Vol. 32 No. 3 June/July 2020

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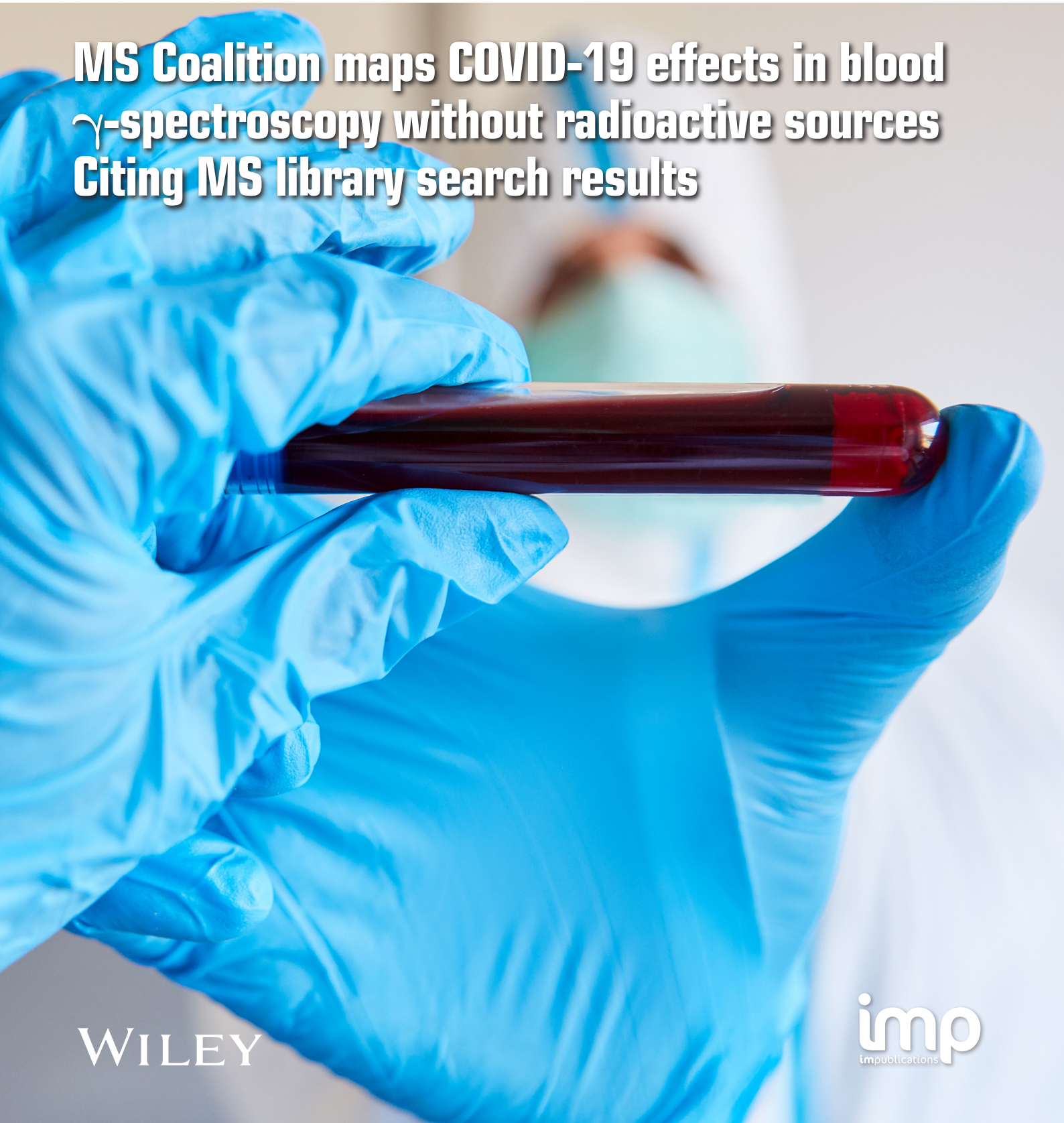
MS Coalition maps COVID-19 effects in blood

γ-spectroscopy without radioactive sources

Citing MS library search results



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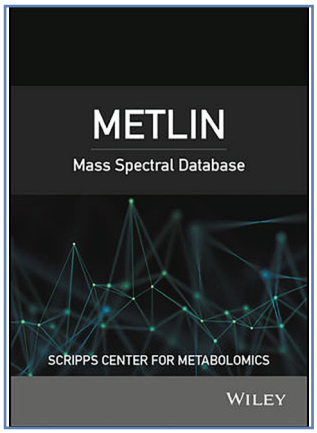
METLIN Mass Spectral Database

By Wiley*



Developed by the Scripps Center for Metabolomics, METLIN Mass Spectral Database by Wiley is the most complete MS/MS database in the field. With content divided into 3 libraries – experimental spectra, in-silico spectra, and searchable chemical structures – METLIN by Wiley has optimized its searching capabilities and validated spectrum quality to ensure the broadest coverage available.

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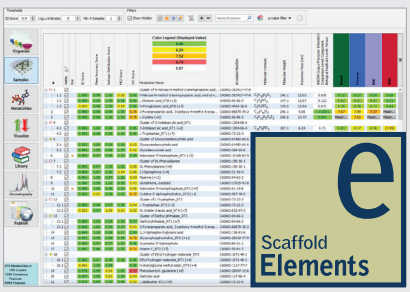

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EDITORIAL

In our article, Marco Bortoluzzi and Valentina Ferraro tell us about "^{138}La as a useful isotope for gamma spectroscopy without radioactive sources". It is difficult to provide practical experience with gamma spectroscopy in undergraduate laboratories, and the authors have been investigating alternative sources, and the ^{138}La isotope is a suitable candidate. As well as some background on gamma spectroscopy and radiochemistry, Marco and Valentina also provide some sample exercises that could be used for undergraduate practicals.

Our second contribution is from David Sparkman, who will be well known to all readers in the mass spectrometry field. David is concerned about the "Importance of citing full details of spectral library and search program" in papers. As he points out, "even when the same mass spectrum is searched against two different versions of the NIST/EPA/NIH EI Mass Spectral Library using the same search program, two different compounds can be found as the first Hit". There are many possible reasons for this, but the version of the spectral library used and the particular search program used make a difference, and should be included in all references.

Tony Davies and Mohan Cashyap discuss "COVID-19 #2: Compliant data processing from your home office" with help from a number of industry experts. Whilst there are undoubted computing and networking issues for regulated industries in allowing working from home as if the user was in the lab, they are not insurmountable.

Kim Esbensen has enlisted the help of two business consultants who have given their views on "Theory of Sampling—an approach to representativity offering front line companies added value and potential substantial savings". They show how fundamental accurate analytical results (and that, of course, requires representative sampling) are to many aspects of business and of trade between companies. Some appreciation of the Theory of Sampling and its importance should be in place in the board room and within senior management. Time for a CSO, a Chief Sampling Officer, perhaps?

We also have details of new mass spectrometry products launched at the ASMS 2020 Reboot virtual event. Given the deadline for printing this issue, we only have early information on some products and not all have been released as we go to press.

Finally, there are links to selected content published online that we have not had space to include here: page 29.

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An international group of mass spectrometrists have formed the COVID-19 MS Coalition, to work together to look at the ways in which the novel coronavirus is present in patients' blood and examine in detail how the virus is structured. Find out more on page 6.

