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I have driven past the World Heritage Site of Stonehenge many times, but never visited: something for my bucket list! So, I was fascinated to learn of how atomic spectroscopies have solved one of the mysteries around the stones of Stonehenge. The full story is on page 6, along with many other interesting applications of developments in imaging spectroscopy, circular dichroism, nano- and micro-spectroscopies, and medical applications including Alzheimer's, heart disease and cancer.

After this journey through some of the wide range of applications of the versatile tool of spectroscopy, Andreas Noelle and Gerd Hartmann describe *The UV/Vis+ Photochemistry Database*. This collection of photochemical data and information was started in 1999 and is now an online database updated weekly. Find out the full story on [page 17](#).

Gary Sharman, Marcel Simons and Tony Davies have a clear idea "When to Automate Spectroscopic Data Processing". I am sure everyone realises that automation is not a panacea, but it can improve the accuracy of manual tasks as well as freeing up our time for more challenging tasks. The authors explore some

particular examples they have come across and lessons learned from them.

In Kim Esbensen's Sampling Column, Jean-Sébastien Dubé and François Duhaime start a two-part series on "Chemical analysis of contaminated soil for sound environmental site assessment" with Part 1 on the critical role of proper sampling. Proper sampling of particulate matter for instrumental analysis is a common task, and is crucial for representative measurements. Unfortunately, it is often performed with a disregard for the Theory of Sampling, and so fails.

There are also our regular New Products and Diary sections. We have checked the events listed in the Diary and they are up-to-date as we go to press.

Finally, there are links to selected content published online that we have not had space to include here: [page 19](#).



La Michael

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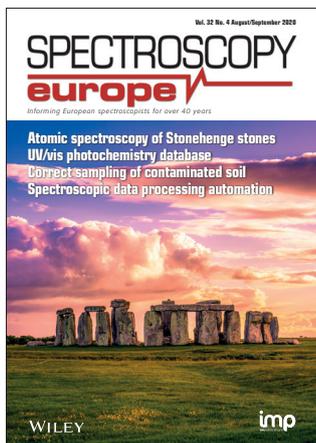
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Atomic spectroscopies have been used to identify the source of the huge (20 tonnes) sarsen stones at Stonehenge: they came from just down the road! Find out more on page 6.

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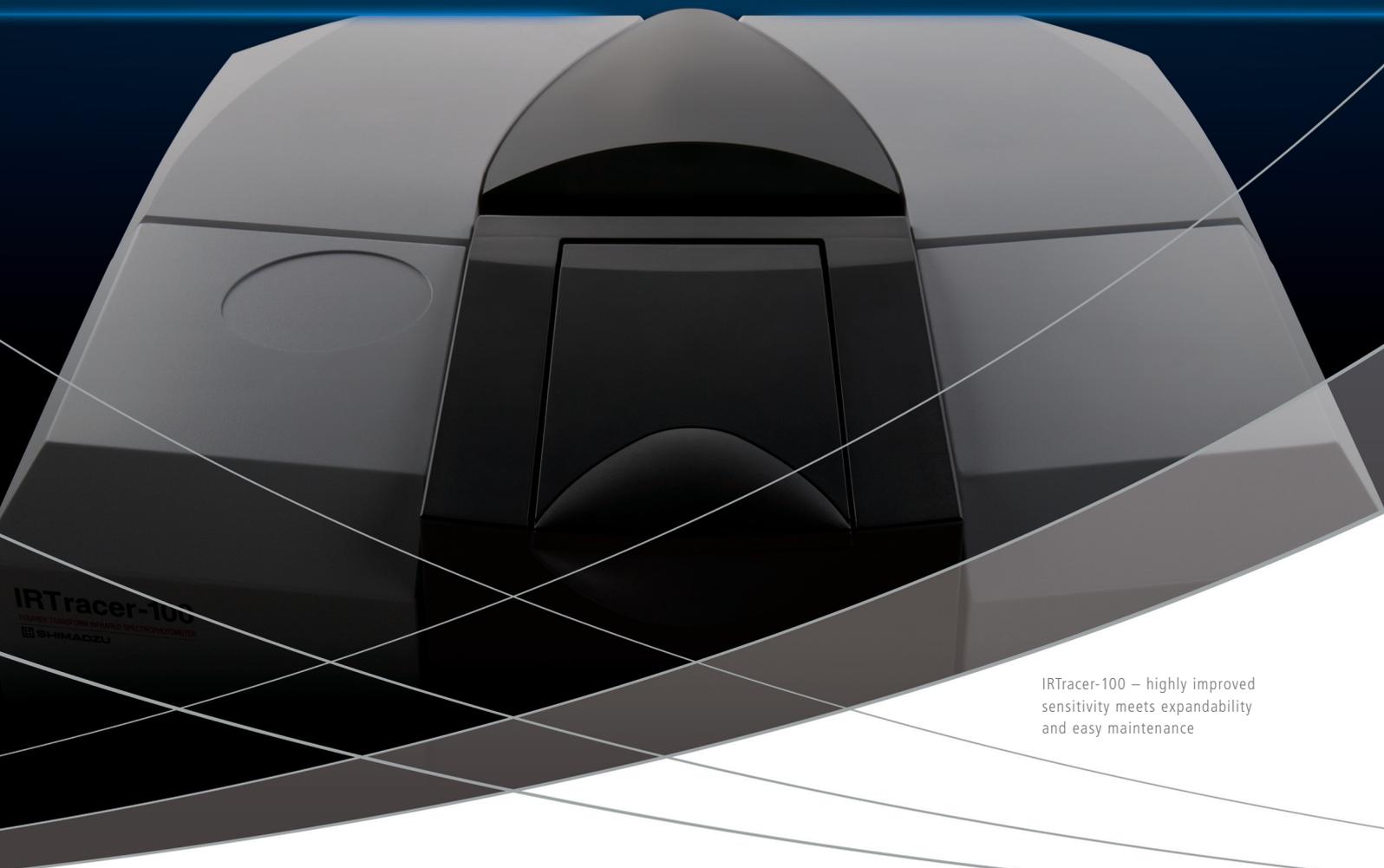
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The Shimadzu IRTracer-100

New Levels of Performance and Quality Created by Excellent Sensitivity, Speed and Resolution



Atomic spectroscopy of Stonehenge stones reveals their origin

The ancient stone circle at Stonehenge in the UK has been of fascination for us and our ancestors for around 5000 years. Now, we wonder at how the vast stones were transported to the site and erected. In the past, the fascination was with the movement of the Sun and the passing of the seasons. It has been known for some years that the smaller “bluestones” came from the Preseli Hills in Wales. However, larger (up to 7 m) and heavier (20 tonnes) stones, known as sarsens, are also used at Stonehenge, and the origin of these—more homogeneous in composition—has been impossible to identify until now.

A paper in *Science Advances* (doi.org/d5d7) reports a study that has pinpointed the source of the sarsens to an area around 15 miles north of the stone circle site. The breakthrough came when a core—drilled from Stonehenge’s “Stone 58” during repair work in the 1950s—was returned to English Heritage from Florida last year. This was at the request of one of those involved at the time, Mr Robert Phillips. This presented a unique opportunity to analyse the interior of one of the sarsens with destructive techniques.

The team first carried out non-destructive testing of all the remaining sarsens at Stonehenge using portable x-ray fluorescence spectrometry (pXRF). This revealed that most, including Stone 58, shared a similar chemistry and came from the same area. They then analysed sarsen outcrops from across the south of the UK using inductively coupled plasma mass spectrometry (ICP-MS) and ICP-atomic emission spectrometry (ICP-AES). The return of the core drilled from Stone 58 in the 1950s offered the opportunity to perform destructive analysis as well using ICP-MS and ICP-AES, and to compare this with the compositions of a number of stones from possible sources around the country. This showed that the composition of Stone 58 matched the chemistry of sarsens at West

Woods, just south of Marlborough: about 40 minutes drive from Stonehenge today.

English Heritage Senior Properties Historian Susan Greaney said: “To be able to pinpoint the area that Stonehenge’s builders used to source their materials around 2500 BC is a real thrill. Now we can start to understand the route they might have travelled and add another piece to the puzzle. While we had our suspicions that Stonehenge’s sarsens came from the Marlborough Downs, we didn’t know for sure, and with areas of sarsens across Wiltshire, the stones could have come from anywhere. We can now say, when sourcing the sarsens, the over-riding objective was size—they wanted the biggest, most substantial stones they could find and it made sense to get them from as nearby as possible. This is in stark contrast to the source of the bluestones, where something quite different—a sacred connection to these mountains perhaps—was at play. Yet again this evidence highlights just how carefully considered and deliberate the building of this phase of Stonehenge was.”

Professor David Nash, University of Brighton, said: “It has been really exciting to harness 21st century science to understand the Neolithic past, and finally answer a question that archaeologists have been debating for centuries. We were able to investigate the chemistry of the sarsens at Stonehenge using x-ray fluorescence, a non-destructive technique. This showed that most of the stones shared a similar chemistry and likely came from a similar source. We then applied mass spectroscopy to samples from sarsen outcrops across southern England and to tiny pieces of the Phillips’ Core from Stonehenge. Each outcrop was found to have a different geochemical signature, but it was the chance to test the returned core that enabled us to determine the source area for the Stonehenge sarsens. We’re incredibly grateful to the Phillips family for returning the core to us.”

Imaging spectroscopy to track plant pathogens through the atmosphere

The multidisciplinary team of scientists has been selected for a \$750,000 NASA grant to combine their expertise in remote sensing, climate and earth system computer modelling, plant pathology and genomics to study the effects of soilborne plant pathogens which can travel in dust clouds from Africa to the Western Hemisphere. They will also use Earth system modelling to predict how regions will change over time and how that may influence plant disease dispersal with dust.

If the origins and landing spots of specific pathogens can be better predicted, farmers can be advised on how to avoid practices that would increase its spread, such as those that kick up dust from farm fields, and perhaps grow less susceptible crops where such dust falls.

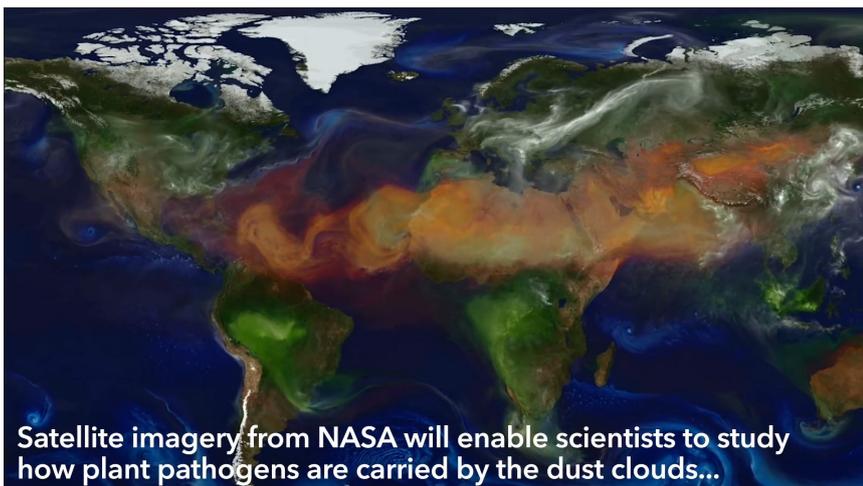
"We lose anywhere from 15% to 30% of the global harvest to plant diseases annually; here in 2020, people still die because they don't have access to food, because of losses due to plant disease", said principal investigator Katie Gold,

assistant professor of plant pathology. "Remote sensing can do a lot help mitigate the impacts of plant disease on the global food supply."

"It's just a fascinating combination of cross-disciplinary work that's going to allow us to address things that no one has been able to address before", said co-investigator Natalie Mahowald, professor of Earth and atmospheric sciences and an expert in atmospheric modelling.

NASA's Release of Research Opportunities in Space and Earth Science Interdisciplinary Science grant is for three years, which will allow the team to lay the foundation for a global surveillance system to assess risk and track and potentially prevent the global spread of plant diseases.

Other co-investigators include Ryan Pavlick, an imaging spectroscopy technologist at NASA's Jet Propulsion Lab, and Sharifa Crandall, assistant professor of soilborne disease dynamics and management at Pennsylvania State University.



NASA remote sensing satellites will help Cornell University faculty members Katie Gold, assistant professor of plant pathology, and Natalie Mahowald, professor of Earth and atmospheric sciences, study the effects of soil-borne plant pathogens which can travel in dust clouds from Africa to the Western Hemisphere. Credit: John Munson/Cornell University

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Company News

130 years of Pfeiffer Vacuum

Pfeiffer Vacuum is celebrating its 130 years in business. The company introduced the turbomolecular pump in 1958. Arthur Pfeiffer founded the company in Wetzlar, Germany, in 1890, and he initially devoted his attention to the production of remote ignition systems for gas lamps. Once electric light bulbs had become established, the company founder quickly turned his attention to the new lighting technology, which led him to look at the vacuum technology used in its production. Arthur Pfeiffer quickly recognised the significance that vacuum technology could have in practically all areas of industry and research—and he subsequently concentrated entirely on this field.

Partnership developing low-cost hyperspectral imaging system

Academics at the University of the West of Scotland (UWS), the University of Strathclyde (UoS) and the James Hutton Institute (JHI), led by Glasgow-based product design firm Wideblue, are developing a new type of hyperspectral imaging (HSI) system. The UK Government-funded collaboration has the potential to introduce an affordable spectral imaging technology to help agricultural businesses monitor and maximise crop production in fields and greenhouses. The sensors in development are expected to be up to 90% cheaper than equivalent equipment currently on the market and have the potential to make high-resolution spectral imaging technology significantly more accessible to the agricultural sector and beyond.

The team's HSI system, which is being manufactured by Wideblue, uses UWS-designed linear variable optical filters. This spectral image data is subsequently captured and analysed using data processing software designed by UoS. It is anticipated that adopting the technology will allow farmers to monitor various crop attributes including plant

LC-MS analysis of municipal wastewater as a predictor for population socioeconomics

Analytical techniques have been used for many years to monitor chemicals in sewage, for instance tracking cocaine use through its metabolites. Now, an extensive analytical study has been coordinated with the 2016 Australian Census. In a paper published in *Environmental Science & Technology Letters* ([doi.org/d5s8](https://doi.org/10.1039/C9EM00158A)), Dr Saer Samanipour of the Van 't Hoff Institute for Molecular Sciences (HIMS) in Amsterdam reported on the use of liquid chromatography tandem mass spectrometry for analysis of municipal wastewater as a predictor for population socioeconomics.

The study was carried out jointly with colleagues at the Queensland Alliance for Environmental Health Sciences of the University of Queensland, Australia. By coinciding the sampling phase of the study with the 2016 Australian Census, the researchers were able to correlate the socioeconomic characteristics of Australian communities (following from the census) with sewage composition (following from chemical analysis).

The researchers collected samples from more than 100 sewage-treatment plants and analysed these for the occurrence of "marker" chemicals such as nicotine, caffeine, amphetamine and many more. In total, they looked for 40 chemicals that revealed smoking habits, dietary characteristics and the use of drugs, antidepressants and antipsychotics.

