/32-103

NEW PRODUCTS

Ionisation-assisting substrates for MALDI MS



Hamamatsu Photonics have added four new products to their "DIUTHAME (Desorption Ionisation Using Through Hole Alumina MEmbrane)" series of ionisation-assisting substrates, that drastically cut the pretreatment time needed to ionise samples by laser in mass spectrometry. These new products include a miniature type, ideal for mass spectrometry imaging of small samples, and a glass-slide size type for mass spectrum measurement that can be directly set into spectrometer holders. The expanded lineup for the DIUTHAME series now includes a total of nine types, allowing users to select the DIUTHAME that best matches the size of samples to measure with MALDI-TOF MS.

Hamamatsu Photonics

http://link.spectroscopyeurope.com/32-099

Direct mass detection system

Waters has introduced the new RADIAN™ ASAP™ system, a direct mass detector engineered for non-mass-spectrometry (MS) experts to conduct fast and accurate analyses of solids and liquids with minimal sample preparation. It uses single quadrupole MS technology, combined with a dedicated Atmospheric Solids Analysis Probe (ASAP) source, enabling results to be obtained in seconds after a sample is loaded into the system. Gaseous analyte molecules are ionised by N_2 plasma, guided into the instrument and separated by their mass-to-charge ratio. Users obtain real-time sample classification and quality assessment in less than a minute, without the need for a chromatographic separation.

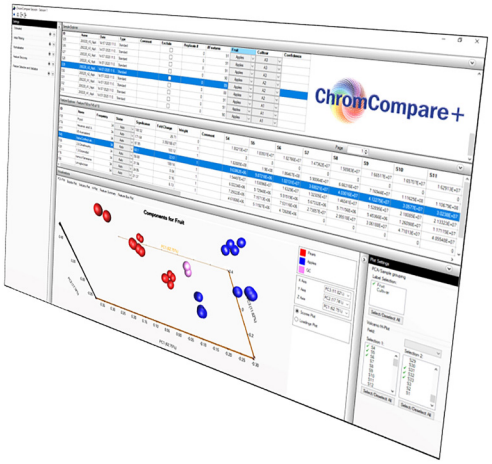
The RADIAN ASAP System is compatible with a variety of Waters software solutions, including OpenLynx, MassLynx™, IonLynx™ and LiveID™. Waters has released the latest iteration of its LiveID Software, LiveID 2.0, in conjunction with RADIAN ASAP. Offering an intuitive, modern interface and easy-to-interpret results, LiveID Software offers model building capabilities for classifying samples and determining their authenticity. LiveID Software now also offers real-time spectral library matching for identifying sample compounds.

There will be applications in pharmaceuticals, for instantaneous assessment of reaction progress and the identification of purification fractions; in forensics, to identify illicit drugs from a library of known compounds; in food and beverage, to verify product authenticity and safety, adulteration or degradation by suppliers and regulators; in chemicals and materials, to streamline quality control and product development workflows such as material release testing or formulation performance testing; and in academia, to supply academic laboratories with a robust and reliable solution for teaching and method development.

Waters

http://link.spectroscopyeurope.com/32-101

GC and GC-MS chemometrics platform



Sepsolve Analytical has announced ChromCompare+™, a chemometrics platform for comparing multiple GC and GC×GC chromatograms and extracting useful insights into the constituents that are present. It offers the flexibility to process data from a broad range of GC and GC-MS platforms and works with a wide range of file formats. ChromCompare+™ allows analysts to simplify data analysis using automated, untargeted workflows, discover hidden differences between sample classes, account for retention time drifts using automated alignment of chromatograms and use prediction models to automatically classify unknown samples. This is particularly valuable in discovery workflows, such as clinical research, food and beverage authenticity or fragrance profiling, when analysts do not know the important

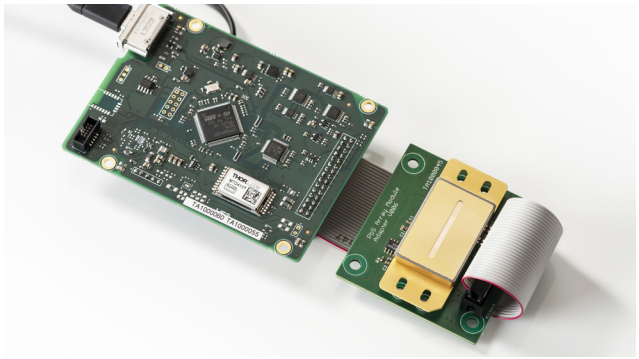
differences between sample classes, so must investigate all components, often within complex matrices.
Sepsolve Analytical
http://link.spectroscopyeurope.com/32-097