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

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

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

Europe and the Rest of World
Vanessa Winde
Wiley-VCH Verlag GmbH & Co. KGaA,
Boschstraße 12, 69469 Weinheim, Germany
Tel: +49 6201 606 721
E-mail: vanessa.winde@wiley.com

Paid subscriptions
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CONTENTS

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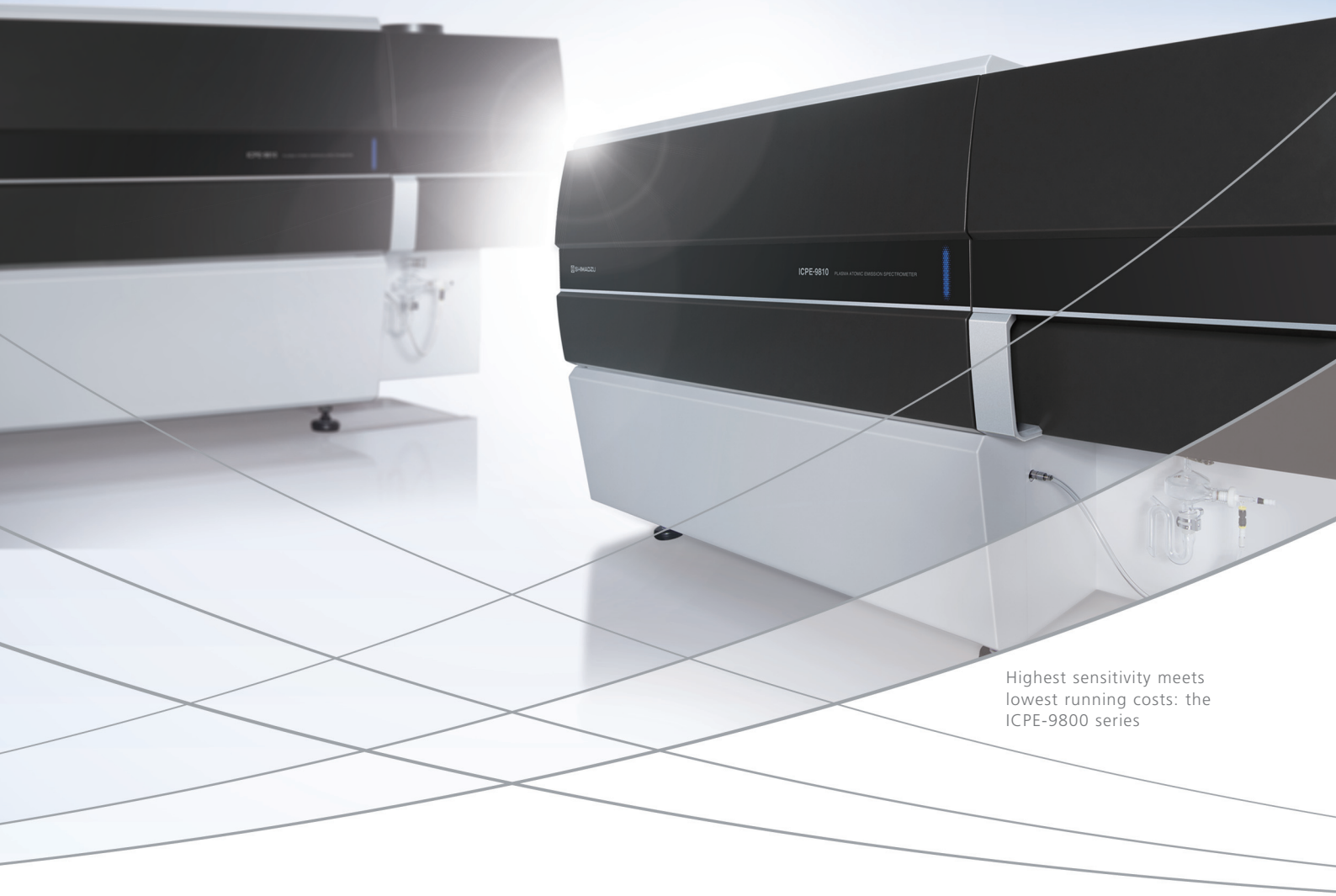

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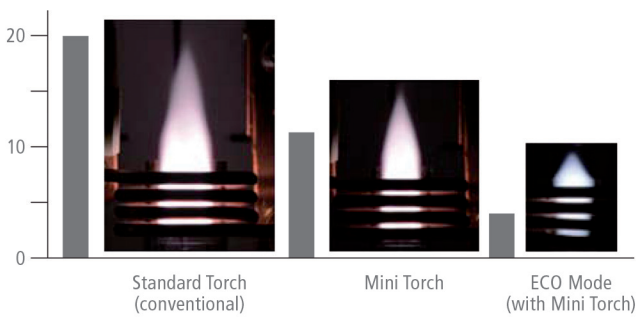

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NEWS

Awards

Yuki Ozaki to receive Charles Mann Award for 2020

The Charles Mann Award for 2020 will be presented to Professor Yukihiro Ozaki of Kwansei Gakuin University. The award recognises an individual who has demonstrated advancement(s) in the field of applied Raman spectroscopy presented at the FACSS SciX conference; and/or demonstrated dedication to the advancement of the Raman spectroscopy programme at the FACSS SciX conference and/or the ASTM Raman subcommittee.

Yuki Ozaki

Yuki Ozaki obtained his PhD (1978) degree from Osaka University, Osaka, Japan. After that he spent two and a half years at the National Research Council, Canada as a research associate and then he joined the Jikei University School of Medicine in Tokyo in 1981 as an assistant professor. Immediately after joining the medical school he started studies of medical applications of Raman spectroscopy, particularly non-destructive Raman studies on cataract genesis. In 1980s he was also involved in Raman microscope studies of gallstones. In 1989 he moved to Kwansei Gakuin University. In early 1990s using FT-Raman he demonstrated the potential of Raman spectroscopy in exploring cancer tissues at the molecular level. In 1997 he started biomedical applications of SERS. He published a number of papers on SERS immunoassay and label-free protein detection. Also, he has been one of driving forces in the investigations of both electromagnetic and chemical mechanisms of SERS. Recently, Professor Ozaki reported non-destructive diagnosis of oesophageal cancer in an early stage with Raman spectroscopy. In this study he employed new idea in chemometrics (a kind of neural network) for the diagnosis of cancer tissues. Professor Ozaki has carried out interesting Raman spectroscopy studies on fundamental biology; for example, he reported non-destructive analysis of mouse embryo development and its qualitative evaluation using Raman spectroscopy.

Gordon F. Kirkbright and Edward Steers Bursary Awards 2021

The Gordon F. Kirkbright bursary award is a prestigious annual award that assists a promising early career scientist of any nation to attend a recognised scientific meeting or visit a place of learning. The fund for this bursary was established in 1985 as a memorial to Professor Gordon Kirkbright in recognition of his contributions to analytical spectroscopy and to science in general.

Owing to the generosity of one of the ABS former trustees, an eminent atomic spectroscopist, Professor Edward B.M. Steers, they are now, from 2020, in the position of being able to award an annual Edward Steers bursary, in addition to the long-standing Gordon Kirkbright bursary, to similarly assist a promising early scientist engaged in or utilising analytical spectroscopic techniques.

The ABS Trust defines early career as being either a student, or an employee in a non-tenured academic post or in industry, within seven years of award of PhD excluding career breaks. The same conditions apply to each bursary.

Applications are invited for both the 2021 Gordon Kirkbright Bursary and the 2021 Edward Steers Bursary. Although both funds are administered by the ABS Trust, the Kirkbright award is not restricted to spectroscopists, but is open to all involved with or utilising analytical science-based techniques.

Application forms can be downloaded from http://www.abstrust.org/kirkbright-and-steers-bursary-awards or for further information visit http://www.abstrust.org or contact abstrustuk@gmail.com.

The closing date for entries is 30 November 2020.

Mass spec coalition to map COVID-19 effects and treatments in blood

A new coalition of more than 500 scientists from around the world has been created to share data on COVID-19 gleaned from the use of mass spectrometry techniques to examine people's blood and other biomarkers. Announced in *The Lancet* (doi: http://doi.org/dww4), and coordinated from The University of Manchester, the

2020 Thomson Medal Award winners

The International Mass Spectrometry Foundation has awarded the 2020 Thomson Medals to Professor Alison Ashcroft (University of Leeds, UK) and Professor Ron Heeren (Maastricht University, Netherlands). Congratulations to both.