The extensive chemical analysis of all samples, combined with advanced data analysis, led to the conclusion that the chemical fingerprint of sewage is a quite accurate predictor of the demographic make-up of the community connected to the sewage plant. The researchers developed a model based on their results and tested this with data from nine sewage plants not included in the initial analysis. For the communities around these plants, they were able to estimate key data on education, occupation, income, social life, housing and more within a 25% error margin. They were able to



make quite specific predictions ranging from the number of people that had finished secondary school to the number of single parents and car owners.

The researchers conclude that wastewater analysis can be a valuable technique for assessing population socioeconomics, complementary to the vastly more expensive and logistically challenging method of a census.

Airborne water leak detection using spectral imaging

Almost half of countries' drinking water may be lost before it reaches the tap. A large share of the losses happens in large diameter mains crossing rural areas, where companies have trouble monitoring them due to traditional field surveys being costly and time consuming. European researchers have developed a surveillance service using planes (to survey wide areas) and drones (for sites that are difficult to access) equipped with multispectral and infrared cameras. To analyse the data, they used the "Triangle method", which combines surface temperature measurements and a vegetation index. This is based on



health, hydration levels and disease indicators. As a result, it is envisaged that farmers will be able to optimise the impact of fertilisation; save water by employing more efficient irrigation methods; and, critically, spot diseases and infestations at an earlier stage, allowing them to avoid blights.

The consortium has applied for a patent on the overall low-cost hyperspectral imaging camera technology which is currently being deployed on a trial basis at a number of locations including the James Hutton Institute, Dundee.

Hyperspectral imaging partnership

Headwall Photonics and perClass BV have announced a partnership to enable the generation of classification models using the Headwall MV.X and perClass Mira software on a PC. Those models are then uploaded to IP67-rated MV.X imaging systems on the production line with no need for dedicated computers to store and process the raw hyperspectral data. The MV.X systems host a runtime version of perClass Mira that outputs the classified data through an industry-standard GenICam interface to robotic systems to take action along the line without any further processing. Models can be easily updated if requirements or situations change; for example, the material being inspected changes or even more accurate classification models are developed over time.

Merck acquires Resolution Spectra Systems

Merck has acquired Resolution Spectra Systems, a company producing Raman spectroscopy instrumentation for bioprocess analytical monitoring. Financial details of the acquisition were not disclosed.

Analytik Jena Celebrates 30th Anniversary

Analytik Jena is celebrating its 30th anniversary. The company was founded in

the fact that leaks lead to lower surface temperatures, which can be detected by a thermal infrared camera. However, the thermal response of vegetated soils is different from bare ones, making it difficult to obtain an unequivocal answer in terms of moisture content and potential water loss. The researchers, therefore, added a parameter measuring the vegetation cover fraction, which is inferred by hyperspectral cameras, to get a temperature-dependent humidity scale which varies according to the vegetation. The

system has been developed under the EU [WADI project](#).

Compared to competing technologies, the WADI system's can monitor complex networks and long pipes (50–90 km h⁻¹ depending on the use of drones or planes) and, as it's airborne, can reach inaccessible or secluded locations over all kinds of terrain. Moreover, the cost of conventional ground detection techniques ranges from 1000 to 5000 euro per km, while the airborne technology ranges from 50 to 200 euro per km.

Spectral imaging identifies health of single trees

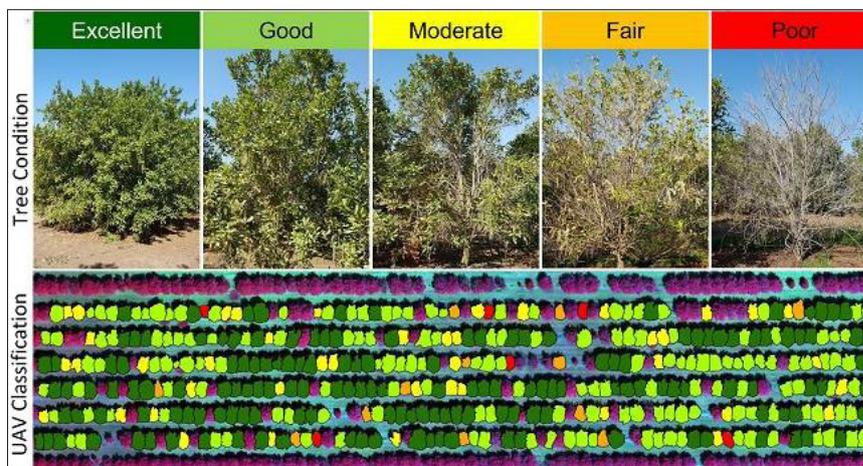
Researchers Kasper Johansen and Matthew McCabe at KAUST's Water Desalination and Reuse Center, with co-workers in Australia, have demonstrated the potential of using combined data from satellite and unmanned aerial vehicle (UAV) flights to accurately map the condition of macadamia trees in Queensland. Macadamia nuts are an important export for Australia, but the trees take 15 years to mature and provide their maximum yield, so farmers must identify early any trees that are suffering. "Currently, macadamia farmers rely on manual visual assessment of each tree and its irrigation system, which is time-consuming and often inconsistent", says Johansen, "whereas UAV and satellite remote

sensing could evaluate thousands at a time."

The team studied three plantations containing trees of various ages and varieties. The health of each tree was assessed by an experienced agronomist using a five-point scale from excellent to poor condition. The researchers flew a UAV over the sites to record spectral images in green, red and near infrared, and they acquired data across several more spectral bands from the Worldview-3 earth observation satellite. Using training data from a few hundred trees, the team developed models relating the spectral data to the ground measurements. The models were then able to correctly classify over 98% of the other trees.

May 1990 by Klaus Berka, Walter Maul and Jens Adomat as one of the first companies created in Jena after the fall of the Berlin Wall. During the 1990s, the company expanded both nationally and internationally. This was followed by the addition of production and development capacities for proprietary analytical solutions such as spectrophotometers, spectrometers and elemental analysers. The IPO in 2000 initiated a further phase of international expansion and the business was gradually expanded to include the life science, liquid handling and laboratory automation sectors. Since 2015, the Jena-based company has been a wholly owned subsidiary of the Swiss family business Endress+Hauser.

Analytik Jena plans to celebrate the anniversary with their customers, partners and employees in a special way. Thirty selected stories from customers, partners and employees will be published throughout the year on www.analytik-jena.com/30years.



By combining data from UAV and satellite surveys, Johansen and co-workers were able to classify the health of individual trees on a five-point scale across large areas of macadamia plantations. Farmers can then use a map, like the one shown, to find and treat trees at risk. ©2020 Kaspar Johansen

Interestingly, although the detailed spectra from Worldview-3 provided the most accurate models of tree health, the pixel size of 1.2-m was too coarse to identify individual tree crowns, which could only be done with the

high-resolution UAV data. This highlights the benefits of a combined UAV-satellite approach.

Their work was published in *ISPRS Journal of Photogrammetry and Remote Sensing* ([doi.org/d45j](https://doi.org/10.1016/j.isprsjprs.2020.04.015)).

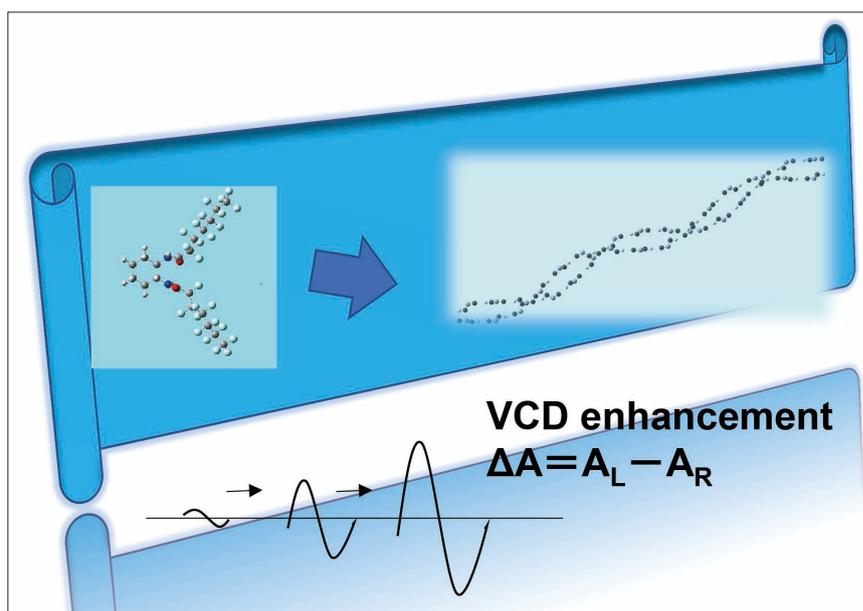
A new horizon for vibrational circular dichroism spectroscopy

Vibrational circular dichroism (VCD) spectroscopy is an extension of circular dichroism spectroscopy into the infrared (IR) and near infrared regions where vibrational transitions occur in the ground electronic state of a molecule. The method offers the advantage of studying the chiroptical properties of a wide range of molecules in non-crystalline states. However, due to the weakness of the signals, one measurement requires several hours to yield reliable results. Accordingly, its target has been limited to a stable molecule in a solution. To overcome this difficulty, a group at Ehime University applied the VCD method to supramolecular systems. Their results are reported in *Physical Chemistry Chemical Physics* ([doi.org/dw9z](https://doi.org/10.1039/C9CP00000A)).

Solid state and time-step VCD methods were developed to determine the mechanism of chirality amplification from the microscopic to supramolecular scales. The VCD signals were enhanced in the following three cases: (i) chiral gels with hundreds of

molecules arranged in stereoregularity, (ii) chiral metal complexes with low-lying excited states in the IR region and (iii) a molecular pair interacting

stereo-selectively on a solid surface. Finally, they described an on-going project involving the construction of a multi-dimensional VCD system.



Time-step VCD measured monitoring in situ the progress of chirality amplification from microscopic to supramolecular scales (©Royal Society of Chemistry).

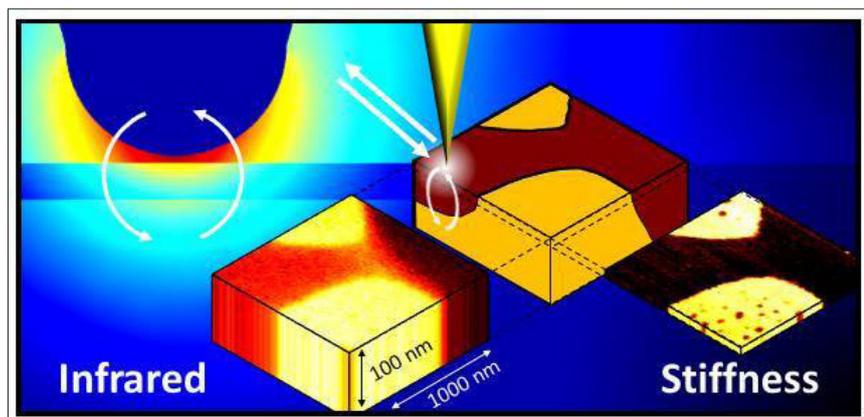


Illustration of sub-surface infrared nanoimaging. Copyright: CIC nanoGUNE

IR nanospectroscopy in 3D

Researchers from the Nanooptics Group at CIC nanoGUNE (San Sebastian, Spain) have shown that nanoscale infrared imaging, which is currently used as a surface-sensitive technique, can be employed for chemical nanoidentification of materials that are located up to 100 nm below the surface. The results further show that the infrared signatures of thin surface layers differ from those of sub-surface layers of the same material, which can be exploited to distinguish the two cases. The findings, published in

Nature Communications (doi.org/d4vb), push the technique one important step further to quantitative chemometrics at the nanoscale in three dimensions.

Optical spectroscopy with infrared light, such as Fourier transform infrared (FT-IR) spectroscopy, allows for chemical identification of organic and inorganic materials. The smallest objects which can be distinguished with conventional FT-IR microscopes have sizes on the micrometre-scale. In nano-FT-IR (which is based on near-field optical microscopy), IR light is scattered at a sharp metallised tip of a scanning-probe microscope. The tip is

scanned across the surface of a sample of interest and the spectra of scattered light are recorded using FT detection principles. Recording of the tip-scattered light yields the sample's IR spectral properties and thus the chemical composition of an area located directly below the tip apex. Because the tip is scanned across the sample surface, nano-FT-IR is typically considered to be a surface-characterisation technique.

Importantly though, the IR light that is nano-focussed by the tip does not only probe a nanometric area below the tip, but in fact probes a nanometric volume below the tip. Now the researchers at CIC nanoGUNE showed that spectral signatures of materials located below the sample surface can be detected and chemically identified up to a depth of 100 nm. Furthermore, the researchers showed that nano-FT-IR signals from thin surface layers differ from that of sub-surface layers of the same material, which can be exploited for determination of the materials distribution within the sample. Remarkably, surface layers and sub-surface layers can be distinguished directly from experimental data without involving time-consuming modelling.

Improved AFM-IR method

Researchers at the Beckman Institute for Advanced Science and Technology have developed a new method to improve the detection ability of nanoscale infrared chemical imaging using atomic force microscopy. These improvements were developed in the lab of Rohit Bhargava and will reduce the noise that is associated with the microscope, increasing the precision and range of samples that can be studied. The study was published in *Nature Communications* (doi.org/d5v8).

Atomic force microscopy is used to scan the surfaces of materials to generate an image of their height, but the technique cannot easily identify the molecular composition. Researchers have previously developed a combination of AFM and infrared spectroscopy called AFM-IR. The AFM-IR microscope uses a cantilever to measure subtle

movements of the sample introduced by shining an IR laser. The absorption of light by the sample causes it to expand and deflect the cantilever, generating an IR signal.

"Although the technique is widely used, there is a limit to its performance", said Bhargava. "The problem is that there were unknown sources of noise that limited the quality of the data."

Catherine Murphy, the head of the Department of Chemistry and the Larry Faulkner Endowed Chair in Chemistry, said this new technique developed by the Bhargava lab is exciting. "Our group is interested in using this technique immediately to learn about protein deformation on complex surfaces", Murphy said.

The researchers created a theoretical model to understand how the instrument works and, therefore, identify the

sources of noise. Additionally, they developed a new way to detect the IR signal with improved precision.

"The cantilever deflection is susceptible to noise which becomes worse as the deflection increases," said Seth Kenkel, a graduate student in the Chemical Imaging and Structures Laboratory, which is led by Bhargava. "Instead of detecting cantilever deflection, we used a piezo component as a stage to maintain zero deflection. By applying a voltage to the piezo material, we can maintain small deflection with low noise while recording the same chemical information which is now encoded in the piezo voltage."

Instead of moving the cantilever, the researchers use the movement of the piezo crystal to record the IR signal. "This is the first time anyone has controlled a piezo actuator to detect the signal. Other

researchers work around challenges such as noise by using more complex detection systems that don't address the underlying problems associated with AFM-IR", Kenkel said.

"People have only been able to use this technique to measure samples that have a strong signal because of the noise problem", Bhargava said. "With the improved sensitivity, we can image a much smaller volume of samples, like cell membranes."

In addition to measuring more diverse samples, the researchers also hope to use this technique to measure smaller sample volumes. "We could use this technique to look at complex mixtures that are present in small volumes, like a single lipid bilayer", Bhargava said.