New vacuum pumps from Leybold

Leybold has expanded its ECODRY plus product family of dry multi-stage Roots vacuum pumps for laboratory, R&D and analytical applications. The new, smaller pumps, ECODRY 25 and 35 plus, complete the range of quiet, low-maintenance and economical fore-vacuum pumps and fill the gap between SCROLLVAC 18 plus and ECODRY 40 plus. The new pumps are particularly quiet, with a noise level of 52 dB(A). They also emit neither oil vapour nor particles, important in research institutes and laboratories where a clean working environment is essential. Their maintenance interval is five years, during which they require no servicing. Customers have the option of connecting a pressure gauge directly to the vacuum pump. With the help of the gauge, the pump monitors the pressure: if this is low enough, the pump can reduce its speed and becomes quieter, more energy-efficient and emits less heat.
Leybold
http://link.spectroscopyeurope.com/32-105

NIR

NIR line array modules



trinamiX has introduced the second generation of its 256-pixel NIR line array module, which have increased quantum efficiency and improved thermal stability. They are photoresistive lead sulphide detector arrays and are ITAR-free. They have peak detectivity at 2700 nm and a usable wavelength range of 1000–3000 nm at temperatures as high as 20 °C. An evaluation kit for the array module is available to help customers with system design and support a quick start to using the detector.
trinamiX
http://link.spectroscopyeurope.com/32-107

NMR

Bruker introduces new version of Fourier 80 benchtop FT-NMR system

Bruker has introduced the new Fourier™ 80 system, an 80 MHz Fourier Transform Nuclear Magnetic Resonance (FT-NMR) benchtop spectrometer equipped with a novel, ultra-stable 80 MHz permanent magnet; it requires no cryogens, water cooling or special lab infrastructure. The Fourier 80 has been designed for excellent line-shape, resolution and sensitivity in 80 MHz homonuclear ^{1}H or heteronuclear $^{1}H/^{13}C$ FT-NMR experiments. The latest version now offers even greater sensitivity and 20 % improved resolution performance. It can be operated by the easy-to-use GoScan™ software for NMR beginners, or by Bruker's TopSpin™ NMR software with the extensive TopSpin library of 1D and 2D

NEW PRODUCTS

homonuclear and proton–carbon heteronuclear experiments and pulse programs.

The latest version of the Fourier 80 now includes a pulsed field gradient which has been used in high-field NMR spectroscopy for decades to quickly and conveniently obtain essentially artefact-free spectra. Gradients allow users to enhance solvent or water suppression, perform DOSY experiments and acquire two-dimensional NMR spectra within minimal experiment time. Another new feature is the option of an industry-standard, robust and high-throughput PAL sample changer. Fourier 80 users with the PAL sample changer can run up to 132 samples, including 12 reference samples, thereby dramatically increasing throughput and efficiency. GoScan now also operates the sample changer to run samples overnight or over a weekend. The Fourier 80 offers workflows and protocols for academic, pharma and industrial chemistry research, as well as for forensics and organic synthesis control. It can be incorporated into science education to introduce students to the power of FT-NMR. An optional teaching package with recommended experiments and spectra interpretation guide is also available.

Bruker

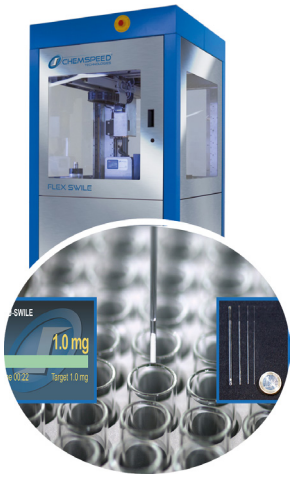
http://link.spectroscopyeurope.com/32-098

Flex Swile

Chemspeed Technologies have introduced a sample preparation solution for NMR spectroscopy and LC/MS. This makes use of Chemspeed's SWILE technology that provides automated one-to-one gravimetric "pick & decision dispense". Dispensing mass ranges from <100 µg to 100 mg with ±10 µg resolution. Disposable glass tips ensure there is no cross-contamination; they are available in different diameters depending on the amount to be dispensed. The NMR and LC/MS sample preparation solution provides automated sample dispensing into NMR tubes or LC vials.

Chemspeed Technologies

http://link.spectroscopyeurope.com/32-106



RAMAN

Handheld SERS analyser for food contamination

Metrohm has introduced the Misa: a handheld Raman instrument to detect and identify trace level food contamination in complex food samples in the field. Misa uses Surface Enhanced Raman Scattering (SERS) for trace detection of contaminant molecules. A mobile app provides intuitive, guided workflows with immediately available results and automated analysis quickly and accurately identifies trace contaminants. The instrument comes with dedicated applications which eliminate sample analysis method development. Further, the mobile platform enables remote sharing of results, location and hazard alerts.