Details on the history of the awards and previous winners can be found on the IMSF website (https://www.imss.nl/awards.html).

COVID-19 MS Coalition is made up of many of the world's leading mass spectrometry experts who will work together to look at the ways in which the novel coronavirus is present in patients' blood and examine in detail how the virus is structured. The aim is to refine testing approaches, stratify treatment options, determine isolation requirements and bring much needed speed into measurement aspects of novel therapeutic development programmes—for COVID-19 and future threats.

The coalition partners are also looking for biomarkers that will determine how a given individual will respond to the virus. These allow hospital labs to predict the outcome of the disease and to target treatment accordingly. By finding the biological pathways that alter as the disease takes hold, and considering genetic risk factors, mass spectrometry will provide crucial evidence as to why people respond differently. Mass spectrometry will be also be able to help develop effective treatments by targeted studies that measure the decrease in these markers.

The researchers will also attempt to define the precise structure of the viral spike protein and other antigens. Mass spectrometry is the only method that can map the complex sugar network that coats the surface of the viral spike protein and the human receptor. Coalition partners are working to see which parts of the virus are involved in the interaction with cells, and how this interaction allows the virus to open and drop the infective RNA into the human host. This detailed mapping of the interaction is vital in the development of vaccines, designed to be a weaker form of the virus.

COVID-19 therapy potential targets highlighted by new mass spectrometry technique

A team of biochemists and virologists at Goethe University and the Frankfurt University Hospital were able to observe how human cells change upon infection with SARS-CoV-2, the virus causing COVID-19 in people. The scientists tested a series of compounds in laboratory models and found some which slowed down or stopped virus reproduction. These results now enable the search for an active substance to be narrowed down to a small number of already approved drugs (*Nature*, http://doi.org/dw7s). Based on these findings, a US company reports that it is preparing clinical trials. A Canadian company is also starting a clinical study with a different substance.

Since the start of February, the Medical Virology of the Frankfurt University Hospital has been in possession of a SARS-CoV-2 infection cell culture system. The Frankfurt scientists in Professor Sandra Ciesek's team succeeded in cultivating the virus in colon cells from swabs taken from two infected individuals returning from Wuhan (http://doi.org/ggpw78). Using a mass spectrometry technique developed at the Institute for Biochemistry II at Goethe University Frankfurt, researchers from both institutions were together able to show how a SARS-CoV-2 infection changes the human host cells. The scientists used a particular form of MS called the multiplexed enhanced protein dynamics (mePROD) method, which they had developed only a few months previously. This method makes it possible to determine the amount and synthesis rate of thousands of proteins within a cell.

The findings paint a picture of the progression of a SARS-CoV-2 infection: whilst many viruses shut down the host's protein production to the benefit of viral proteins, SARS-CoV-2 only slightly influences the protein production of the host cell, with the viral proteins appearing to be produced in competition to host cell proteins. Instead, a SARS-CoV-2 infection leads to an increased protein synthesis machinery in the cell. The researchers suspected this was a weak spot of the virus and were indeed able to significantly reduce virus reproduction using translation inhibitors, which shut down protein production.

Twenty-four hours after infection, the virus causes distinct changes to the composition of the host proteome: while cholesterol metabolism is reduced, activities in carbohydrate metabolism and in modification of RNA as protein precursors increase. In line with this, the

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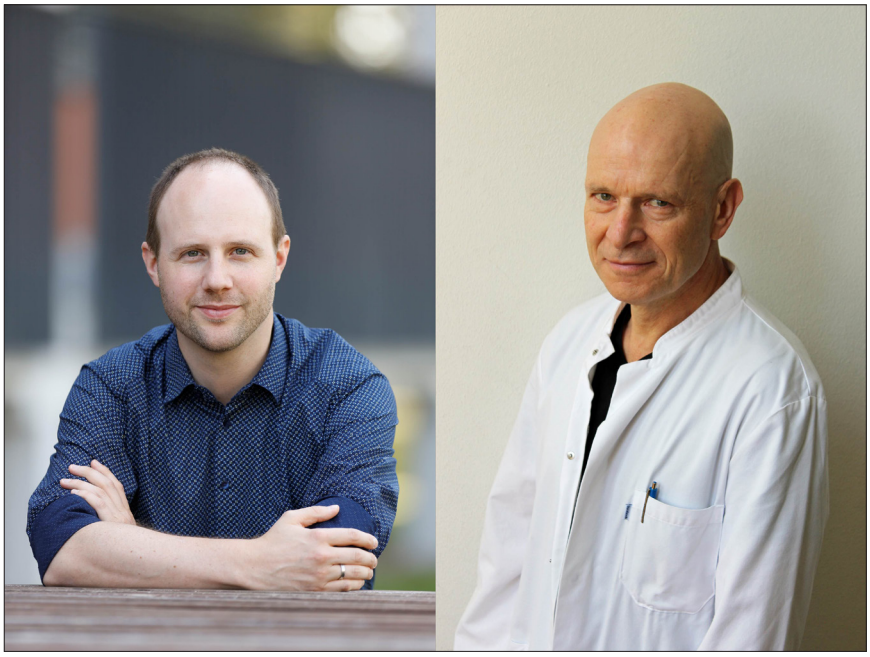


Left: Dr Christian Münch (Credit: Uwe Dettmar for Goethe University Frankfurt). Right: Prof. Dr. rer. nat. Jindrich Cinatl (Credit: University Hospital Frankfurt).

scientists were successful in stopping virus reproduction in cultivated cells by applying inhibitors of these processes. Similar success was achieved by using a substance that inhibits the production of building blocks for the viral genome.

In keeping with common practice since the beginning of the corona crisis, the Frankfurt researchers made these findings immediately available on a preprint server and on the website of the Institute for Biochemistry II (http://pqc.biochem2.de#coronavirus). Professor Ivan Dikic, Director of the Institute, comments: "Both the culture of 'open science', in which we share our scientific findings as quickly as possible, and the interdisciplinary collaboration between biochemists and virologists contributed to this success. This project started not even three months ago, and has already revealed new therapeutic approaches to COVID-19."

Professor Sandra Ciesek, Director of the Institute for Medical Virology at the University Hospital Frankfurt, explains: "In a unique situation like this we also have to take new paths in research. An already existing cooperation between the Cinatl and Münch laboratories made it possible to quickly focus the research on SARS-CoV-2. The findings so far are a wonderful affirmation of this approach of cross-disciplinary collaborations."

Among the substances that stopped viral reproduction in the cell culture system was 2-Deoxy-D-Glucose (2-DG), which interferes directly with the carbohydrate metabolism necessary for viral reproduction. The US company Moleculin Biotech possesses a substance called WP1122, a prodrug similar to 2-DG. Recently, Moleculin Biotech announced that they are preparing a clinical trial with this substance based on the results from Frankfurt, https://www.moleculin.com/covid-19/.

Based on another one of the substances tested in Frankfurt, Ribavirin, the Canadian company Bausch Health Americas is starting a clinical study with 50 participants: https://clinicaltrials.gov/ct2/show/NCT04356677?term=04356677&draw=2&rank=1

Dr Christian Münch, Head of the Protein Quality Control Group at the Institute for Biochemistry II and lead author, comments: "Thanks to the mePROD-technology we developed, we were for the first time able to trace the cellular changes upon infection over time and with high detail in our laboratory. We were obviously aware of the potential scope of our findings. However, they are based on a cell culture system and require further testing. The fact that our findings may now immediately trigger further *in vivo* studies with the purpose of drug development is definitely a great stroke of luck." Beyond this, there are also other potentially interesting candidates among the inhibitors tested, says Münch, some of which have already been approved for other indications.