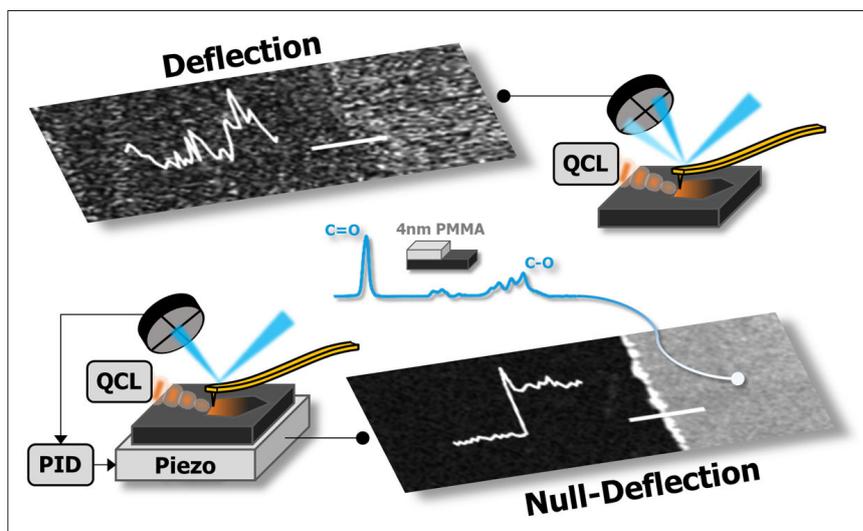


Image illustrating the previous deflection AFM-IR detection compared to the new null-deflection approach.

Infrared microspectroscopy reveals preservation of ancient fossils

Our understanding of life on Earth millions of years ago derives largely from our knowledge of fossils. However, most fossils are from only the last ~10% of Earth's history, which is just 500 million years. Fossils that are over 500 million years old are extremely rare because early organisms were microscopic, only the thickness of a hair and lacked hard shells or skeletons that could resist decay and allow them to become part of the fossil record. To understand how these early organisms could be preserved, IR microspectroscopy was performed using the Multimode Infrared Imaging and Microspectroscopy (MIRIAM) beamline at Diamond Light Source. IR microanalysis allowed researchers to identify at the micron scale the minerals surrounding 800–1000 million-year-old microfossils, and it was determined that an aluminium-rich clay known as kaolinite was responsible for their preservation. Kaolinite was previously shown to be toxic to bacteria, so its presence prevented the early organisms from being destroyed.

These observations suggest that the early fossil record might be biased to regions that are rich in kaolinite, such as the tropics. Moreover, the lack of animal fossils in these samples, despite having favourable fossilisation conditions

demonstrates that animals were yet to evolve 800 million years ago.

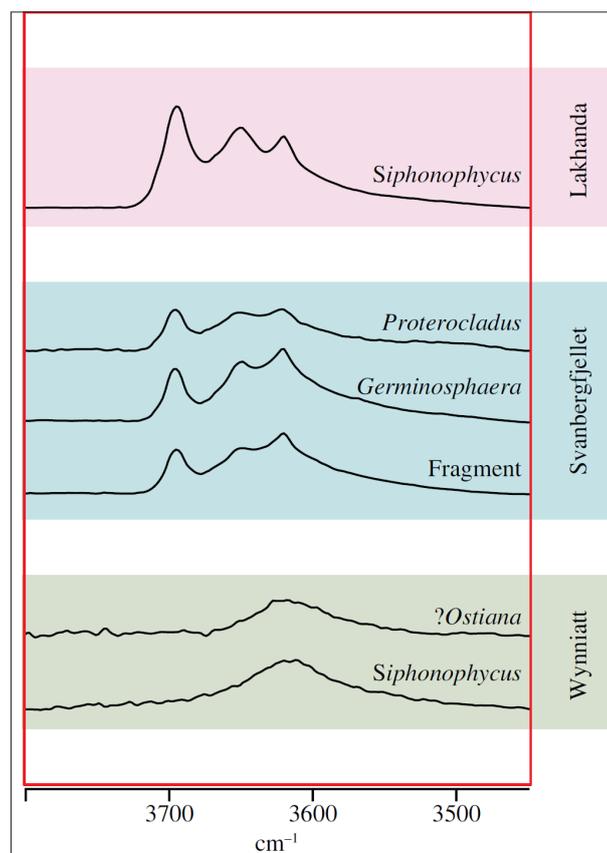
The collaboration between University of Oxford, Harvard University, Massachusetts Institute of Technology, Yale University and Diamond Light Source set out to investigate how these microfossils could be preserved. Lead author of the study and Post-Doctoral Research Fellow at the University of Oxford, Dr Ross Anderson, says: "We want to know what the conditions are that are conducive to preservation because then we can more easily find these elusive fossils. It will also help us to understand how this early fossil record is biased towards certain environments or time periods that are more favourable to preservation."

The team investigated microfossils that had been exceptionally preserved in mudstone from three locations: the billion-year-old deposits of the Lakhanda Group in Russia, and the 800 million-year-old deposits of Svanbergfjellet in Svalbard and Wynniatt in Canada. The fossils from these time periods are microscopic, not visible to the eye and are compressed into two dimensions parallel to the layers of rock. Dr Anderson explains: "To find them we have to cut a very thin layer of rock about 30 μm in thickness parallel to the layers of

sediment, from which the fossil is visible under a microscope. Once we've found the fossil, we prepare it for mineralogical analysis by using an ion beam to cut a vertical slice of the fossil that is only 1 μm thick."

The thin sections of rock were prepared at Oxford and Harvard and analysed at Diamond for mineralogical microcomposition. Dr Anderson and his team were interested in the composition of the rock around the fossils to understand why they had been preserved. To explore the mineralogy of the rock at the micron scale around the fossil and to obtain enough signal to detect the clay changes, both the spatial resolution and the sensitivity allowed by the IR microscope at the MIRIAM beamline at B22 were needed. Synchrotron IR microanalysis was the optimum method to experimentally prove the difference in types of minerals around the preserved microfossils.

Recent studies by Anderson and colleagues had identified that the clay mineral kaolinite, which is rich in aluminium, is toxic to the types of bacteria responsible for the decay of marine organisms. Intriguingly, IR microspectroscopy showed that the microfossils were also coated in the same kaolinite, which resembled a preservative halo. Dr Anderson notes: "It would suggest



Representative synchrotron IR microspectra (M–OH region) showing the mineralogical compositional changes of the clays of the different microfossils. Picture credit: Diamond Light Source

that the early fossil record is biased to environments where kaolinite is likely to be common—typically tropical environments. Interestingly, all the fossils studied come from rocks that were formed in the tropics.”

Importantly, the lack of animal fossils in the 800 million-year-old rocks, despite having favourable preservation conditions, strongly indicates that animals had not evolved at this time point. Another important implication of the protective kaolinite haloes is in the discovery of fossils on other planets. Having a rich understanding of how microscopic life can be fossilised is key to finding proof of life on other planets. In fact, NASA launched the *Perserverance* rover to Mars on 30 July 2020 to collect rock and soil samples to uncover signs of possible life. It is believed its Martian landing site, the Jezero Crater, could have kaolinite, which would hopefully improve the chances of finding intact microfossils.

In the future, Dr Anderson and the rest of the team intend to study microfossils from other deposits of different time periods. He concludes: “The method we have developed really enhances

the tools with which palaeontologists can characterise the composition of exceptionally preserved fossils and their surrounding rock. It will be applicable to a wide range of questions across the geological record.”

The work is reported in *Interface Focus* (doi.org/dzcy).

Charge detection MS provides insight into protein co-aggregation in Alzheimer’s disease

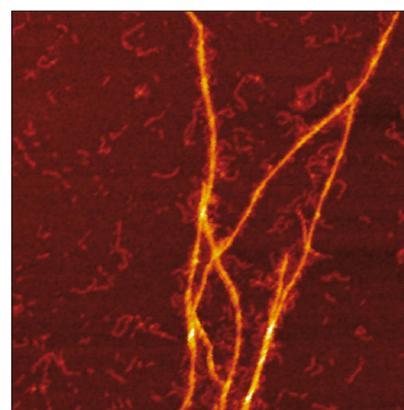
A research team lead by Ludmilla Morozova Roche of Umeå University, Sweden, has provided the mechanistic insight into protein co-aggregation in Alzheimer’s disease. The templating mechanism of S100A9 amyloids on A β fibrillar surfaces during co-aggregation process was revealed by synergy of biophysical methods including charge detection mass spectrometry, microscopy, kinetic and microfluidic analyses.

Amyloid formation is of the key clinical significance, since this process is involved in numerous neurodegenerative diseases such as Alzheimer’s, Parkinson’s and others. These diseases

affect millions of the aging population world-wide. Often it is difficult to draw the line between these diseases or they may occur simultaneously, which is known as disease comorbidity.

While the amyloid formation process was studied extensively, little is known about the specific mechanisms of co-aggregation of different amyloid species together, underlying the comorbidity of the diseases. In Alzheimer’s disease, the amyloid-neuroinflammatory cascade is manifested in co-aggregation of A β with proinflammatory S100A9 protein, which leads to intracellular and extracellular amyloid assembly, amyloid plaque depositions and cellular toxicity.

Deciphering the interactions between proinflammatory S100A9 protein and A β_{42} peptide in Alzheimer’s disease is fundamental since inflammation plays a central role in the disease onset. Here the researchers used charge detection mass spectrometry (CDMS) together with biophysical techniques to provide mechanistic insight into the co-aggregation process and differentiate amyloid complexes at a single particle level. Combination of mass and charge distributions of amyloids together with reconstruction of the differences between them and detailed microscopy reveals that co-aggregation involves templating of S100A9 fibrils on the surface of A β_{42} amyloids. Kinetic analysis further corroborates that the surfaces available for the A β_{42} secondary nucleation are diminished due to coating by S100A9 amyloids, while



AFM image of the amyloid templating of S100A9 fibrils on the surface of A β fibrils. Image: Jonathan Pansieri

the binding of S100A9 to A β ₄₂ fibrils is validated by a microfluidic assay. The researchers demonstrate that synergy between CDMS, microscopy, kinetic and microfluidic analyses opens new directions in interdisciplinary research.

The work was published in *Chemical Science* (doi.org/d5w3).

NIR spectroscopy poised to improve treatment for serious heart arrhythmia

Researchers have demonstrated that a new mapping approach based on near infrared (NIR) spectroscopy can distinguish between fat and muscle tissue in the heart. This distinction is critical when using radiofrequency ablation to treat a serious heart rhythm problem known as ventricular tachycardia. Radiofrequency ablation, the only treatment for ventricular tachycardia, involves identifying areas of the heart that are triggering abnormal signals and then heating them to the point that abnormal signals can no longer be transmitted. During the procedure, it's important, yet challenging, to identify precisely where to deliver energy while avoiding healthy tissue.

In *Biomedical Optics Express* (doi.org/d3dd), research team leader Christine P. Hendon from Columbia University and a multidisciplinary group of colleagues show, for the first time, that an ablation

catheter incorporating NIR spectroscopy mapping can successfully distinguish various tissue types in hearts donated from patients with cardiovascular disease. Today, most clinical heart mapping systems are based on functional measurements such as voltage. "An optical measurement that provides information about the underlying tissue composition has the potential to be used with standard functional methods to improve ablation success rates", said Hendon.

"By using near infrared wavelengths in addition to visible wavelengths, we can probe deeper into the tissue", said Hendon. "The technique lets us distinguish various types of tissue within human hearts because fat, muscle and ablation lesions all have different scattering and absorption wavelength dependent properties."

The approach could not only be used to guide ablation procedures and evaluate how well they worked, but might also provide information that could be used to develop new computational models that would help advance the understanding of mechanisms involved in arrhythmia.

"Once an abnormal area has been identified and heated to form an ablation lesion, it is important for the operator to know if that lesion was placed successfully and had the desired effect," said Hendon. "Direct measurement of tissue characteristics affords the possibility of

improved ability both to find abnormal tissue and to determine how well it has been treated."

Using NIR spectroscopy during radiofrequency ablation required the researchers to develop new ablation catheters that incorporated optical fibres for emitting and detecting light, as well as a custom tip for tracking the instrument. They also developed new signal and data processing techniques, a workflow for rendering anatomical tissue maps and a catheter tracking system to enable spatial mapping of the tissue.

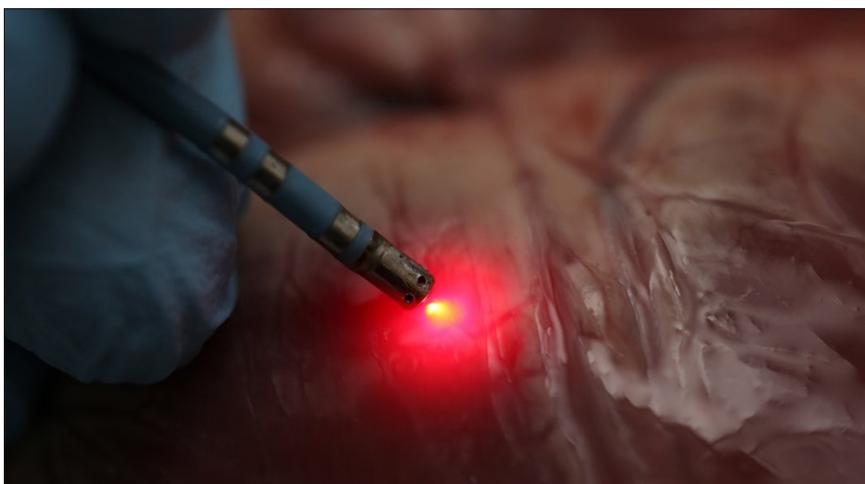
Using the new catheter, the researchers tracked the position of the instrument as it moved along the heart surface. At each location they recorded reflectance spectra and used this to compute an optical index for both fat and lesion tissues. The experiments were performed on donor hearts from deceased people with cardiovascular disease to replicate what would likely be encountered in the clinic.

"So far we have extremely encouraging results", said Hendon. "Our work shows that optics can have a large and impactful role within the field of cardiac electrophysiology."