Metrohm

http://link.spectroscopyeurope.com/32-095

New RMS1000 Raman microscope from Edinburgh Instruments

Edinburgh Instruments has launched the new RMS1000 Raman microscope, an open architecture, research-grade confocal Raman microscope. It has been designed to be adapted to almost any modern, state-of-the-art Raman application. Applications beyond Raman, such as time-resolved fluorescence microscopy and fluorescence lifetime imaging (FLIM), are possible with the RMS1000. It features integrated and external lasers, five-position grating turrets, two spectrograph options, is truly confocal, four simultaneous detectors, internal standards and auto-calibration and comes with Ramacle® software.

Users of the RMS1000 will also benefit from access to the KnowItAllTM Raman Identification Pro spectral library which is available for material identification and advanced analysis. Data acquisition methods such as single measurements, multiple and accumulated scans, kinetic scans and generation of maps (accessory dependent) are implemented by intuitive and user-friendly wizards.

Edinburgh Instruments

http://link.spectroscopyeurope.com/32-102

Renishaw introduces Particle Analysis module

Renishaw has introduced the Particle Analysis software module for its inVia™ confocal Raman microscope. The module automates the inVia microscope so that it can identify particles on images and then chemically analyse them using Raman spectroscopy. The software module pinpoints multiple particles for automated Raman analysis and reports the results in an easy-to-navigate format. It gives chemical information on each particle and its morphology statistics. This enables you to easily spot correlations between particle size, shape and chemistry. The new module also works with Renishaw's Correlate™ module so that images from other microscopy systems can be used to guide Raman analyses on the inVia microscope. The Particle Analysis module rapidly and comprehensively analyses multiple particles and can be used in many applications. The Particle Analysis software is an optional module for Renishaw's WiRE™ 5.4 software, for use with the inVia confocal Raman microscope.

Renishaw

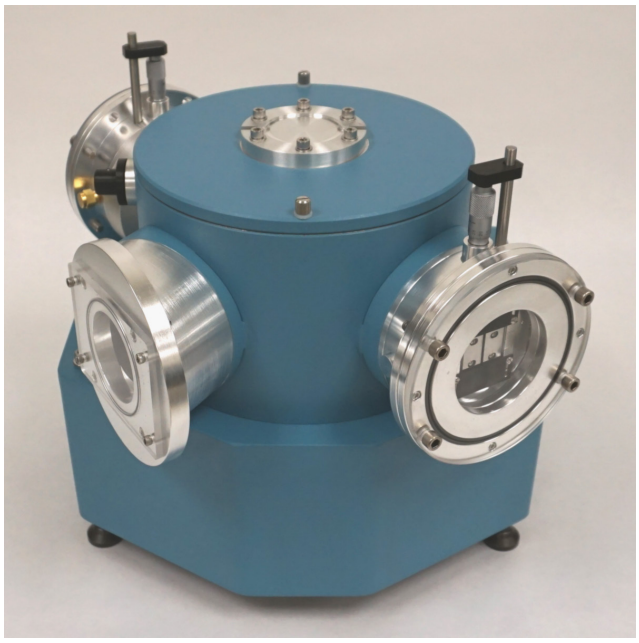
http://link.spectroscopyeurope.com/32-092

UV/VIS

Improved UV monochromator

McPherson has introduced the improved compact Model 234/302 monochromator. Internal surfaces have an optimised low scatter finish. It is available with a series of aberration-corrected diffraction gratings, allowing users to tune performance from 30 nm to 1100 nm. The optics have wavelength optimised reflective coatings, platinum, aluminium and aluminium enhanced with magnesium fluoride are available. Grating densities range from 2400 to 300 grooves per millimetre. Every instrument ships with masterpiece grating(s) for best instrument performance and lowest scatter. Customised gratings are available for special tasks. The new Model 234/302 may also be equipped with a grating turret. This helps the user by broadening the accessible wavelength range without breaking vacuum or purge.

The McPherson 234/302 has a digital grating drive for precise wavelength selection and positioning. Micrometer-adjustable slits vary from 0.01 mm to ~3 mm in width and 2 mm to ~20 mm in height. Software is available along with LabVIEW drivers. This instruments normal incidence design optionally has multiple input or output ports. It can be used as a spectrograph with a microchannel plate intensifier or CCD detector, or as a scanning monochromator, while remaining under vacuum.