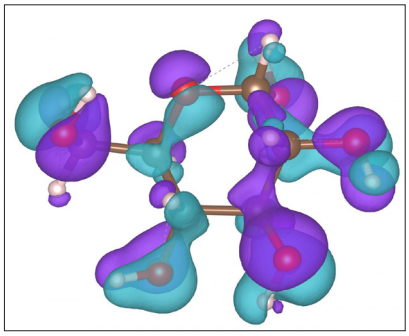
Professor Jindrich Cinatl from the Institute of Medical Virology and lead author explains: "The successful use of substances that are components of already approved drugs to combat SARS-CoV-2 is a great opportunity in the fight against the virus. These substances are already well characterised, and we know how they are tolerated by patients. This is why there is currently a global search for these types of substances. In the race against time, our work can now make an important contribution as to which directions promise the fastest success."

2D "sandwich" is a SERS superstar

Tests at Rice University's Brown School of Engineering of a two-dimensional Janus compound showed it could be an effective and universal platform for improving the detection of biomolecules via surface-enhanced Raman spectroscopy (SERS). Using glucose to test the material proved its ability to boost its Raman enhancement factor by more than 100,000 times, which the researchers say is comparable to the highest-reported enhancement factor for 2D substrates.

Metallic SERS media often prompt side reactions that create background noise. Janus MoSSe synthesised at Rice University is non-metallic. "This work mainly addresses whether we can enhance the target molecules' signal strength", said materials scientist and principal investigator Jun Lou. "We wanted to know if we could make it stand out from the background noise."

MoSSe introduced by the Lou lab in 2017 was produced by chemical vapour deposition. Molybdenum sits in the middle with a layer of sulfur on one side



A model created at Rice University illustrates charge distribution in glucose. The light blue region shows the electron cloud distribution in a single glucose molecule. The purple regions show the drastic charge redistribution when anchored to Janus MoSSE and detected via surface-enhanced Raman spectroscopy. Credit: Lou Group/Rice University

and another of selenium on the other; hence the two-faced Janus characterisation. The different electronegativities of each layer make it a SERS "superstar", said lead author and Rice alumnus Shuai Jia, a former graduate student in Lou's lab.

"The dipole created between the top sulfur and the bottom selenium lands out-of-plane, and this creates an electrical field a few nanometres beyond the MoSSe", Jia said. That field interacts with molecules that come close, enhancing their vibrational intensity enough to be detected.

The researchers noted tests with MoSSe also detected molecules of the neurotransmitter dopamine and that the substrate should be adaptable to sense other molecules.

Lou said that there is room for improvement. "We're looking at hybrids of MoSSe with some metallic nanoparticles, and also trying to enhance the dipole strength", he said.

This work is reported in *Nanoscale* (http://doi.org/dvt7).

NMR spectroscopy authenticates argan oil

Researchers from the Quadram Institute have developed a new way to test the authenticity of argan oil, one of the world's most expensive edible oils. In recent years, it has become sought after as an ingredient in cosmetics and health products, significantly increasing its value. Using a benchtop NMR spectrometer developed by Oxford Instruments, the method can screen for quality and authenticity, detecting when argan oil has been mixed with cheaper alternatives. A screen of 28 commercially available products labelled as 100% argan oil found four that were not pure argan, strongly indicating adulteration with other oils. With demand for argan oil soaring, this new method can help protect consumers from fraud, and support honest producers and suppliers.

Argan oil is one of the world's most expensive edible oils.

Argan oil is made from the kernels of the argan tree, which grows exclusively in Morocco, and is recognised as a Protected Geographical Indication product. It is traditionally produced by hand and used both as a medicine, particularly for skin conditions, and as part of the diet, where it also is reputed to provide health benefits. Scientific validation of its health claims is patchy, but like olive oil it can be a key component of a healthy Mediterranean style diet. It is rich in fatty acids, polyphenols and other components that have been linked to reductions in chronic health conditions.

More recently, there has been an explosion in demand driven by the cosmetics industry, keen to include it as an ingredient in hand creams, haircare products and cosmetics. Mechanical presses have replaced hand extraction, but the trees themselves are slow growing, so demand remains high, as does the price. The market is estimated to reach over $500 million by 2027, so it's no wonder it's known colloquially as Moroccan liquid gold.

All of this makes argan oil an attractive target for fraudsters who can substitute cheap vegetable oils for argan oil, which costs around $30 per litre. Because it is used as an ingredient in cosmetics, the consumer, and even the manufacturer, may not notice the difference. Dr Kate Kemsley and her team from the Quadram Institute, working with Oxford Instruments, have developed a new way of authenticating argan oil that is quick, high throughput and robust. The method uses a benchtop NMR spectrometer to measure the profile of different mono-unsaturated (MUFA), poly-unsaturated (PUFA) and saturated fatty acids (SFAs). Through a collaboration with the Centre National de l'Energie des Sciences et des Techniques Nucléaires (CNESTEN) in Rabat, Morocco, the researchers obtained samples of argan oil with known provenance. Samples came from different regions and were taken at different times, to allow the researchers to get a view of the natural variation in the fatty acid profiles.

Samples of argan oil deliberately mixed with sunflower oil at different ratios were also tested, and these could

be distinguished from 100% argan oil. The researchers also tested a range of different vegetable oils. Most could be distinguished, but some, for example bran oil, looked very similar based on their MUFA, PUFA and SFA profiles. Whilst these compounds make up the major part by weight of these oils, there are a range of other minor compounds present including phenolics, tocopherols and free fatty acids. Using bespoke computational techniques to incorporate this information from the whole spectrum allowed them to identify 15 different types of oil as different to argan oil.

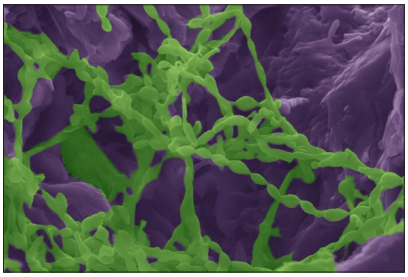
The test only takes 5 minutes and requires no sample preparation or solvents. It doesn't need to be run in a laboratory and requires no particular expertise to perform, making it suitable for *in situ* testing. It provides a clear decision and gives information with an accuracy and precision suitable for "typical value" food labelling. The research was funded by the Biotechnology and Biological Sciences Research Council (BBSRC), part of UKRI, with support from Oxford Instruments and the Food and Agriculture Organisation of the United Nations.

In a survey of commercially available oils, the researchers bought 28 different products labelled as 100% argan. Four of these failed the authentication test, most probably due to adulteration with other vegetable oils. This shows that there is already argan oil fraud taking place. With demand for argan oil showing no signs of fading, this new method should give retailers and manufacturers a new tool to help ensure that the products they supply to consumers are what they say they are.

Details of the research have been published in *Magnetic Resonance in Chemistry* (doi: https://doi.org/drf9).

Raman imaging shows how microorganisms can survive in parched regions

In Northern Chile's Atacama Desert, one of the driest places on Earth, microorganisms are able to eke out an existence by extracting water from the rocks they colonise. Through work in the field and laboratory experiments, researchers at the University of California, Irvine, as well as Johns Hopkins University and UC Riverside, have gained an in-depth understanding of the mechanisms by which some cyanobacteria survive in harsh surroundings. The new insights, published in *Proceedings of the National Academy of Sciences* (doi: http://doi.org/dvhb), demonstrate how life can flourish in places without much water in evidence—such as Mars—and how people living in arid regions may someday derive hydration from available minerals.



Microorganisms (highlighted in green) colonise gypsum rock (highlighted in purple) to extract water from it. University of California, Irvine and Johns Hopkins researchers ran lab experiments to understand the survival mechanisms of these cyanobacteria, confirming that they transform the material they occupy to an anhydrous state. David Kisailus / UCI

The research team focused on the interactions of *Chroococcidiopsis*, a desiccation-resistant cyanobacteria found in deserts around the world, and gypsum, a water-containing calcium sulfate-based mineral. The colonising lifeforms exist beneath a thin layer of rock that gives them a measure of protection against the Atacama's high solar irradiance, extreme dryness and battering winds. Gypsum samples harbouring cyanobacteria and sent them to Kisailus' lab for materials analysis.