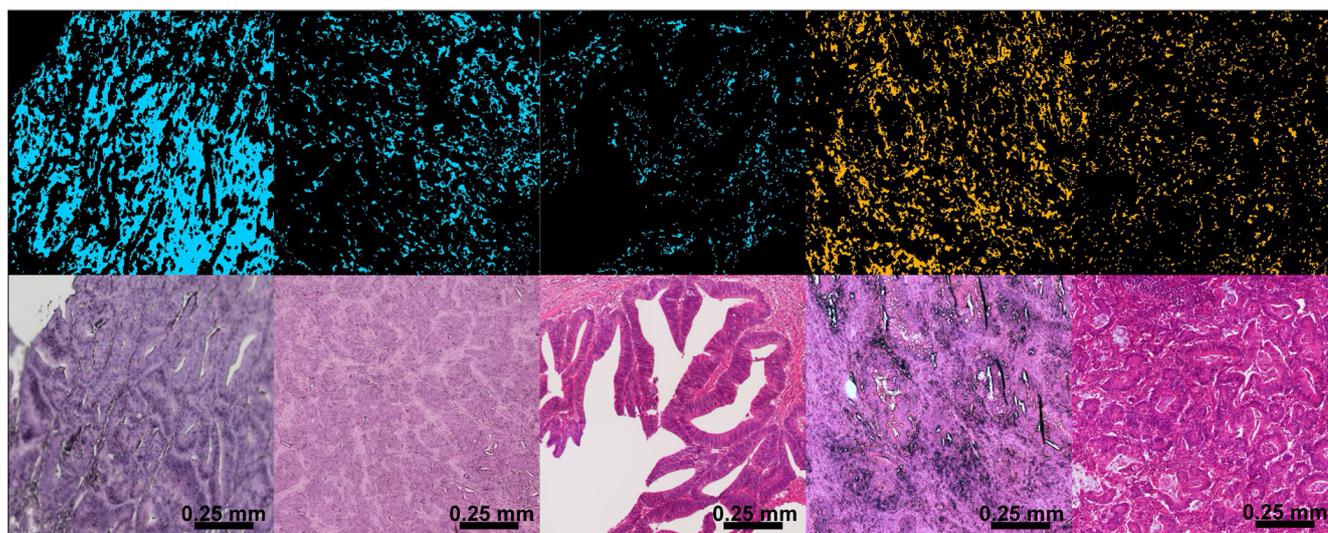
The researchers are now working on a new catheter prototype that would more fully integrate the mapping processes. They also plan to demonstrate the method in large animals to test how well it works with heart muscles moving and blood circulating through the heart.

IR imaging to classify colorectal cancer

A research team from the Prodi Centre for Protein Diagnostics at Ruhr-Universität Bochum (RUB) has used infrared (IR) microscopes based on quantum cascade lasers to classify tissue samples of colorectal cancer from routine clinical operations in a marker-free and automated way. Artificial intelligence enabled the researchers to differentiate between different tumour types with great accuracy within approximately 30 minutes. Based on the classification, doctors can predict which course the disease will take and, consequently, choose the appropriate therapy. The team published their work in *Scientific Reports* (doi.org/d5w5).



A new ablation catheter incorporating NIR spectroscopy mapping can successfully distinguish various tissue types in hearts. This distinction is critical when using radiofrequency ablation to treat heart rhythm problems. Credit: Christine P. Hendon, Columbia University. Photographer: John Abbott



Infrared (IR) imaging for microsatellite stable (MSS; light blue) and high microsatellite instability (MSI-H) versus hematoxylin and eosin (H&E) staining of the same tissue section. Shown are example images for three MSS colorectal cancers (CRCs; light blue) and two MSI-H CRCs (orange). The IR images present cancer spectra only; therefore, the background with other tissue types is black. Reproduced from *Scientific Reports* (doi.org/d5w5), CC-BY.

A distinction is made between microsatellite stable (MSS) and microsatellite unstable (MSI) tumours in colon and other cancers. Microsatellites are usually function-less, short DNA sequences that are frequently repeated. Patients with MSI tumours have a significantly higher survival rate. This is due to a mutation rate of cancer cells that is about 1000 times higher, which makes their growth less successful. Moreover, innovative immunotherapy is more successful in patients with MSI tumours. "It is therefore important for the prognosis and the therapy decision to know what kind of tumour we are dealing with", says Professor Anke Reinacher-Schick, Head of the Department of Haematology and Oncology at the RUB clinic St Josef Hospital. To date, differential diagnosis has been carried out by immunohistochemical staining of tissue samples with subsequent complex genetic analysis.

The potential of IR imaging as a diagnostic tool for the classification of tissue (so-called label-free digital pathology) has already been demonstrated in earlier studies by the group headed by Professor Klaus Gerwert from the RUB Department of Biophysics. The method recognises cancer tissue without prior staining or other marking and, consequently, also works automatically with the aid of artificial intelligence. Unlike

the conventional differential diagnosis of microsatellite status, which takes about one day, the new method requires only about half an hour.

The protein research team has significantly improved the method by optimising it for the detection of a molecular change in the tissue. Previously, the tissue could be only morphologically visualised. "This is a big step that shows that IR imaging can become a promising method in future diagnostics and therapy prediction", says Klaus Gerwert.

In collaboration with the Institute of Pathology at RUB headed by Professor Andrea Tannapfel and the Department of Haematology and Oncology at the RUB St Josef Hospital, the research team conducted a feasibility study with 100 patients. It showed a sensitivity of 100% and a specificity of 93%: all MSI tumours were correctly classified with the new method, only a few samples were falsely identified as MSI tumours. An expanded clinical trial is now starting, which will be carried out on samples from the Colopredict Plus 2.0 registry study. Initiated by Andrea Tannapfel and Anke Reinacher-Schick, the registry study allows the validation of the results from the published work. "The methodology is also of great interest to us, because very little sample material is used, which can be a decisive advantage in today's

diagnostics with an increasing number of applicable techniques", explains Andrea Tannapfel.

In future, the method is to be introduced into the clinical workflow to assess its potential for precision oncology. "Following an increasingly targeted therapy of oncological diseases, it is very important to provide rapid and precise diagnostics", concludes Anke Reinacher-Schick.

Synchrotron IR photothermal spectroscopy at the nanoscale

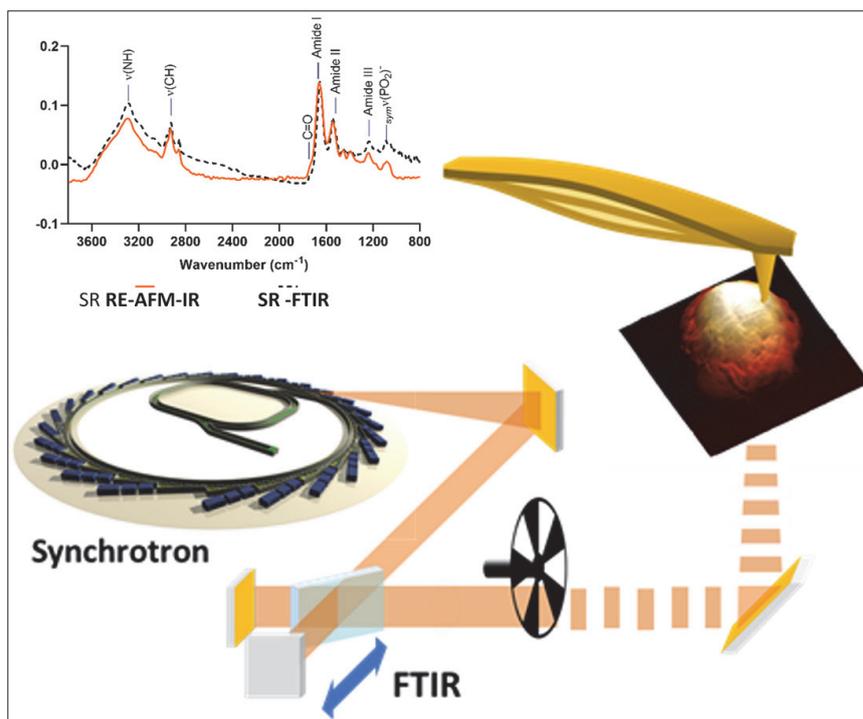
Synchrotron infrared (IR) nanospectroscopy has been used for the first time to measure biomolecular changes induced by a drug (amiodarone) within human cells (macrophages) and localised at 100 nm scale, i.e. two orders of magnitude smaller than the IR wavelength used as probe. This was achieved at the Multimode InfraRed Imaging and Micro Spectroscopy (MIRIAM) beamline (B22) at Diamond Light Source, the UK's national synchrotron facility. This was achieved by an international team made up of researchers from the School of Cancer and Pharmaceutical Science at Kings College London, the Department of Pharmaceutical Technology and Bio-pharmacy at University of Vienna,

and the scientists of the MIRIAM B22 beamline at Diamond.

Their recent paper, now published in *Analytical Chemistry* (doi.org/d4mc), is titled "Synchrotron photothermal IR Nanospectroscopy of macrophages drug-induced phospholipidosis". It outlines the application of Resonance Enhanced infrared Atomic Force Microscopy (RE AFM IR) by synchrotron radiation, to interrogate biological matter at the subcellular level, in this case a cellular model of drug-induced phospholipidosis (DIPL). Instead of the traditional method to evaluate DIPL—visual confirmation by electron microscopy of the lipid bodies or the use of fluorescence labelling technique—they used IR broadband illumination from the Diamond synchrotron together with AFM detection to achieve both molecular specificity and enhanced spatial resolution needed to localise metabolic changes within the cell.

Dr Andrew Chan of King's College London as principal investigator explains: "The model study based on J774A-1 macrophages exposed/not exposed to amiodarone has clearly demonstrated that RE AFM IR with synchrotron radiation is capable of extracting local molecular information from small organelles within a single cell in a label-free manner. This is remarkable because the determination of lipid content in vacuoles is crucial in the study of DIPL. This will have high impacts on the development of inhaled medicines whereby DIPL is one of the key indications of adverse response from the body to foreign particles."

AFM topography maps showed amiodarone-treated cells had enlarged cytoplasm, and thin regions of collapsed vesicles. The IR maps of the whole cell were analysed by exploiting the IR overall signal versus AFM-derived cell thickness, also on lateral resolution around 100 nm. Vibrational band assignment of the nanospectra was possible too: all characteristic peaks for lipids, proteins and DNA/RNA were identified. Additionally, both band ratio and unsupervised chemometric analysis of synchrotron IR nanospectra from the nuclear and perinuclear regions of the cells showed that the cytoplasm of amiodarone-treated cells had



Schematic of synchrotron photothermal IR nanospectroscopy on a mammalian cell.

significantly elevated band intensities in the regions corresponding to phosphate and carbonyl groups, indicating detection of phospholipid-rich inclusion bodies typical for cells with DIPL.

Principle Beamline Scientist at the MIRIAM beamline at Diamond and one of the work's authors, Dr Gianfelice Cinque comments: "Our experiment is—to my knowledge—a world first by Synchrotron photothermal IR Nano spectroscopy in life sciences, and proved that photothermal IR Nano spectroscopy can successfully scan across mammalian cells and reveal the inner molecular fingerprint via the full IR spectrum, thanks to synchrotron IR broadband coverage. This achievement has been the conclusion of a long experimental effort by the IR beamline B22 team of Diamond—especially the expert work by Dr Mark Frogley and Dr Ioannis Lekkas."

Furthermore, Dr Cinque noted that the cell model system and the drug treatment exemplified the method capability by spatially localising morphology and biochemistry at subcellular scale. What was remarkable was that the quality of the nanospectra achieved was such that typical vibrational features observed by

IR microscopy on biological cells were clearly captured, but for the first time at the nanoscale, providing subcellular biochemical information in a label-free manner.

He went on to explain that the MIRIAM beamline (B22) experience in Synchrotron IR nanospectroscopy—i.e. Synchrotron RE-AFM-IR spectroscopy—offers unique chemical and morphological insight at sub-wavelength or 100 nm resolution across a variety of real-life research especially in soft matter, such as microplastic effect in living tissue, antimicrobial surface phenomena, microfossil and biogeology at submicron scale, organic microelectronics analysis, micro-composite materials and mesostructures.

More research capacity will be offered soon at the MIRIAM beamline B22, as a new nanoIR end station is expected from mid-2021. Beyond the current expertise in synchrotron IR photothermal nanospectroscopy, the upgrade will allow new methods (e.g. AFM IR in tapping mode), and crucially complement them with IR scattering scanning optical microscopy (s-SNOM), pushing the spatial resolution even further at the 10s of nanometres scale.

The UV/Vis⁺ photochemistry database

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The *science-softCon UV/Vis⁺ Photochemistry Database* (www.photochemistry.org) is a large and comprehensive collection of extended ultraviolet, vacuum ultraviolet, ultraviolet, visible and near infrared spectral data and other photochemical information assembled from published peer-reviewed papers. The database contains photochemical data including absorption, fluorescence, photoelectron, and circular and linear dichroism spectra, as well as quantum yields and photolysis-related data that are critically needed in many scientific disciplines. This article gives an outline regarding the structure and content of the *science-softCon UV/Vis⁺ Photochemistry Database*. The accurate and reliable molecular level information provided in this database are fundamental in nature and help in proceeding further to understand photon-, electron- and ion-induced chemistry of molecules of interest, not only in spectroscopy, astrochemistry, astrophysics, Earth and planetary sciences, environmental chemistry, plasma physics, combustion chemistry, but also in applied fields such as analytical chemistry, medical diagnostics, pharmaceutical sciences, biochemistry, agriculture and catalysis.

Introduction

Photochemical data and information such as absorption spectra, fluorescence spectra, photoelectron spectra, circular and linear dichroism spectra, quantum yields etc. are important parameters needed in many scientific disciplines. Back in 1999, there was deemed to be a need for publicly accessible on-line databases containing such data and information in digital format (machine-readable). A first *UV/Vis Spectra of Atmospheric Constituents* CD-ROM¹ was published which contained, at that time, the largest collection of ultraviolet/visible (UV/vis) spectral data available free-of-charge. Based on this CD and the motivation to provide spectral data and information in digital format to the scientific community via the World Wide Web, the *UV/Vis Spectra Database* went on-line in August 2000 as a non-profit project.

In the beginning, the on-line database contained about 1200 spectra/datasheets for 120 substances and the compiled data extended beyond atmospheric research to allow for interdisciplinary application. To enable platform independent usability, both the spectral data as well as the datasheets (metadata such as publication, authors, source, wavelength range, temperature, pressure,

phase etc.) are available as plain ASCII text. To guarantee the high quality of the fast growing *UV/Vis⁺ Spectra Database*, an international "Scientific Advisory Group" (SAG) was established in 2004, and the database was operated in accordance with the "open access" definitions and regulations of the CSPR Assessment Panel on Scientific Data and Information (International Council for Science, 2004, ICSU Report of the Committee on Scientific Planning and Review Assessment Panel on Data and Information).²

Since 2004, in addition to publishing the on-line database, every two years a mirror of the on-line database has been published on CD-ROM. The latest edition in the *science-softCon UV/Vis⁺ Spectra Database* series was published in 2019.³ The on-line database currently (as of July 2020) contains about 14,200 spectra/datasheets as well as 5300 graphical representations for about 3000 substances, and is sub-divided into 28 substance groups (e.g. hydrocarbons, pharmaceuticals, pesticides, polycyclic aromatic hydrocarbons etc.). The database is updated weekly. In addition to the inclusion of new data, a main focus of the database is the preservation of data from older publications.

A more detailed description of the database and its applications has been published recently.⁴

As mentioned by the CSPR Assessment Panel on Scientific Data and Information, database maintenance and management are costly.² Collection of data, preparation of metadata and provision of professional data management expertise and institutional support for data dissemination and permanent archiving will add to the overall expense of specific research projects and maintaining the larger research infrastructure.