Model 234/302 with third port ready for CCD

McPherson

http://link.spectroscopyeurope.com/32-104

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.):	Organisation Name:
First Name:	Country:
Last Name:	E-mail:
Job Title:	

☐ I wish to receive a free digital copy of *Spectroscopy Europe*. I understand that you will send e-mails to me to alert me to new content and the publication of each issue. 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
- ☐ Inductively Coupled Plasma (ICP)
- ☐ ICP/MS
- ☐ Atomic Fluorescence
- ☐ Chemicals and Reference Materials
- ☐ Data Handling/Chemometrics
- ☐ Gamma-Ray Spectroscopy
- ☐ Ion Mobility
- ☐ Imaging
- ☐ Infrared Spectroscopy
- ☐ Laser Spectroscopy
- ☐ LIBS
- ☐ Luminescence/Fluorescence
- ☐ Mass Spectrometry
- ☐ MRI
- ☐ Mobile Spectrometers
- ☐ Mössbauer Spectroscopy
- ☐ Near Infrared
- ☐ NMR, ESR, EPR
- ☐ Photonics and Optics
- ☐ Polarimetry
- ☐ Process spectroscopy
- ☐ Raman spectroscopy
- ☐ Sampling
- ☐ Separation Science
- ☐ Spectroradiometry
- ☐ Surface Analysis
- ☐ Terahertz Spectroscopy
- ☐ UV/Vis
- ☐ X-Ray Diffraction
- ☐ X-Ray Spectrometry

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_____

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

IM Publications Open LLP will use the information you have provided to fulfil your request.

We ask you to consider allowing us to send you information from relevant companies on their behalf. Of course, we never disclose your details to them. By allowing this you help us continue to provide *Spectroscopy Europe* to you free of charge.

☐ Tick to receive e-mail with information from carefully selected spectroscopy companies (your details are never disclosed)

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

Conferences

2021

24–26 February, Online. **11th Workshop on Hyperspectral Image and Signal Processing: Evolution in Remote Sensing (WHISPERS).** http://www.spectroexpo.com/whispers/

25 February, Online. **Hyperspectral Sensing Meets Machine Learning and Pattern Analysis (HyperMLPA).** http://www.spectroexpo.com/hypermlpa/

26 February, Online. **2nd Symposium on Short Wave Infrared Imaging and Spectroscopy (Swllms).** http://www.spectroexpo.com/swiims/

25–28 April, Oviedo, Spain. **5th International Glow Discharge Spectroscopy Symposium (IGDSS2021) (postponed from April 2020).** pete@masscare.co.uk, https://www.ew-gds.com/forthcoming-events/

June/July, Skagen, Denmark. **International Association for Spectral Imaging (IASIM).** 2020@iasim.net, https://2020.iasim.net

6–10 June, Philadelphia, PA, United States. **69th ASMS Conference.** https://www.asms.org/conferences/annual-conference/future-annual-conferences

21–25 June, Burgos, Spain. **LED2020.** led2020burgos@cenieh.es, https://led2020burgos.cenieh.es

18–23 July, Boston, MA, United States. **XXIX International Conference on Magnetic Resonance in Biological Systems (ICMRBSXXIX).** https://www.icmrbs2020.org

1–6 August, Freiberg (Sachsen), Germany. **Geoanalysis 2021.** geoanalysis2021@hzdr.de, https://geoanalysis2021.de.

6–10 September, Heraklion, Crete, Greece. **NanoBio Conference 2021.** info@nanobioconf.com, https://nanobioconf.com.

18–21 October, Trondheim, Norway. **2nd Nordic Metabolomics Conference.** mila.knoff@ntnu.no, https://www.ntnu.edu/isb/nmc2021.

Exhibitions

2021

24–26 February, Online. **Spectro Expo 2021.** https://www.spectroexpo.com.

8–12 March, Online. **Pittcon 2021: Conference on Analytical Chemistry and Applied Spectroscopy.** pittcon-info@pittcon.org, https://pittcon.org

Accelerate Your Publication

Letters published in 3 weeks subject to satisfactory review

JSI—Journal of Spectral Imaging is an Open Access, peer-reviewed journal dedicated to the rapid publication of leading research in spectral, hyperspectral and chemical imaging, as well as in related areas such as remote sensing, and chemometrics and data handling for spectral image data.

- *JSI* Letters offer extremely fast publication whilst retaining thorough peer review, with publication within three weeks of submission, subject to satisfactory review.
- *JSI* Letters can either express the view of the author on a particular aspect of spectral imaging that they wish to comment on, or can very briefly report an interesting aspect of the author's work: perhaps details of a chemometric routine or a sampling procedure. The conclusions should represent a significant advance in the understanding of an important issue and carry major implication for spectral imaging as a discipline.
- Authors are expected to submit revised manuscripts within 48 hours of receiving the referees' reports.
- Given the accelerated treatment accorded to Letters, it is particularly important that they are written to a standard of English to allow rapid refereeing.

impopen.com/jsi-author-centre