In one of the most striking discoveries of the study, the researchers learned that the microorganisms change the very nature of the rock they occupy. By extracting water, they cause a phase transformation of the material—from gypsum to anhydrite, a dehydrated mineral. According to DiRuggiero, the impetus for the published work came when Wei Huang, a UCI postdoctoral scholar spotted data showing an overlap in concentrations of anhydrite and cyanobacteria in the gypsum samples collected in the Atacama.

"Our analysis of the regions of rock where microbes were colonised revealed a dehydrated phase of calcium sulfate, suggesting that they extract water from the rock to survive", Kisailus said. "We wanted to do some more controlled experiments to validate that hypothesis." DiRuggiero's team then allowed the organisms to colonise half-millimetre cubes of rock, called coupons, under two different conditions: one in the presence of water, to mimic a high-humidity environment, and the other completely dry. Amid moisture, the gypsum did not transform to the anhydrite phase.

The cyanobacteria "didn't need water from the rock; they got it from their surroundings", Kisailus said. "But when they were put under stressed conditions, the microbes had no alternative but to extract water from the gypsum, inducing this phase transformation in the material."

His team utilised a combination of electron microscopy and Raman spectroscopy to examine the interactions between the biological and geological counterparts, finding that the organisms bore into the rock by excreting a biofilm containing organic acids. Huang employed a modified electron microscope equipped with a Raman spectrometer to discover that the cyanobacteria used the acid to penetrate the gypsum in specific crystallographic directions—only along certain planes where they could more easily access the water existing between faces of calcium and sulfate ions.

Kisailus said the project was a great example of interdisciplinary collaboration between microbiologists and materials scientists. "Researchers have suspected for a long time that microorganisms might be able to extract water from minerals, but this is the first demonstration of it", DiRuggiero said. "This is an amazing survival strategy for microorganisms living at the dry limit for life, and it will guide our search for life elsewhere."

Researchers have developed a way to use smartphone images of a person's eyelids to measure blood haemoglobin levels. To take the measurement, the patient pulls down the inner eyelid to expose the small blood vessels underneath. A healthcare professional or trained person then uses the smartphone app developed by the researchers to take pictures which are then automatically analysed to determine the haemoglobin level. Credit: Young Kim, Purdue University

Smartphone-based spectral super-resolution spectroscopy to measure haemoglobin levels

Researchers have developed a way to use smartphone images of a person's eyelids to assess blood haemoglobin levels. The ability to perform one of the most common clinical lab tests without drawing blood could help reduce the need for in-person clinic visits, make it easier to monitor patients who are in critical condition, and improve care in low- and middle-income countries where access to testing laboratories is limited.

"Our new mobile health approach paves the way for bedside or remote testing of blood haemoglobin levels for detecting anaemia, acute kidney injury and haemorrhages, or for assessing blood disorders such as sickle cell anaemia", said research team leader Young Kim from Purdue University. "The COVID-19 pandemic has greatly increased awareness of the need for expanded mobile health and telemedicine services."

Kim and colleagues from the University of Indianapolis, Vanderbilt University School of Medicine in the US and Moi University School of Medicine in Kenya report the new approach in *Optica* (doi: http://doi.org/dwt6). They used software to transform the built-in camera of a smartphone into a hyperspectral imager that reliably measures haemoglobin levels without the need for any hardware modifications or accessories. A pilot clinical test with volunteers at the Moi University Teaching and Referral Hospital showed that prediction errors for the smartphone technique were within 5–10% of those measured with clinical laboratory blood.

"This new technology could be very useful for detecting anaemia, which is characterised by low levels of blood haemoglobin", said Kim. "This is a major public health problem in developing countries, but can also be caused by cancer and cancer treatments."

The researchers used spectral super-resolution spectroscopy, which uses software to virtually convert photos acquired with low-resolution systems such as a smartphone camera into high-resolution digital spectral signals. The researchers selected the inner eyelid as a sensing site because microvasculature is easily visible there; it is easy to access and has relatively uniform redness. The inner eyelid is also not affected by skin colour, which eliminates the need for any personal calibrations.

To perform a blood haemoglobin measurement with the new technique, the patient pulls down the inner eyelid to expose the small blood vessels underneath. A healthcare professional or trained person then uses the smartphone app developed by the researchers to take pictures of the eyelids. A spectral super-resolution algorithm is applied to extract the detailed spectral information from the camera's images and then another computational algorithm quantifies the blood haemoglobin content by detecting its unique spectral features.

The mobile app includes several features designed to stabilise smartphone image quality and synchronise the smartphone flash light to obtain consistent images. It also provides eyelid-shaped guidelines on the screen to ensure that users maintain a consistent distance between the smartphone camera and the patient's eyelid. Although the spectral information is currently extracted using an algorithm on a separate computer, the researchers expect that the algorithm could be incorporated into the mobile app.

"Our work shows that data-driven and data-centric light-based research can provide new ways to minimise hardware complexity and facilitate mobile health," says Kim. "Combining the built-in sensors available in today's smartphones with data-centric approaches can quicken the tempo of innovation and research translation in this area."

Photothermal spectroscopy measures energy conversion efficiency

Conversion of energy is a constant process but measuring the efficiency of this conversion is not an easy task. Quantifying the heat emission of the object that absorbs energy has been proven to be a good indicator. Scientists at the Tokyo University of Science have devised a photothermal spectroscopy technique that can perform this measurement easily and accurately, and this novel technology can shed light on the

energy transfer processes in systems ranging from plants to solar cells.

Energy is in a constant universal cycle of use, reform and reuse. Through this process, there are often several situations where energy is received in one form and converted to another form, or even to a non-energy form; photosynthesis is an example for this. However, what is the efficiency of such energy conversion? To put in simple terms, energy conversion efficiency is the ratio of useful output of an energy-converting system like a plant and the total energy that it receives in the first place. This value is important especially in the planning of energy-efficient structures like solar cells. However, although the theory is simple, there is no established method to accurately measure factors that determine light energy conversion efficiency, like total energy or total electrical power generated.

One alternative technique that has been explored for solving this issue is the measurement of heat over light. Everything that absorbs energy tends to dissipate that energy in the form of heat. This release of heat is greater immediately following energy absorption and reduces as time passes. This is in contrast with light emission by systems like "phosphorescent" materials, which absorb energy and only release light much later. Therefore, measuring heat release as a function of excitation light wavelength—the photothermal excitation spectrum (PTES)—can be a viable method to measure energy conversion efficiency. Photothermal deflection spectroscopy is one method for the direct application of PTES. However, very little research has investigated PTES independently of light emission.

The team at the Tokyo University of Science led by Professor Eiji Tokunaga had previously created a Sagnac interferometer photothermal deflection spectroscopy (SIPDS) technique, which improved the efficiency of the existing techniques by one magnitude. Photothermal spectroscopy detects the heat generated when the irradiated light is absorbed by the sample, and therefore, it can measure the absorption spectrum for samples of any shape and with any properties, such as "scatterers" whose transmitted light cannot be measured. Professor Tokunaga says, "Since about 2010, we have been working on increasing the sensitivity of photothermal deflection spectroscopy using an interferometer, and with the collective efforts of everyone including students, we could analyse samples in air that have seldom been analysed before, giving us the ability to measure the absorption spectrum over the entire visible light range. This upgraded technology enabled us to evaluate the quantum efficiency of luminescence or chemical energy conversion."

Taking this technology a step forward, these scientists, along with Dr Kohsei Takahashi and Dr Naoto Hirosaki from the Sialon Group, National Institute for Materials Science (NIMS), have now integrated "balanced detection", which is essentially a technique to measure small variances in values, into their SIPDS technology. This new innovation uses a white-light lamp as a source of energy and can measure the photothermal excitation spectrum of materials in the air. The scientists noticed that no heat is generated, meaning that light energy is converted to effective energy, so the difference from the absorption spectrum could be measured to determine the light energy conversion efficiency.