"Full and open access" to data implies equitable, non-discriminatory access to all data that are of value for science. It does not necessarily equate to "free of cost" at the point of delivery. There are several economic models for providing scientists with access to data for research and education.² The *UV/Vis⁺ Photochemistry Database* allows free and open access to all metadata, and cost-recovery pricing for data (or data licenses) in order to support the full data infrastructure. A choice of charged subscriptions giving full access to the data are available: for example an annual campus-wide licence provides full access to all data and information for less than USD 1 per day (for universities, governmental

organisations, non-profit organisations) and a "One-Time Registration" licence allows perpetual access to all data and information. Both licenses include a copy of the 12th edition of the *UV/Vis+ Spectra Database* CD-ROM.³ In addition, those colleagues who support us in maintaining the database through the provision of new or missing data and information can get personal free-of-charge access to all data and information. More information is available at www.photochemistry.org.

Database structure and content

The database contains spectral information (gas, liquid and solid phase) from the extreme ultraviolet to the near infrared spectral regions (EUV-VUV-UV-Vis-NIR) and related data (e.g. information concerning publications on quantum yield studies or photolysis studies) from published peer-reviewed papers. Besides absorption spectra, which comprise most of the available data, fluorescence spectra, photoelectron spectra, circular and linear dichroism spectra, quantum yields etc. are available. The database is structured into 28 categories, which just provide a rough classification.

The datasheets provide metadata (substance name, formula and CAS number, data source, full reference, including title, authors, journal and DOI when available, spectral range and resolution, temperature, pressure, phase etc.), as well as data in various forms obtained and presented in the literature. This includes, for example, absorption data measured over a specific wavelength/energy range in tabulated form. In many applications (e.g. quantum yield studies or photolysis studies), the absorption cross-section (σ) or the molar extinction coefficient (ϵ) at a specific wavelength (λ) are determined, and these single wavelength data are also included in the database. For many substances temperature dependent data are available.

Most of the available data are from published peer-reviewed papers (>99%), data presented at scientific meetings and conferences are also available (<0.5%), as well as data from PhD theses, reports and unpublished material (<0.5%). As an example of

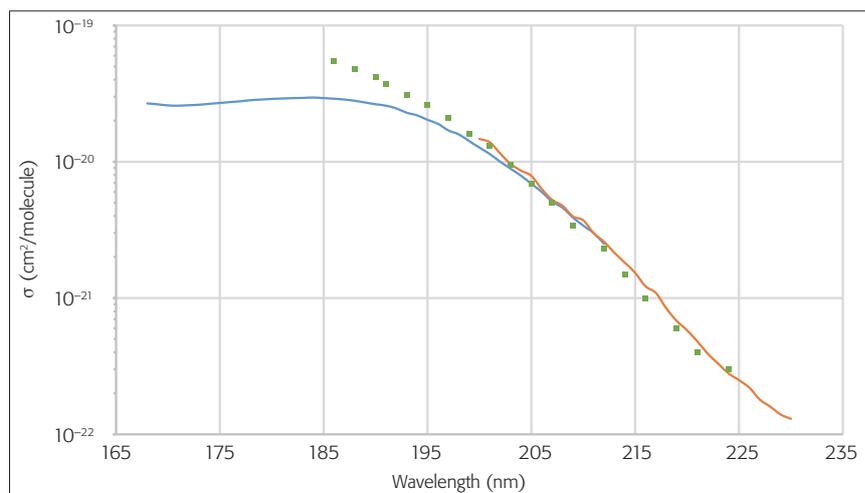


Figure 1. UV absorption spectrum of COF_2 obtained by Noelle⁵ (blue curve), Noelle *et al.*⁶ (red curve) and Molina and Molina⁷ (green squares).

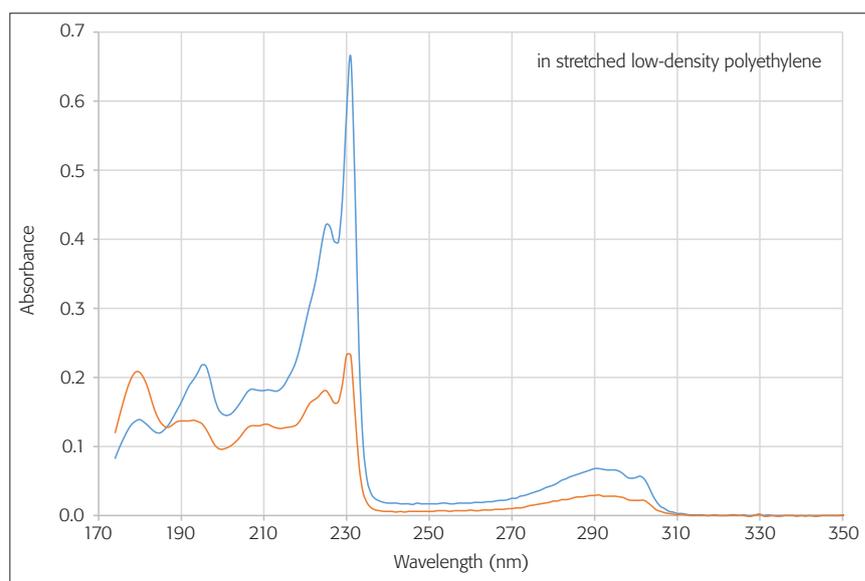


Figure 2. Linear dichroism absorbance spectrum of dibenzo-*p*-dioxin.⁸ The absorbance curves were recorded with the electric vector of the sample beam parallel (blue line) and perpendicular (orange) to the stretching direction of the polyethylene polymer.

the database structure and contents, absorption data of carbonyl fluoride (COF_2) from three different sources are presented in Figure 1. The data sets are as provided by the authors or listed in the relevant publications. To enable platform independent usability, all data are provided as plain ASCII text.

More recently, almost 3000 graphical representations, mostly from older publications have been digitised and added to the database. In addition, we

have converted more than 100 datasets from "floppy discs" and hence prevented these data from being lost as technology has evolved.

Since 2019 the database has been extended to include circular and linear dichroism spectral data (Figure 2). The absorbance curves were recorded with the electric vector of the sample beam parallel and perpendicular to the stretching direction of the polyethylene polymer.

Outlook

The *science-softCon UV/Vis⁺ Photochemistry Database* is continually evolving and growing. As of July 2020, it contains about 14,200 spectra/datasheets as well as 5300 graphical representations for about 3000 substances and is sub-divided into 28 substance groups (e.g. hydrocarbons, pharmaceuticals, pesticides, polycyclic aromatic hydrocarbons etc.) This is a tremendous effort and requires a lot of manpower, not to mention technical infrastructure. We hope that the database proves useful to the scientific community and will facilitate their day-to-day work.

Since the support by the scientific community is crucial for such a photochemistry database, we would like to encourage all colleagues to assist us in maintaining the database and join our initiative "Photochemical Data and Information Sharing Platform—Share Photochemical Data & Information, Find Answers".

This initiative should develop the photochemical database towards a photochemical data sharing platform. The advantage of such a photochemical data sharing platform is that the more scientists provide their data for inclusion in the database, the better is the chance for all users to find specific photochemical data within the database. In addition, the platform becomes increasingly beneficial for use across multiple disciplines.

Database examples (datasheet, data, graph) are available at www.photochemistry.org.

Since the "UV/Vis⁺ Photochemistry Database" is operated as a non-profit "open access" database, any support from both sides, academic and commercial, would be highly appreciated.

Acknowledgements

The authors would like to thank the members of the SAG and the colleagues who support us in maintaining the database.

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Web content

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- Application: Bioorthogonal photocatalytic reactions of flavins converting Pt-VI substrates into Pt-II anticancer drugs, <https://bit.ly/3kvh931>
- Application: Optimisation of SERS for glucose sensing, <https://bit.ly/2XlgO35>
- Application: Understanding Electron Multiplying Gain, <https://bit.ly/2DC162Q>



When to automate spectroscopic data processing

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I read with interest a recent article in *Chemical Science* originating out of Jonathan Goodman's group at the University of Cambridge. Jonathan is another long-standing IUPAC campaigner for scientific data standardisation and his group has been working on an improved solution to tricky nuclear magnetic resonance (NMR) spectra interpretation.¹ Their approach exploits modern higher processing speeds to enhance their fully automatic molecular structure elucidation software. Their DP4-AI uses the quantum chemical Gauge-Independent Atomic Orbital Density Functional Theory (GIAO-DFT) method calculations starting from chemical structures with undefined stereochemistry. ¹H and ¹³C-NMR peak picking algorithms handle noisy spectra to predict relative stereochemistry. A statistical value is generated for the likelihood that each of the candidate molecules is correct based on the analysed spectra with almost no need for human intervention. This makes it an ideal tool to rapidly solve difficult problems like natural product library validation.

Clearly, there is still strong demand for improved NMR data interpretation and prediction software. I wondered how much such systems were being used on a day-to-day basis in industry, so talked to Gary Sharman, who has enjoyed a 20-year career in analytical science in the pharmaceutical industry and Marcel Simons, a very experienced NMR expert and one of my old colleagues at AkzoNobel/Nouryon.

Why do automation?

Many years ago, I heard a comment that has stuck in my mind and still raises a smile when I have occasion to remember it. One of the pharmaceutical industry customers of Creon-LabControl AG were testing an innovative combined ultraviolet/visible (UV/vis) and mass spectrometry (MS) automated approach for natural product library screening against "known chemistry" to select extracts for further work. After testing for a while, the customer explained to the software developers the reason behind his excitement. Completely ignoring the technological advances and clever programming that had gone into the system being tested, the customer simply pointed out that the automated spectroscopic data processing system effectively eliminated the boring repetitive work. Extracts that were of no interest (known chemistries) were automatically removed allowing him and his team to very rapidly focus on the extracts of interest that were potential new active molecules. "I can finally spend most of my time doing the expert job my company is actually paying me for".

So much for the thoughts that people increasing automation might be responsible for taking jobs away from spectroscopists! Gary Sharman highlighted three areas that can be seen as major drivers for better automation:

- Lost opportunities: problems that we would not even dare to start without automation.
- Free up time for more interesting work. We all became spectroscopists

for the tricky, interesting problems, not to churn a handle on routine analysis and be bookkeepers. Let automation take care of the drudgery so you can focus on the fun problems. (Like the UV/vis-MS example above.)

- Less silly mistakes/book-keeping errors. We all like to think we are accurate and precise, but the fact is humans make lots of silly mistakes, particularly in collating data. Computers do not make these kinds of mistakes.

Have realistic expectations

The danger of having so much automation at our fingertips is that we might be setting ourselves up for some spectacular falls when the automation encounters problems it simply cannot master. You often see this in much simpler systems such as gas chromatography (GC)/MS database search results of electron ionisation spectra. We have discussed many innovative solutions in this column in the past, but time and again I see reports where the first database hit is cited as being the compound identified—even if the chemistry of the proposed molecule can have nothing to do with what is actually being worked on. If the scientist/student had taken the time to look further down the hit list they would have found a substance that made much more sense in terms of the experiments being undertaken.

So, as Gary put it... If you want perfectly assigned NMR spectra every time—give up now! A much better aim is

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to really ask yourself what level of errors you are prepared to tolerate, and how that trades off against effort. For example, consider the quality control of a large library; without automation you may conclude it cannot be done. With automation perhaps we have 5% false positives. It is not perfect, but surely better than having no data on purity.

So, ask yourself what level of errors you are willing to accept. Be realistic. Everyone says “I want 100% accuracy”, but not even an experienced spectroscopist can achieve that. You might make a trivial error like mixing up two samples or simply working on complex chemistries which you are unfamiliar with.

The automation process

Gary described the process in a similar way to Jonathan Goodman’s group and this actually applies for different types of spectroscopy (Figure 1).

Although this might be seen as a rather simple schema, it is good to see how automation will benefit us at the various steps in the process.

- Data preparation and metadata extraction. Not to be overlooked—this may be one of the quick wins. For example, automatically finding and opening connected bits of data, looking up a structure and loading it, saving results—all parts that take time and are tedious bookkeeping, but every process needs them.
- Data processing such as peak picking and categorisation. This can be a very crucial part of the process. Many automated structure validation “mistakes” that are just down to poor peak picking of the data.
- Prediction—unless we are looking up a known thing in a database, we typically must predict the expected result to allow comparison. This could be quite simple (what is the expected

ion for MS) or complex (a prediction of NMR by *ab initio* methods).

- Matching predicted to experimental. For some applications, this may be trivial: is the biggest peak in the mass spectrum the same as the m/z I expect. For proton NMR, with the complexities of coupling, overlap and higher order effects, it is exceedingly difficult.
- Scoring and output—we need to return a useful value that can be used to set actions. We might also want to return “quality factors” that indicate if the result is to be believed or if manual review is a good idea: these two things may well be orthogonal. A fail in the test may not mean the data needs review, and a pass may not mean it is a valid result.

Review by exception strategy

Although you may regard this as an oversimplification, manual analysis is “slow and accurate”. Automation is often seen as “fast but error prone”. By flagging samples for review where there is a reason to believe the automated result may be suspect, we can get the best of both worlds (Figure 2).

We do not work alone!

One of the critical questions which we are always asking is exactly how does some new wonder-software fit into our daily working practices and processes?

- The automation steps are only half the problem—how are you going to link your process to other processes in your organisation? This can make or break the automation. Workflow tools like the Swiss KNIME, the Konstanz Information Miner (a free and open-source data analytics, reporting, and integration platform)² or Biovia’s Pipeline Pilot³ can

be valuable here. Also, having information exposed through APIs or web services makes integration easier.

- Constraints. You may have to work with legacy systems, other software with particular requirements or unhelpful interfaces to other data. This can be a major part of the problem that impacts design and implementation.
- The soft part—no one likes to be told by a computer they made a mistake. To get acceptance for a system, it may need thought about how people are informed of failures. For example, an e-mail saying you did something wrong with your boss copied in is probably a bad move. Flagging an error to an expert who reviews it and has a quiet word might be more accepted.
- New problems. Real world data is not perfect. Low signal-to-noise, poorly prepared samples and other components like residual solvents may lead to failures that a person would deal with as part of accepted normal practice.
- Edge cases. Software is built and validated on limited sets of test data. You can guarantee that over time edge cases will be detected that it does not handle well. Hopefully over time, more and more edge cases are dealt with and they become less and less frequent.

So, sticking with the world’s COVID-19 theme, an Automated Structural Verification (ASV) software package like Mestrelab’s “Verify” module can do an excellent job of assigning a molecule, such as a pharmaceutical active ingredient in a clean sample. Expecting a perfect assignment every time may be setting our sights too high. Imperfections do not stop a system being useful.

Enabling non-spectroscopist colleagues

Marcel Simons and colleagues have been working hard to help support colleagues from other disciplines in a speciality chemicals research and manufacturing area in a way that embodies many of the advantages listed above,

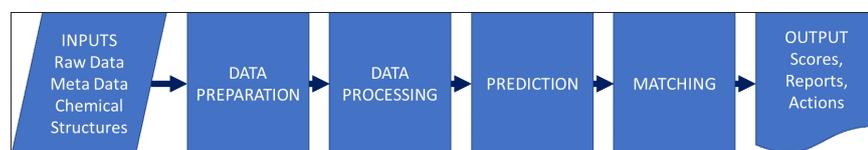


Figure 1. Parts of an automation process; not all processes have all parts. As well as the steps themselves, the inputs and outputs and their interfaces to other systems may be key to success.