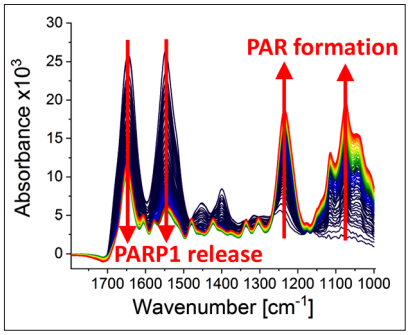
They were able to use this technology to measure the heat spectrum (PTES) of a high-efficiency luminescent red phosphor of white LED, made by NIMS, successfully and compared it with their photoluminescence excitation spectrum (PLES), which showed the amount of light emitted by the phosphor as a function of excitation light wavelength (see figure). This comparison provided accurate photoluminescence efficiency values of the phosphor as well, which is a measure of how well a substance can emit light. "We can use our technology to measure the thermal relaxation spectra of materials over the whole visible range in the weak excitation limit 50 $\mu W cm^{-2}$, which is a never-before-achieved breakthrough", remarks Professor Tokunaga. Thus, the measurement of energy conversion efficiency, which had previously required expensive and different devices such as phosphors, solar cells and photosynthesis [to measure the converted effective energy (emission energy, electrical energy, chemical energy)], could be done using one simple, unified method. They reported their work in *Applied Sciences* (http://doi.org/dtph).

Once developed further, measuring the energy conversion efficiency of even photosynthesis in "live" leaves can be performed. Hopefully, these findings can stimulate and accelerate research aimed at improving the conversion efficiency of substances and realise a society with high energy conversion efficiency.

Real-time IR observation of enzymatic processes on DNA

DNA strand breaks can contribute to the development of cancer and the ageing process. Researchers from the Departments of Biology and Chemistry of the University of Konstanz have now been able to observe in real time the molecular processes that take place at DNA strand breaks by means of infrared spectroscopy. DNA damage in general and DNA strand breaks in particular occur every day in all cells of the human body. This is due to internal influences such as free radicals, which are produced during inflammatory processes and cellular respiration, and external ones, such as cosmic background radiation or X-rays in the course of medical diagnostic measures. DNA strand breaks can lead to cell death or to mutations and thus contribute in the long term to cancer development or the ageing process.

Cells possess molecular tools to repair such DNA strand breaks very efficiently. One of them is the enzyme poly(ADP-ribose) polymerase 1 (PARP1), which detects DNA strand breaks and thereby initiates downstream repair processes. By binding to a DNA strand break, PARP1 is (catalytically) activated and uses the substrate nicotinamide adenine dinucleotide (NAD+) to produce poly(ADP-ribose) (PAR), a chain-shaped biopolymer. This serves as a signal transmitter in the cell and coordinates the further DNA damage response. In the further course of the process, PARP1 detaches from the site of damage again, thus clearing the way for subsequent



Infrared spectra at different points of time (0–79 min) after the poly(ADP-ribosyl)ation reaction started due to the addition of PARP1 substrate NAD+. The following can be observed: the dynamic formation of the biopolymer poly(ADP-ribose) (absorption bands at 1236 cm^{-1} and 1074 cm^{-1}) and the detachment of PARP1 from the DNA strand break (absorption bands at 1645 cm^{-1} and 1548 cm^{-1}). Credit: Modified from Krüger *et al.* (http://doi.org/dtpd).

steps in DNA repair. This process is of medical importance, even more so as pharmacological inhibitors of PARP1 have recently been introduced into cancer therapy.

Scientists at the University of Konstanz (working groups of Professor Aswin Mangerich and Professor Alexander Bürkle, Department of Biology, and working group of Professor Karin Hauser, Department of Chemistry) have now been able to visualise in detail the biochemical processes which PARP1 fulfils at a DNA strand break. To this end, they used attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR), which had also been successfully used in a previous study on the interactions of the tumour suppressor protein p53 with DNA and PAR.

"What is special about our new study is that we can now investigate the molecular processes that PARP1 undergoes at DNA strand breaks in real time. This enabled us to unveil dynamic changes in the protein structure and thus gain further insights into the underlying mechanisms," said Dr Annika Krüger, who worked on the project as part of her doctoral thesis. She is now pursuing research at the Karolinska Institute in Stockholm, Sweden.

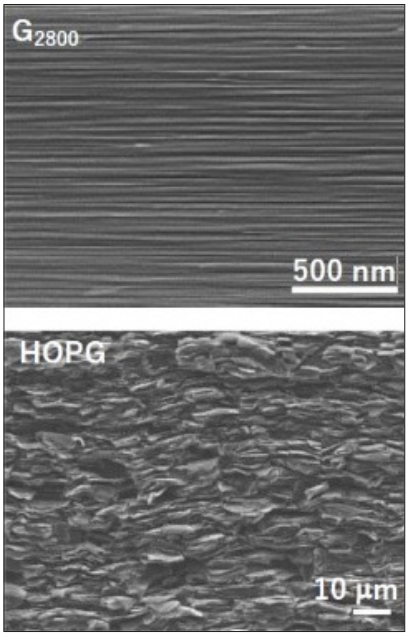
As a matter of principle, this spectroscopic method can be used to investigate also other enzymatic processes that take place at the DNA, in detail and with molecular resolution. In the long term, this may contribute to better understanding of the mechanisms of cancer development and ageing, as well as of the mode of action of anticancer drugs. The study was published in *Nature Communications* (http://doi.org/dtpd).

Ultrasonic spectroscopy technique shows new character of graphite

Graphite consists of layers of graphene and the layers are bonded via weak van der Waals (vdW) forces, a ubiquitous attraction between all molecules. It was believed that the elastic constant of graphite crystal did not exceed 40 GPa. This is because the elastic constants obtained from experiments using artificial highly oriented pyrolytic graphite (HOPG) were low due to structural defects in the graphite (see bottom of Figure) and theoretical calculations also demonstrated that the elastic constant of graphite was less than 39 GPa.

Since a direct characteristic of an interplanar interaction is the elastic constant along the c axis of graphite, which reflects the interlayer bond strength, the elastic constant of graphite has been used to validate proposed theoretical approaches, and its accurate measurement is critical to thoroughly understanding vdW interactions. In this study, Kaneka Corporation created a high-quality defect-free monocrystalline graphite by heating high orientation polyimide thin films at high temperatures; however, it was very difficult to measure the elastic constant of this crystal (10 µm in diameter, 1 µm in thickness) along the thickness direction. So, this group experimentally obtained the elastic constant of graphite using picosecond laser ultrasound spectroscopy. They applied a laser of 1 µm in diameter to the surface of a multilayered graphene for one 10 trillionth of a second to generate ultra-high frequency ultrasound. By accurately measuring the longitudinal wave sound velocity along the thickness direction, they obtained the elastic constant.

Although it had been thought that the interplanar bond strength of graphite



Cross-section scanning electron micrography images of specimen G2800 (top) and a highly oriented pyrolytic graphite (HOPG) specimen (bottom). Credit: Kaneka Corporation, Physical Review Materials (2020).

was very weak, the results of this study showed that it had a strong bond strength: the elastic constant was nearly 50 GPa, which cannot be explained by conventional theories. In this study, the short-range correlation effect selectively strengthened the potential energy surface (PES). This anharmonic PES enhanced the elastic constant of graphite. Using the ACFDT-RPA+U method, they demonstrated that the elastic constant reached 50 GPa due to the short-range correlation effect.

Lead author Kusakabe Koichi says, "Our research group shows that graphite exhibits its superiority in a highly crystalline state. We have created high-quality, high-crystallinity graphite, which has stronger interplanar bond strength than previously believed. Applying ultrasonic measurement techniques to this defect-free monocrystalline graphite thin film will lead to the production of highly sensitive sensors for identifying biological matter such as proteins in non-destructive testing."

Their research results were published in *Physical Review Materials* (http://doi.org/dwgc).

ARTICLE

^{138}La as a useful isotope for gamma spectroscopy without radioactive sources

Marco Bortoluzzi* and Valentina Ferraro
Dipartimento di Scienze Molecolari e Nanosistemi, Università Ca' Foscari Venezia, Via Torino 155, 30172 Mestre (VE), Italy
Contacts
markos@unive.it (0000-0002-4259-1027)
valentina.ferraro@unive.it (0000-0003-2694-2955)

Introduction

Radioactivity is inescapably present in everyday life.[1] Radiochemistry is a topic of significant interest, strongly connected with energy production, medicine and environmental protection. Experiences concerning gamma spectroscopy in Inorganic and Physical Chemistry undergraduate laboratories are, however, hardly widespread, because of obvious problems related to students' safety and the storage of radioactive sources.[2] Basic knowledge about radioactive decays is usually offered by General and Inorganic Chemistry courses, but the information is not supported by hands-on learning. Safe laboratory experiments could give insight into several aspects related to radioactivity, such as naturally occurring radioisotopes, decay modes and lifetimes, interactions of radiation with matter and detection techniques. The idea of carrying out undergraduate gamma spectroscopy experiments with elements commonly classified as non-radioactive strongly limits the choice of possible samples. Lanthanum is particularly interesting for such a purpose because of the naturally occurring ^{138}La isotope.

The ^{138}La radioactive isotope constitutes 0.0902% of naturally occurring lanthanum. The half-life ($t_{1/2}$) of this isotope is very long, 1.05×10^{11} years. ^{138}La disintegration is due to two parallel processes. 34% of the isotope undergoes β^- decay, with the conversion of a neutron into a proton, turning into the stable ^{138}Ce. The process is associated with the emission of a 788.7 keV gamma photon (Equation 1). The remaining 66% of ^{138}La disintegrates by electronic capture (EC), i.e. the conversion of a proton to a neutron by interaction with an electron. The EC process leads to the formation of stable ^{138}Ba with the emission of a 1435.8 keV gamma photon (Equation 2).[3] The EC and β^- processes also produce neutrinos (ν_e) and antineutrinos ($\bar{\nu}_e$).[4]

$$^{138}\mathrm{La} \xrightarrow{\beta^-} {}^{138}\mathrm{Ce} + \gamma(788.7\ \mathrm{keV}) + \bar{\nu}_e, \quad 34\% \qquad (1)$$

$$^{138}\mathrm{La} \xrightarrow{\mathrm{EC}} {}^{138}\mathrm{Ba} + \gamma(1435.8\ \mathrm{keV}) + \nu_e, \quad 66\% \qquad (2)$$

The two emissions from ^{138}La are comparable with those of other radioactive isotopes present in the environment. For example, the gamma photon emitted by the EC decay of ^{40}K to ^{40}Ar has 1460.8 keV energy.[5] ^{137}Cs undergoes β^- decay with formation of the metastable ^{137m}Ba. The subsequent decay to the nuclear ground state releases a 661.7 keV gamma photon.[6] The gamma photons from ^{138}La have higher energy if compared to those emitted by refined samples of ^{238}U. In this case, the radioactivity is associated with the α decay of the isotope, which leads to the formation of ^{234}Th. The latter transforms by β^- decay in ^{234}Pa and the process is associated with the emission of gamma photons having 63 keV and 92 keV energy.[7] To better understand the energy scale, just consider that, according to the relationship $E=mc^2$, the mass of an electron is equal to 511 keV.

Scintillation detectors

Gamma photons emitted by ^{138}La can be detected by an instrument known as a scintillator or scintillation detector. A crystal of suitable material can emit a weak flash consisting of photons in the visible–ultraviolet range if hit by a gamma photon. The number of photons constituting the flash is proportional to the energy of the incident gamma photon. One of the most popular materials for scintillator crystals is thallium-activated sodium iodide, NaI(Tl).[8]

Gamma photons weakly interact with matter. For example, the intensity of a 1000 keV gamma photon beam is halved by a lead shield of 0.87 cm thickness.[4] At a first approximation, the absorption of gamma radiation by a material decreases with increasing energy. The scintillator crystal must be relatively large to detect high-energy gamma photons such as those from ^{138}La. The efficiency is, however, limited to few percentage points.

A photocathode coupled to the crystal emits electrons when hit by light, proportionally to the number of photons in the flash and, therefore, to the energy of the incident gamma photon. The electrons emitted by the photocathode can be amplified by a photomultiplier

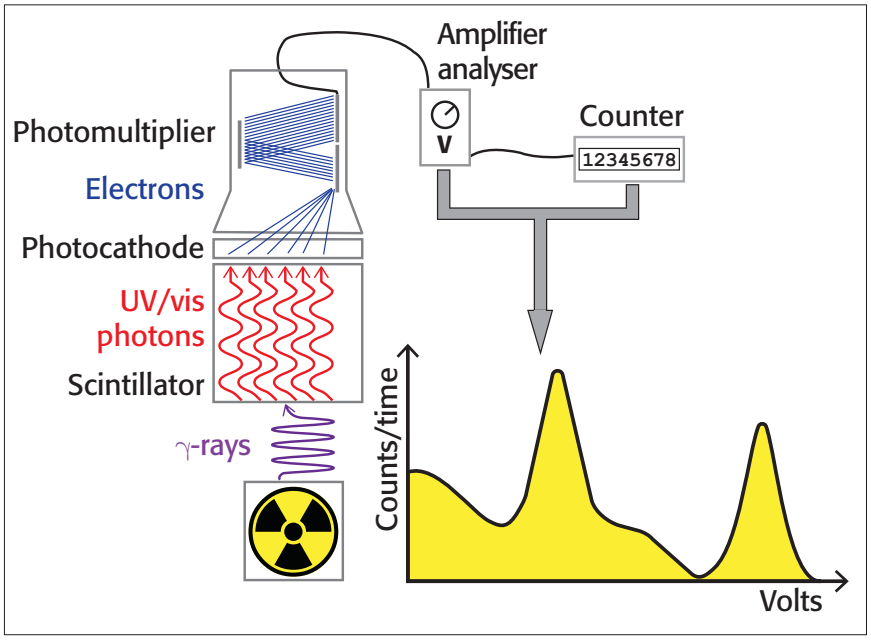


Figure 1. Schematic representation of a scintillation detector.

tube (PMT), thus obtaining a detectable electrical impulse. The amplitude of the impulse, expressed in volts, is linearly proportional to the energy of the gamma photon. The number of electrical impulses in a given time interval can be measured with a counter. With an amplifier/pulse height analyser, it is possible to select the range of pulse amplitudes (in volts) to be counted and build a gamma spectrum with the pulse amplitudes in the abscissa, while the number of counts is reported in the ordinate. The main components of a scintillation detector are depicted in Figure 1.

If the energy of the gamma photons is completely absorbed by the scintillator, the corresponding photopeak is observed in the spectrum. The energy resolution of the photopeak is defined as the ratio between the half-height width and the peak energy. The ratio decreases, i.e. the energy resolution improves, on increasing the photon energy.[8]

The incomplete absorption of energy by the crystal involves the presence in the spectrum of a continuum of signals with energy lower than the photopeak. Gamma photons can impact the electrons of the material by transferring part of their energy, thus obtaining a new photon with lower energy and different direction (Compton scattering).[4] The photon generated after the collision can exit the crystal and, in this case, only the energy transferred to the electron will be counted. It can also happen that the gamma photon first hits the material surrounding the source (e.g. a lead shield), transferring part of its energy to it, and the new photon thus generated can interact with the scintillator. The photon energy generated by the Compton scattering, E'_{γ}, depends on the energy of the incident photon E_{γ} and the angle θ between the two photons, as shown in Equation 3. In the equation, m is the mass of the electron and mc^2 is 511 keV. The effects of Compton scattering are depicted in Figure 2.

$$E'_{\gamma} = \frac{E_{\gamma}}{1+\left(\frac{E_{\gamma}}{mc^2}\right)(1-\cos\theta)} \quad (3)$$

E'_{γ} coincides to E_{γ} if the angle θ is zero, while the minimum E'_{γ} value is for θ = 180°. As a consequence, in the presence of Compton scattering the largest energy transfer to the scintillator occurs when the γ photon hits the scintillator and a γ′ photon exits the crystal at 180° with respect to the first one (Compton edge). For example, if E_{γ} is 1435.8 keV, E'_{γ} is 216.9 keV and the energy measured by the detector is 1218.9 keV (1435.8 – 216.9 keV). On the other hand, if the γ photon is emitted from the source in the opposite direction with respect to the crystal, the impact with the shielding can produce a photon at 180° with respect to the incident one and the detector could count the 216.9 keV γ′ photon.