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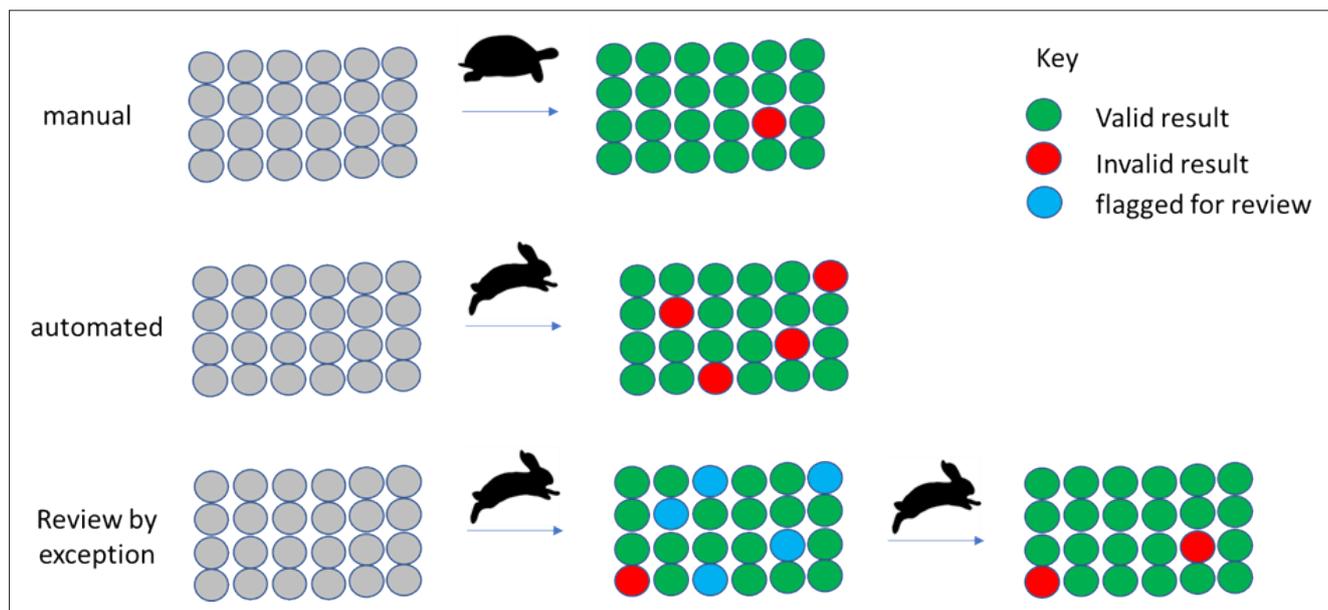


Figure 2. Automation supporting and reducing manual data analysis by focussing on the suspect results.

but in a quite different environment. Their challenges are far more to do with quantitative analysis by NMR rather than purely structure elucidation. The Expert Capability Group's open shop NMR has to cope with a very high workload of business- and time-critical samples—often being generated out of normal laboratory hours. They started configuring automated spectroscopic data analysis back in 2006. With instrument vendor support, they have developed and deployed over 30 automated methods that do tasks such as data processing for manufacturing plant support. These methods go well beyond the out-of-the-box tools, and are designed to work using simple sampling strategies on all liquid samples with usable signals even without the use of deuterated solvents.

The automation results are basically processed spectra and a dedicated Excel file with the desired integrals and calculated molar ratios and/or calculated and normalised weight percentages. Depending on the targeted recipient of the automated processing and the demands of the specific business customers, conditional formatting is applied highlighting the results in green if the processing has delivered the expected result and red if the data is not what was expected and

additional actions are potentially required (Figure 4).

Conclusions

So, it looks like there is a good clear case for continuing to develop faster and less error prone automated spectroscopic data processing. Jonathan's group have made their new software available under the Open Source MIT license, so if you feel like trying it out while you sit at home worrying about a second COVID-19 wave it can be downloaded from GitHub.⁴

Gary was one of the authors on a recent paper that pulled together many

Compound	m/m%	mol%	A/C
A	76.2	61.3	3.92
B	19.4	35.2	
C	4.4	3.4	

Compound	m/m%	mol%	A/C
A	66.0	49.1	2.38
B	27.7	46.4	
C	6.3	4.5	

Figure 4. At the end of a complex automated NMR data processing method, the customers question may boil down to "is the ratio of the concentration of two compounds within specific target boundaries to the quality criteria". In this figure, the results show a pass and the lower a fail.

of the topics discussed here.⁵ The paper discusses an automated system to verify new compound registrations. At its core was Mestrelab's Verify engine which automatically verified registered structures against their NMR and liquid chromatography-MS data. This was wrapped in a web service to make access by external processes simple. Bookkeeping tasks, scheduling and interfaces to other systems were taken care of by a KNIME server, and a streamlined review process was put in place to ensure there was a human face put on dealing with any problem samples.

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Chemical analysis of contaminated soil for sound environmental site assessment. Part 1: the critical role of proper sampling

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Proper sampling of particulate matter for instrumental analysis is a common task in many applied scientific, technology and engineering fields. It is a crucial task for ensuring that measurements made on a given set of samples are representative estimate of the parameters of interest in the original sampling target. Unfortunately, sampling particulate matter is in many fields performed without a scientific basis, mostly because its critical role is ignored, or at best, misunderstood, and because of an unawareness of, sometimes a disregard for, the Theory of Sampling. This two-part column illustrates this important point using experience in the field of geo-environmental engineering.

Environmental site assessment guidelines require representative sampling, but do not define how: a recipe for decision-making disaster

A noteworthy example of how sampling is performed without a proper scientific basis is the sampling involved in environmental site assessment of *contaminated soil*. In this context, soil samples are analysed for their content of contaminants (chemical, physical). For chemical contaminants, analytical protocols generally require a few grams of soil for analysis only, and specify that this small quantity **must** be representative of the field parcel from which it is derived. This implies that a few grams of soil **must** represent a volume up to several hundred cubic metres of particulate matter in the field. This implies a mass reduction of nothing less than six to nine orders of magnitude, while ensuring that at each stage of the mass reduction process the resulting sub-sampled quantity of matter still

represents the entire original soil parcel. With the current state-of-affairs in this field (guidelines, standards, tradition, ignorance) this is a well-nigh impossible task. We find it incumbent upon us to sound a serious alarm within the field of geo-environmental engineering—but the examples and lessons described below have a much wider impact in many applications fields with *similar* heterogeneity issues.

The representativeness of an analytical measurement, i.e. the degree to which it represents the real contaminant content in the soil, compositionally as well as spatially, is directly related to the representativeness of the *sampling process*. This means the degree to which the proportion of each type of constitutive element of the soil, particles and contaminant(s) is preserved during the “from-field-to-analysis” sampling/sub-sampling process.

However, in the vast majority of current cases, the degree of

representativeness is not assessed, far less even mentioned. In most guidelines for sampling of contaminated soils, representativeness is a vague concept, mostly owing to some form of wishful thinking. Without a formal **definition** of representativeness and guidelines on **how to obtain** a desired degree of sampling representativeness (called “fit-for-purpose representativeness”), sampling is performed more or less intuitively, haphazardly or based on subjective judgement. This approach is called grab sampling in the Theory of Sampling (TOS). It most commonly involves taking the desired mass of soil (“not-too-much”) from some accessible part of the soil in *one increment*. In today’s practice in the field, this would result in a grab sample of a few hundred grams which is sent to the laboratory, where a grab sub-sample of a few grams is then taken for analysis.

Below are two realistic, real-world examples of how this approach to

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sampling can produce extremely poor results.^a

Assessment of zinc contamination

The first example is typical of a common situation in the practice of environmental assessment. A field sample from a site contaminated with zinc (Zn) was sent to an analytical laboratory by a geo-environmental consultant charged with the environmental assessment study. Field and laboratory sampling were performed by grab sampling. As per common practice, the laboratory was charged with providing a single analytical result from the material in the container delivered. This measurement resulted in a Zn concentration of 1900 mg kg^{-1} , thus indicating a contamination well above the regulation threshold for the current usage of the site (see further below). This result would lead to the demand that the soil from the parcel must be removed.

However, several *in situ* semi-quantitative measurements were also made by the consultant on the soil parcel with the use of a portable X-ray fluorescence spectrometer, and these had indicated a possibly smaller concentration.

Therefore, the consultant asked the laboratory for “a second measurement” based on the same sample container. This time the results came in at 79 mg kg^{-1} . Such a major discrepancy “naturally” prompted a third measurement, which, however, failed to detect any Zn in the soil! At the end of a very confusing day, a total of seven individual measurements were made based on the same 300g soil sample as shown in Table 1.

Que faire?

As a way of trying to shift the burden of explaining these wildly varying results to the consultant, the laboratory concluded that the sample received was not homogeneous. Although this conclusion is



Typical test pits in geo-environmental engineering site soil characterisation. One attribute rules the day: “significant heterogeneity”. It is obvious that any single field sample (a grab sample in the TOS parlance) will not be able to represent the entire site. For this job, diligent compliance with the TOS’ principle of *composite sampling* is necessary (see part 2).

correct, such a conclusion is profoundly naive as **all** soils are heterogeneous, it is only a matter of to which degree (TOS).

This self-evident truth was exacerbated in the present case by severely “incorrect” sub-sampling in the laboratory (grab sampling from the *same* field sample container). So, whatever heterogeneity was revealed only pertained

to the scale of the volume of the field container. Whether this is the same heterogeneity characterising the significantly larger site volume under investigation is still a completely open question: how well does the field container represent the entire site?

The applicable regulatory thresholds were 140 mg kg^{-1} (I), 500 mg kg^{-1}

Table 1. “Autopsy” of a single 300g field soil sample, and the resulting soil remediation status (categorisation).

Measurement	Concentration (mg kg^{-1})	Categorisation based on measurement
1	1900	>III
2	79	<I
3	<4	<I
4	<4	<I
5	<4	<I
6	700	II–III
7	25	<I

I, II and III represent regulatory thresholds of 140 , 500 and 1500 mg kg^{-1} , respectively.

^aFor the record: the examples and procedures discussed here pertain to significantly heterogeneous materials that **cannot** be subject to mixing before sampling. If a significantly heterogeneous lot to be sampled *happens* to be so small that it is economically feasible to mix it thoroughly in its entirety, the rules of the game have been altered because mixing leads to a significantly reduced distributional heterogeneity. However, the resultant lot is still compositionally heterogeneous and still needs to be treated as such. Such cases are exceedingly rare, and consequently of overwhelmingly little interest within geo-environmental engineering.

Introduction to the Theory and Practice of Sampling

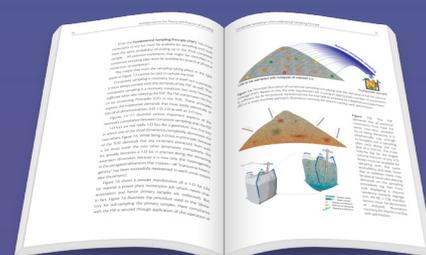
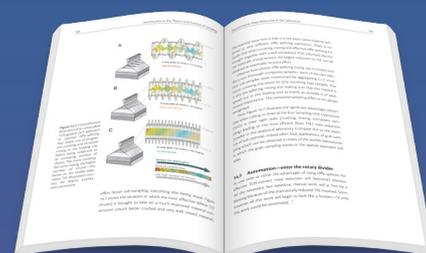
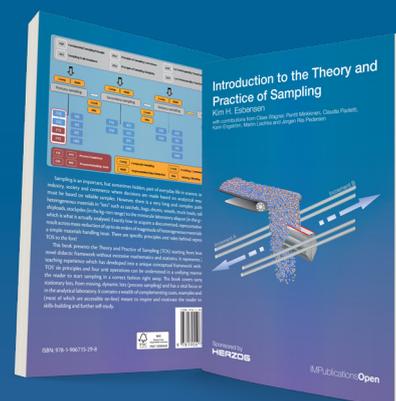
Kim H. Esbensen

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– μ g 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.

NEW
BOOK



“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”

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(II) and 1500 mg kg^{-1} (III), each value representing the maximum allowed Zn concentration in soil for specific usages of the site, or specific means of disposal of the excavated soil. Table 1 also shows the categorisation of the soil with respect to these thresholds based on each of the seven “replicated” measurements.

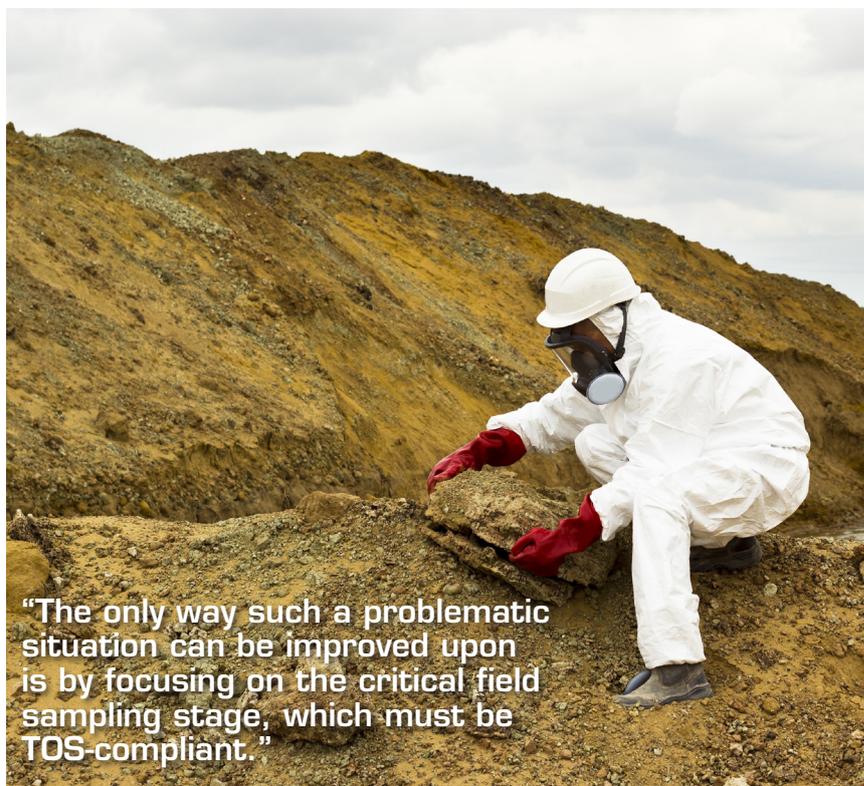
It comes as no surprise that the consultant was now confronted with the confounding problem of correctly *categorising* the soil parcel represented by one field sample, but seven analytical results. If the categorisation decision had been made based on a single measurement, as is the usual practice, a highly significant error would have been introduced. This would have transferred unwarranted significant uncertainty to the site remediation process. The key issue is, of course, that under “normal practices” this would not even have been known to any of the stakeholders involved.

It would be hazardous to fit a statistical distribution to such a small dataset in which 43% of the data are left censored. However, it is possible to roughly *estimate* the categorisation probabilities based on proportions as shown in Table 2.

If the consultant had used the first measurement, as in current practice, he would have categorised the soil as larger than criterion III and, therefore, in need of disposal off site (A). But the probability

Table 2. Estimates of categorisation probabilities (categories A, B, C are identical to categories I, II, III in Table 1).

Category	Probability
$x < A$	0.714
$A < x < B$	0
$B < x < C$	0.143
$x > C$	0.143



“The only way such a problematic situation can be improved upon is by focusing on the critical field sampling stage, which must be TOS-compliant.”

that this decision would have been correct is only 14.3% (Table 2).

The consultant was, therefore, well advised to ask the laboratory for supplemental measurements. While these vary widely, a Kaplan–Meier (KM) estimate of the mean Zn concentration, 388 mg kg^{-1} ,^b indicates that the soil could be categorised as being lower than criterion III, and thus kept on the site. This decision would have had an 85.7% probability of being correct. The problem of categorising the soil becomes more acute when the soil must be excavated and disposed of off site, since the disposal cost is related to the contamination level category. In the present case, based on the singular initial measurement, the soil would have been categorised as larger than criterion III, and disposed of at a larger

cost, most probably incurring unnecessary expenditures from the site owner. However, based on the KM mean of 388 mg kg^{-1} , the soil would have been categorised as between criteria I and II, and thus disposed of at a much smaller cost or even reused as fill material in some jurisdictions. This example illustrates well how much uncertainty can be introduced in the decision-making process if based on a single 300 g field soil sample.

It can come as no surprise then that the documented uncertainty points to the highly likely situation that the target lot from which this single field sample originated must be significantly heterogeneous itself. The key issue is: is the single field sample representative of this target lot? To answer that, attention must be directed elsewhere: **how** was

^bNote that calculating the mean Zn concentration by arbitrarily substituting the censored concentration measurements, i.e. $< 4 \text{ mg kg}^{-1}$, by 0 or 4, we obtain a mean Zn concentration ranging from 383 mg kg^{-1} to 388 mg kg^{-1} . While these estimates of the mean are close to the KM estimate in this case, arbitrary substitution in environmental datasets can lead to unreliable and biased estimates of descriptive parameters). Dennis Helsel (doi.org/fdmnj8) comments on arbitrary substitution: “There is an incredibly strong pull for doing something simple and cheap”. This statement can just as aptly also be applied to grab sampling at all stages from field to aliquot.

SAMPLING COLUMN

Testimony

Understanding what sampling variation is, and how it is estimated, has been a “light-bulb” moment for our analysts after having been introduced to the Theory of Sampling (TOS) principles. So often we have had a situation where analytical work and results can be verified, but our customer still insists it doesn’t meet expectations. Short of driving the poor analyst crazy with re-work tasks, which usually only produces the same “incorrect result”, I now have an avenue of action that allows us to guide the customer and analysts to the path on how to focus on only taking representative samples. This is decidedly more welcome than always having to hear: “Take the sample back to the lab—repeat the analysis”.

Much time is spent determining the combined total uncertainty for specific analytical methods under validation, however, very little attention is given to the preceding sampling errors and the challenges heterogeneity poses to this issue. I now know that sampling errors dominate over their analytical cousins. Also, using variographic characterisation as a quality control tool for process and measurement system monitoring is a very powerful technique that could help process controllers explain the sources of real process variations that occur on their product lines instead of simply following through by blaming the analytical lab. I found that the new international standard DS 3077 (2013) and in particular its use of illustrations and industrial examples captured the true complexity of the principal types of sampling errors and helped to conceptualise the TOS principles in a strikingly visual way, making it easier for a typical chemical analyst to relate to the scenarios involved before analysis. After all, we have to isolate the absolutely smallest aliquot for analysis—as demanded by highly sophisticated analytical instrumentation. It is, therefore, highly surprising that the one area of greatest error affecting analysts’ results is the same topic largely ignored in Analytical Chemistry/Science Training programmes, again the sampling errors. This gives rise to “brilliant” analytical results, i.e. extremely precise results, but for non-representative samples for which accuracy with respect to the lot is not accounted for. In fact the accuracy of the analytical results with reference to the original lot is completely without control—and one cannot even estimate the magnitude of the sampling bias incurred (because it is inconstant, as is another insight provided by TOS). This makes for a very unsure analytical laboratory. After this course I wonder how many questionable results have been released by laboratories all over the world over many, many decades—and the revelations brought about by TOS are still not known!

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the primary sample (the field container) sampled in the field? Were the principles and rules in the TOS complied with, or not?

The situation depicted is common and typical, but it is not acceptable. The only way such a problematic situation can be improved upon is by invoking a stronger

focus on the characteristics of the full sampling process, notably the primary field sampling stage.

This case is also “representative” of the ill-informed practice of pouring more money into the analysis stage, i.e. making a larger number of measurements from each primary sample.

Instead, more care should be taken in reducing variability at the primary sampling stage. It should not be difficult to understand that the debilitating heterogeneity revealed in Table 1 is only a reflection of the state-of-affairs in the singular field sample upon arrival at the analytical laboratory. No manner of repeated analysis based on this sample alone, can produce any information as to the real-world heterogeneity of the **entire** soil parcel, which **must** be larger but to an unknowable degree. The obvious solution is an appropriate deployment of *composite sampling* covering the entire 3-D parcel site.

Preliminary conclusion, Part 1

The first part of the *full* sampling-and-analysis process occur in the field and is often performed by the consultant’s field technician. This gap in the “chain of custody” of the sampling process between the consultant and the laboratory is particularly problematic, especially as much as the current incorrect sampling practices are left without a clear responsibility. No one takes full responsibility for the representativeness of the complete sampling process in such circumstances.

Fix your sampling, not your results

In part 2 we will further illustrate how measurement variability can be controlled at the sampling stage with a second real-world example from a recent study conducted at École de technologie supérieure, Montréal, in partnership with the same consultant involved in the first example presented here. In this second study, we compare the uncertainty derived from grab sampling to that derived from a TOS-compliant composite sampling process.

References

A complete list of References will be included in part 2.

NEW PRODUCTS

INFRARED

FT-IR spectrometer-based milk analyser

PerkinElmer has launched the LactoScope™ FT-B instrument, an FT-IR spectrometer for testing and adulterant screening for liquid dairy products such as whey, raw and skim milk, shelf stable milk and cream with under 40% fat content. It has a smaller footprint than PerkinElmer's Lactoscope™ FT-A system, which analyses milk, yogurt, ice cream, concentrates and creams with up to 55% fat. The FT-B produces results in <45s with a typical accuracy level of under 1% CV (relative standard deviation). For large processors, the instrument can serve as a second instrument in milk intake areas to help ease liquid sample throughputs in addition to the Lactoscope™ FT-A system. For small- to mid-sized dairies and milk intake points with high volumes, the LactoScope™ FT-B instrument also provides low cost of ownership.

The LactoScope™ FT-B is integrated with the easy-to-use Results Plus software and provides rapid implementation with minimal training, as well as analysis and reporting that are compatible with the Lactoscope FT-A solution, PerkinElmer's DA 7250™ NIR Analyser and LIMS systems. This makes it easy to



move between different platforms and instruments across liquid and solid dairy testing. Further, the Lactoscope FT-B instrument ties into the PerkinElmer Net Plus™ cloud solution to enable remote configuration, monitoring, calibration and team collaboration.

PerkinElmer

► <http://link.spectroscopyeurope.com/32-066>

Handheld FT-IR and SERS analyser measures of low concentrations of illegal substances

The Thermo Scientific Gemini analyser now comes with LowDoseID, specifically to address the rising trend of low concentrations of illicit substances. The Gemini combines Fourier transform infrared (FT-IR) and surface enhanced raman scattering (SERS) into one device to facilitate comprehensive and confirmatory chemical identification for substances with a concentration between 1% and 10%. The ability to detect small amounts of substances enhances accuracy and efficiency for first responders, particularly when identifying the presence of Fentanyl, heroin and cocaine.

In addition to new functionality through the introduction of LowDoseID, the Gemini has also undergone a software update. The version 1.8 software enhances ease of use with on-screen colour-coded alerts based on analysis results and an expanded factory chemical library, as well as smoother operation.

Thermo Fisher Scientific

► <http://link.spectroscopyeurope.com/32-082>



MASS SPEC

High-end triple quadrupole mass spectrometer

Shimadzu has released the LCMS-8060NX triple quadrupole mass spectrometer, which has improvements in ease-of-use and robustness. The LCMS-8060NX offers high sensitivity and detection speed, and includes internal mechanical improvements to promote sample ionisation. The new IonFocus unit reduces instrument contamination by guiding ions to the interior more efficiently and removing unwanted components. In addition,

more robust components are used in ion-focusing, enabling more reliable analysis. The LCMS-8060NX therefore offers both higher sensitivity analysis and lower downtime.

The system includes several "Analytical Intelligence" functions to maximise analysis throughput and improve the operational efficiency and productivity of the entire workflow, from preparations for analysis through to data processing. For example, the

NEW PRODUCTS



Shimadzu's LCMS-8060NX triple quadrupole mass spectrometer.

LCMS-8060NX includes a new ionisation unit that is capable of high-sensitivity analysis without the need for complicated adjustments and enables more efficient method development, as well as functions to start up or shut down analytical

instruments automatically in combination with a Shimadzu Nexera LC system.

Shimadzu

► <http://link.spectroscopyeurope.com/32-068>

SCIEX OS software

SCIEX has introduced SCIEX OS Software, initially available on the Echo® MS System and the SCIEX Triple Quad™ 7500 LC-MS/MS System. Key features include: Auto-detection of sample failures results in auto-corrective action; Review by exception flags mean less manual checking of results; Results are optimised with powerful integration algorithms; Peak modelling improves peak finding and integration, requiring less user input and therefore reduces time for data review; Automated result checking and outlier removal minimises the time it takes to establish an approved calibration range; Intuitive audit trails ensure full traceability and accountability to achieve data precision, completeness and consistency.

SCIEX

► <http://link.spectroscopyeurope.com/32-079>



SCIEX Triple Quad 7500 LC-MS/MS system

SCIEX has launched the next generation of its flagship nominal mass spectrometer, the SCIEX Triple Quad™ 7500 LC-MS/MS System – QTRAP® Ready, which has the highest level of sensitivity in the SCIEX portfolio. Workflows can be simplified due to a linear dynamic range of up to six orders of magnitude. The new D Jet™ Ion Guide allows users to characterise analytes that were previously undetectable, and the design also captures more ions from the spray plume. The new OptiFlow® Pro Ion Source reduces the need for extensive sample preparation, allowing users to quantify more trace analytes at lower levels, and integrated E Lens™ Technology increases ion sampling. The OptiFlow® Pro Source's modular capability enables interchange between high flow and low flow, and between ESI and APCI, without manual adjustments.

A simple upgrade path enables additional QTRAP functionality scan types to improve confidence. MRM3 workflows can push quantification levels through matrix interferences.

SCIEX

► <http://link.spectroscopyeurope.com/32-078>



NEW PRODUCTS

Waters' MassLynx software now interfaces with Skyline software

For customers with Xevo TQ-S micro and Xevo TQ-XS mass spectrometers running on MassLynx™ Software version 4.2, Waters is introducing a new interface to Skyline software, the freely available, open source software for targeted proteomics. The interface will enable scientists to develop optimised LC-MS/MS multiple reaction monitoring (MRM) methods for the quantification of peptides or protein digests by the surrogate peptide

approach. The MassLynx/Skyline interface is an easy-to-use tool for auto-optimising and fine-tuning high-sensitivity MRM assays on tandem quadrupole mass spectrometers for bioanalytical or targeted proteomics experiments that are part of drug discovery research. The MassLynx/Skyline interface can be downloaded for free from the Waters Marketplace (see link below).

Waters

▶ <http://link.spectroscopyeurope.com/32-073>

DESI XS imaging for Waters high-resolution mass spectrometers

The new desorption electrospray ionisation (DESI) XS source is now available on the Waters SELECT SERIES Cyclic IMS, SYNAPT™ XS and Xevo™ G2-XS QToF mass spectrometers. Waters acquired the rights to DESI™ from Prosolia and the Purdue Research Foundation in 2018. When coupled to a mass spectrometer, DESI XS produces a visual map of the spatial distribution of small molecule drugs, metabolites and lipids within a variety of sample and surface types. With DESI XS, scientists can image tissue samples, screen metabolites in bacterial colonies, identify individuals from fingerprints taken directly from various surfaces and examine cross-sections of natural products like roots and tubers. As a direct ionisation source, DESI XS eliminates the need for sample preparation and chromatography prior to the introduction of the sample into the mass spectrometer.

Waters

▶ <http://link.spectroscopyeurope.com/32-072>



New fragmentation option for Waters Cyclic IMS system

Waters has introduced the electron capture dissociation (ECD) fragmentation technique for its SELECT SERIES™ Cyclic™ IMS system, through a partnership with e-MSion, Inc. ECD is an electron-based fragmentation technology that, combined with ion mobility mass spectrometry and collision induced dissociation (CID) available with the Cyclic IMS System, gives scientists improved sequence coverage for native proteins and surrogate

peptides and additional structural information about larger proteins that might not have been possible before. The Waters™ Cyclic IMS System can perform scalable ion mobility separations and is also capable of IMSⁿ experiments (e.g. selecting an ion by IMS, fragmenting the ion, selecting a particular fragment by IMS and repeating the process) which derives detailed structural information about a single analyte.

Waters

▶ <http://link.spectroscopyeurope.com/32-071>

Pfeiffer Vacuum introduces new gas analysers

Pfeiffer Vacuum has introduced the OmniStar and ThermoStar GSD 350 compact, portable benchtop gas analysers for use at atmospheric pressure. The gas inlet is fitted with a heated capillary for use at up to 350 °C. This prevents vapours from condensing during process gas analysis. Thanks to the two-stage inlet system, an almost segregation-free gas supply is possible. The ThermoStar solution was specially developed for coupling with thermo balances. The inlet system with a quartz capillary and a platinum orifice ensures that even the smallest concentrations can be analysed. The OmniStar was developed for a wide range of applications and uses a stainless steel capillary as well as a valve which can interrupt the sample gas stream. Unlike other analytical methods, the two new devices allow simultaneous detection of all gases within the mass range.

The new PV MassSpec software enables qualitative and quantitative analyses. This software offers a clear and user-friendly platform for recording and displaying measurement data and

parameter settings, and complete measuring procedures can be programmed and automated. With a range of instruments available, the mass ranges of 1–100 u, 1–200 u and 1–300 u are covered.



NEW PRODUCTS

The new instruments have low detection limits (depending on the mass range) of up to <100 ppb, gas consumption of 1–2 sccm and fast measuring time (up to 1 ms u^{-1}). For extended

New software for DART-MS

In collaboration with SpectralWorks, IonSense have added features to their AnalyzerPro XD software to permit parsing of samples within a single data file to individual data files for each sample. After parsing, each of the individual parsed data files can be exported to CSV files with the mass spectrum masses and intensities. You can also search each parsed data file against any NIST MS database or your own database. Target compounds can be mapped and wells containing your targets can be identified with the visual heat map. Also, the amount in each well can be determined and quantified. Classification with PCA or the use of other statistical analysis techniques can be performed to further analyse your data.

IonSense

► <http://link.spectroscopyeurope.com/32-081>

NMR

2D-barcode NMR tube scanner

The Express Scanner from Ziath ensures the traceability of samples in 2D-barcode nuclear magnetic resonance (NMR) tubes. It can decode a full rack of 2D-barcode NMR sample tubes in under 3 s. Traditionally 2D-barcode NMR tubes are difficult to read with a conventional scanner, as the caps are on the top of the tubes and most scanners are designed to read from underneath. The Express scanner is designed to operate on top of the sample tubes without applying any weight which could damage them. In addition, for Bruker NMR spectrometer users, Ziath provides a dedicated slide for their tube carriers which can only be inserted in one orientation: thereby conserving the origin position (the "A1" position) each time.

The Express Scanner is supplied with a controller which regulates scanning and data processing using Ziath industry-standard Datapaq software (version 3.17) or new DP5 web-enabled software, which enable exporting of tube barcodes in tables, as text, JPEG, XML, XLS files and in JSON formats. The Express Scanner controller can be separated and positioned under your lab bench.

Ziath

► <http://link.spectroscopyeurope.com/32-080>

RAMAN

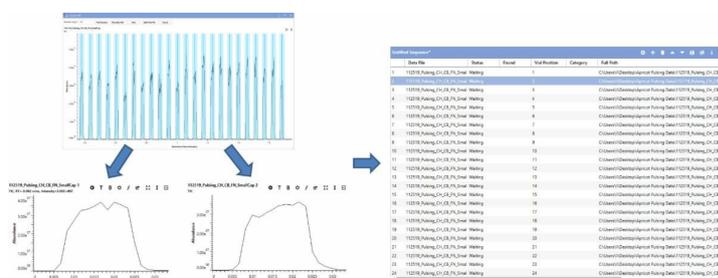
High-sensitivity NIR Raman spectrometer

Teledyne Princeton Instruments has introduced the TPIR-785 Raman spectrometer, an ultra-high-sensitivity spectrometer for NIR Raman spectroscopic application areas such as biology, medical research and life science where NIR excitation lasers (785 nm and 830 nm) are preferred. It uses an f/2 spectrograph with custom-designed lens optics to provide high light

process customisation, an integrated mass calibration device or a controlled purge gas system for corrosive gases are available.

Pfeiffer Vacuum

► <http://link.spectroscopyeurope.com/32-075>



throughput and imaging quality in the NIR spectral range. The TPIR-785 also allows users to tailor the instrument's performance for optimal spectral resolution (up to 5 cm^{-1}) or optimal spectral range (up to 80–3650 cm^{-1}). The system includes a 785 nm Raman probe, a universal fibre adaptor, a manual adjustable slit, and a high-power and temperature-stabilised 785 nm laser.

NEW PRODUCTS

The TPIR-785 Raman system uses a proprietary CCD that has NIR quantum efficiency $>70\%$ at 1000 nm. This “super-deep-depletion” sensor has a 1340×400 array composed of $20 \mu\text{m}$ square pixels and can be thermoelectrically cooled to -90°C for ultra-low dark current, allowing integration times from $10 \mu\text{s}$ to hours. The detector has spectral rates higher than 1 kHz, readout speeds up to 16 MHz and employs two readout ports. A new NIR optimised eXcelon™ process eliminates etaloning fringes and improves quantum efficiency. The high imaging quality spectrograph paired with large format detector allows the use of large fibre bundles for improved collection efficiency or Raman imaging measurement.

Teledyne Princeton Instruments

► <http://link.spectroscopyeurope.com/32-069>

New handled Raman spectrometer

B&W Tek have introduced a new version of their TacticID®-1064 1064 nm handheld Raman analyser that can analyse substances through opaque barriers. The TacticID®-1064 ST uses their patented STRaman® technology.

B&W Tek

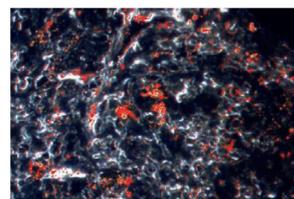
► <http://link.spectroscopyeurope.com/32-074>

Enhanced nanoparticle analysis

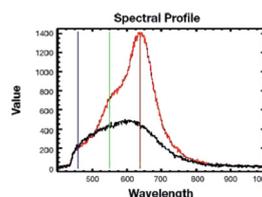
HORIBA Scientific has announced products using technology from CytoViva, Inc. that combine HORIBA's Raman microscopes with CytoViva's hyperspectral imaging (HSI) microscopy module and enhanced darkfield (EDF) illumination. This integration is of interest for applications related to nanomaterials research, drug delivery, nanotoxicology studies and SERS nanoparticles characterisation. HSI microscopy allows rapid imaging across the sample with high sensitivity. Coloured images generated from the spectra guide the user to easily locate nanoparticles and features of interest. CytoViva's patented EDF illumination improves the signal-to-noise ratio up to ten times over standard darkfield microscopes. The detection limit in size improves sufficiently to allow visualising nanoparticles as small as 10 nm when isolated.

Integrating Raman with HSI and EDF allows users rapidly to visualise the sample and target regions of interest. They can then perform Raman measurements from the identical field of view to provide and confirm the chemical identify of nanoparticles or other sample elements.

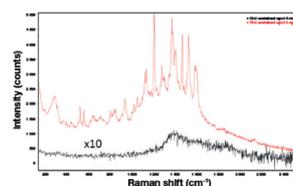
In the figure, gold nanoparticles are imaged in tissue (top), with the corresponding optical (bottom left) and Raman (bottom



Hyperspectral Mapping (Red) of Image Pixels



AuNP Optical Spectrum (Red) and Tissue (Black)



SERs Active AuNP Raman Signal (Red) and Tissue (Black)

right) spectra showing clear differences between the nanoparticles and tissue. Visualisation of the nanoparticles is simple with the HSI, while Raman provides a detailed chemical fingerprint.

HORIBA Scientific

► <http://link.spectroscopyeurope.com/32-070>

SOFTWARE

KnowItAll 2020 upgrade

A new version, KnowItAll 2020, has been released. There is a simplified interface to streamline workflow for spectral search. It also integrates spectral mixture analysis and spectral deconvolution capabilities directly into the interface. Spectral processing and IR and Raman functional group analyses have been streamlined. A new layout combines previously separate applications for each technique into a smart application that can recognise file

type. IR Spectra Classification for Designer Drugs allows researchers to submit an unknown spectrum for quick classification using Wiley's new IR Designer Drug Models. Compound classes include: amphetamines, anabolic steroids, barbiturates, benzodiazepines, cannabinoids, cathinones, cocaine-type substances, opioids, phencyclidine-type substances, phenethylamines, piperazines, steroids, tryptamines.

NEW PRODUCTS

Enhanced Simultaneous Multi-Technique Search has improved ability to search unknown spectra from disparate multiple technique spectra simultaneously and visualise the results on a scatter plot. Increased Chemical Intelligence with The new Related Compounds View provides increased insight into analyses by exploring a fuller view of compounds related to scientific results. This view interconnects all available reference data by structure, name, synonym and CAS registry number to add a high level of chemical intelligence to the entire platform.

DEA Controlled Substance Prediction enables users to match a structure with Drug Enforcement Agency 21 CFR Regulations on controlled substances. New tools in the enhanced structure drawing make it easier to move, highlight and select objects. There is also a more comprehensive set of pre-defined substituent fragments and expanded "fuzzy" structure representation features.

John Wiley & Sons

▶ <http://link.spectroscopyeurope.com/32-083>

UV/VIS

Improved version of the PEBBLE VIS ultra-compact spectrometer

Ibsen Photonics has released an improved version of its ultra-compact PEBBLE VIS OEM spectrometer. The new PEBBLE VIS combines an ultra-compact form factor of only 20 × 15 × 8 mm with high resolution and sensitivity as well as environmental ruggedness. The wavelength range has been shifted towards the UV to cover 360–830 nm, and the resolution is still 6 nm.

X-RAY

New tube-below WDXRF spectrometer from Rigaku

Rigaku Corporation has introduced the Rigaku ZSX Primus IVI tube-below sequential wavelength dispersive X-ray fluorescence (WDXRF) spectrometer. The new spectrometer has a tube-below configuration optimised for the measurement of a variety of sample types including liquids, alloys and plated metals. The efficiency of the new drive sequence decreases time between multiple high-speed precision measurements. The redesigned control systems optimise a series of movement sequences, also resulting in shortened measurement times. A patented vacuum partition system is used for analysing liquids. Because the spectroscopic chamber is separated from the sample chamber when changing helium gas, the conversion from vacuum atmosphere to helium atmosphere can be completed in under two minutes. Helium consumption is further reduced compared to models where the spectroscopic chamber must also be fully purged.

The Rigaku optical system is resistant to impact by sample surface height, and enables suppression of X-ray intensity changes caused by variation in distance from the tube. Accuracy

Software to combine EM spectroscopy and AFM data

Oxford Instruments and Digital Surf have announced the release of Relate software for users of Oxford Instrument's tools for materials characterisation. Relate software provides correlation of spectroscopy (EDS and EBSD) data with AFM data and microscopy images with easy-to-use tools for rapid manual and semi-automated correlation of multiple images. 3D and 2D visualisation of composite data sets show EDS, EBSD and EM image layers combined with topography and material properties as measured by AFM, helping to reveal the micro- and nano-characteristics of a sample. Correlated quantitative data can be analysed by extracting underlying data values in addition to qualitative images (e.g. x-ray counts for each element). Data and images can be organised and reports published in popular formats (PDF, Word etc.). The interactive workflow is documented allowing traceability during the image analysis process.

Oxford Instruments NanoAnalysis

▶ <http://link.spectroscopyeurope.com/32-067>

The pure transmission-based optics inside PEBBLE provide high sensitivity, low stray light and thermal stability. The detector is a CMOS array with 256 pixels, and the electronics interface is a tiny flex-connector that fits on the new miniature DISB-105 read-out electronics.

Ibsen Photonics

▶ <http://link.spectroscopyeurope.com/32-076>

of analysis is enhanced by minimising the effects from differences in fusion moulds used in glass bead formulation and the impact of uneven sample surfaces. The enhanced ZSX Guidance software, that comes with the spectrometer, guides the user through automated optimised analysis settings. The software includes a quantitative application auto-configuration features that automatically sets measurement conditions and various corrections based on the user's own standards. An error prevention function, which can set an access level for each operator, was also added for worry-free operation. The software is programmable for routine, everyday analysis using removable sample trays. Sample ID settings for each tray facilitates easy copy-and-paste for efficient measurements. Improved accuracy of liquid sample analysis is achieved by enabling the correction of geometry effect caused by the geometry of liquid sample cups.

Rigaku

▶ <http://link.spectroscopyeurope.com/32-077>

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Conferences

2020

2 September, United Kingdom. **20th Annual J.A. Woollam Spectroscopic Ellipsometer Online Workshop.** ✉ angela@qd-uki.co.uk, 🌐 <https://qd-uki.co.uk/20th-annual-woollam-workshop/>

20–25 September, Web Conference, Japan. **11th International Conference on Laser-Induced Breakdown Spectroscopy (LIBS2020).** Yoshihiro Deguchi, ✉ ydeguchi@tokushima-u.ac.jp, 🌐 <http://www.fm.ehcc.kyoto-u.ac.jp/SakkaLab/member/sakka/LIBS2020/index.htm>

6–7 October, Sanur, Bali. **The 4th International Seminar on Photonics, Optics, and its Applications (ISPhOA 2020).** 🌐 secretariat@isphoa.org, 🌐 <https://isphoa.org/>

6–8 October, Online. **SPIE Photonex + Vacuum Expo.** 🌐 <https://spie.org/>

[conferences-and-exhibitions/photonex-and-vacuum-expo](#)

12–15 October, Online. **47th Annual Conference of Federation of Analytical Chemistry and Spectroscopy Societies (SciX2020).** ✉ scix@scixconference.org, 🌐 <https://www.scixconference.org/event-3326054>

2021

14–16 January, Amsterdam, Netherlands. **11th Workshop on Hyperspectral Image and Signal Processing: Evolution in Remote Sensing (WHISPERS).** 🌐 <http://www.spectroexpo.com/whispers/>

15 January, Amsterdam, Netherlands. **Hyperspectral Sensing Meets Machine Learning and Pattern Analysis (HyperMLPA).** 🌐 <http://www.spectro-expo.com/hypermlpa/>

16 January, Amsterdam, Netherlands. **2nd Symposium on Short Wave Infrared**

Imaging and Spectroscopy (Swllms). 🌐 <http://www.spectroexpo.com/swiims/>

7–11 March, New Orleans, LA, United States. **Pittcon 2021: Conference on Analytical Chemistry and Applied Spectroscopy.** ✉ pittconinfo@pittcon.org, 🌐 <https://pittcon.org>

1–3 June, Kristiansand, Norway. **10th World Conference on Sampling and Blending (WCSB10).** ✉ contact@wcsb10.com, 🌐 <https://wcsb10.com>

1 June–31 July, , 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>

THE LAST WORD

Arrow Consumable ATR Slides to study adhesive curing

Unreacted aromatic isocyanate (NCO) molecules in laminated packaging can migrate into food to react with water molecules, generating carcinogenic primary aromatic amines. The packaging industry often use FTIR to screen test the decay rate of NCO in adhesive by measuring the reduction of NCO absorbance peak height over time at $\sim 2270\text{--}2250\text{cm}^{-1}$.

Multiple Arrow Slides can also be used to compare the curing speed of different adhesives at the same time. Adhesive curing speed has many variables like OH to NCO mix ratio, curing temperature, relative humidity, the crosslink-ability of starting components and the reactivity of the aromatic NCO component. This can take days or even weeks, so to have just one conventional crystal ATR puck occupied for prolonged periods might not be the coolest idea!

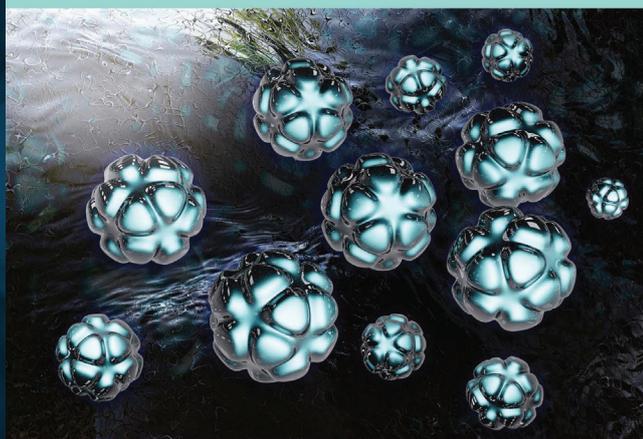
High volume batch sampling using Arrow consumable ATR Slides can really save you time and speed up your work to make your organisation more competitive!

Furthermore, once the polyurethane product is formed on your crystal, it could be difficult to remove and you might run the risk of damaging a relatively more expensive crystal puck; after all glue is designed to stick! So, the next time you test a sticky adhesive, varnish, paint, ink, or coating, why not give the Arrow a go?

Specac



Visit our website (www.specac.com) to learn more about Arrow Consumable ATR Slides
If you have any questions you'd like answered in the next "Last Word" contact us on Sales@specac.co.uk



Nanoparticle Analysis using the Sensitivity of ICP-MS



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