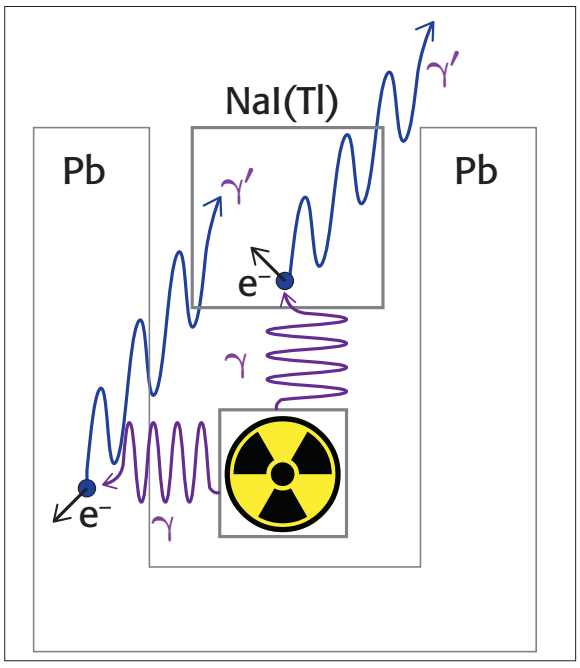


Figure 2. Effects of the Compton scattering on the signals received by the scintillation detector.

Another phenomenon that can lead to the presence in the gamma spectrum of signals at lower energies with respect to the photopeak is pair production, which must be taken into account only for gamma photons having energy greater than 1022 keV.[4,8] It is worth noting that the mass of an electron (or positron) corresponds to 511 keV. Given the mass–energy equivalence, a gamma photon with energy greater than 1022 keV can interact with matter behaving like an electron–positron pair and a residual amount of energy. For example, a 1435.8 keV gamma photon corresponds to an electron–positron pair plus 413.8 keV. The positron interacts with an electron of the scintillator crystal, leading to annihilation and to the formation of two new γ′ photons having energies equal to 511 keV, propagating in opposite directions. The electron consumed by annihilation is replaced in the total balance by that deriving from the incident photon, while the residual energy can be absorbed and counted. If the two 511 keV photons are absorbed by the crystal, then all the energy of the incident photon is detected, increasing the intensity of the main photopeak. If one of the two 511 keV photons escapes from the crystal, the detector instead measures an energy equal to (E_γ – 511) keV, known as a single escape peak. If both 511 keV photons leave the crystal, the energy detected is (E_γ – 1022) keV, corresponding to the double escape peak. The intensities of the single and double escape peaks depend on the size of the crystal used. The process of pair production followed by annihilation can also involve the shielding surrounding the experimental apparatus, and one of the two 511 keV photons can be subsequently counted by the detector (annihilation peak).

Finally, it is worth noting that the electronic structure of the materials undergoes major alterations due to interaction with gamma photons. For example, it is possible to observe peaks in the 75–85 keV range associated with X-rays deriving from transitions in the innermost electronic shell of the lead atoms constituting the shield. Furthermore, if NaI(Tl) is used as a scintillator crystal and the resolution of the spectrum is sufficient, a second peak having 28 keV less energy may appear close to the main photopeak due to the escape from the crystal of the X-photon associated with an internal electronic transition of iodine.[9]

Lanthanum gamma spectrum

In our experiments, a cylindrical NaI(Tl) crystal with diameter and height of 7.6 cm was used, protected by an aluminium layer, with an integrated photodiode and photomultiplier. The declared efficiency is around 2% for 500 keV photons and drops to about 1.3% for energy values around 2000 keV. The crystal produces approximately 10^4 photons/MeV of absorbed energy (wavelength between 300 nm and 500 nm). The scintillator photomultiplier was connected to a preamplifier (Tennelec TC155A), itself connected to a high-voltage power supply (Ortec 556, set at 670V) and an amplifier/single channel analyser (AMP/SCA, Tennelec TC 246). The amplifier was initially calibrated in order to make the spectrum fall in a suitable potential range. The analyser was finally connected to a counter (Ortec 996).

Gamma emission from 257.9 g of lanthanum oxide (La_2O_3, CAS number 1312-81-8, purchased from Strem Chemicals) with natural isotopic abundance was investigated. On considering the ^{138}La percentage in nature, the quantity of ^{138}La in the sample is 1.4 mmol, corresponding to 8.6×10^{20} nuclei (N_0). The radioactive decay follows first-order kinetics, so the activity of the sample ($-dN/dt$) is given by Equation 4:

$$-\frac{dN}{dt} = \frac{\ln 2}{t_{1/2}} N_0 \qquad (4)$$

The activity of the sample considered here is around 180 Bq (Becquerel, disintegrations/second). Another common measurement unit is the Curie (Ci), corresponding to the number of disintegrations per second of one gram of ^{226}Ra ($1\,Ci = 3.7 \times 10^{10}$ Bq). The activity of the La_2O_3 sample is, therefore, around 5×10^{-3} μCi. The sample has very low activity, considering that a radioactive source for didactic purposes is typically around 1 μCi. To detect ^{138}La radioactivity it is, therefore, necessary to shield the source and the scintillator from the natural radioactivity of the environment. In our experiment the La_2O_3 powder was placed in a polyethylene glass with a diameter of 7.1 cm. The height of the powder was 8.2 cm. The glass was introduced in a lead well with internal diameter of 8.6 cm and depth of 11.3 cm. The thickness of the walls was between 3.8 cm and 5.4 cm. The scintillator crystal was placed over the well and the side wall of the crystal was shielded with 3.1-cm-thick lead. An additional lead screen with thickness around 1.2 cm was placed around the PMT.

The gamma spectrum of La_2O_3 reported in Figure 3 was recorded by varying the centre of the SCA potential window with 0.1V steps. The potential window was set to 0.1V. The counter time was 180 min. Every measurement was carried with and without the sample in order to subtract the background. The error associated with the ordinate values was conservatively estimated by the sum of the square roots of the two counts. The inset of Figure 3 was obtained by reducing the potential steps and window to 0.05V, while the counter time was increased to 420 min.

The gamma spectrum shown in Figure 3 highlights the presence of two main peaks, centred at 8.8V and 4.9V, corresponding to the gamma emissions at 1435.8 keV (EC decay) and 788.7 keV (β^- decay) of ^{138}La. The accuracy of the assignment was verified by replacing the sample with ^{40}K (350.6 g of non-enriched KCl, gamma activity 17×10^{-3} μCi, E_γ = 1460.8 keV). On the basis of these data it was possible to roughly calibrate the abscissa scale and report it in keV. The energy resolution of the higher energy peak is around 5%, while for the other peak it is 9%. Because of the different sensitivity of the detector on changing the gamma photon energy, the intensities of the two peaks do not respect the relative probabilities of ^{138}La EC and β^- decays. The two peaks are accompanied by the expected Compton scattering. Probably because of the poor activity of the source, no signal related to pair production was detected. The higher energy peak shows a shoulder at lower energy, but the number of points in the

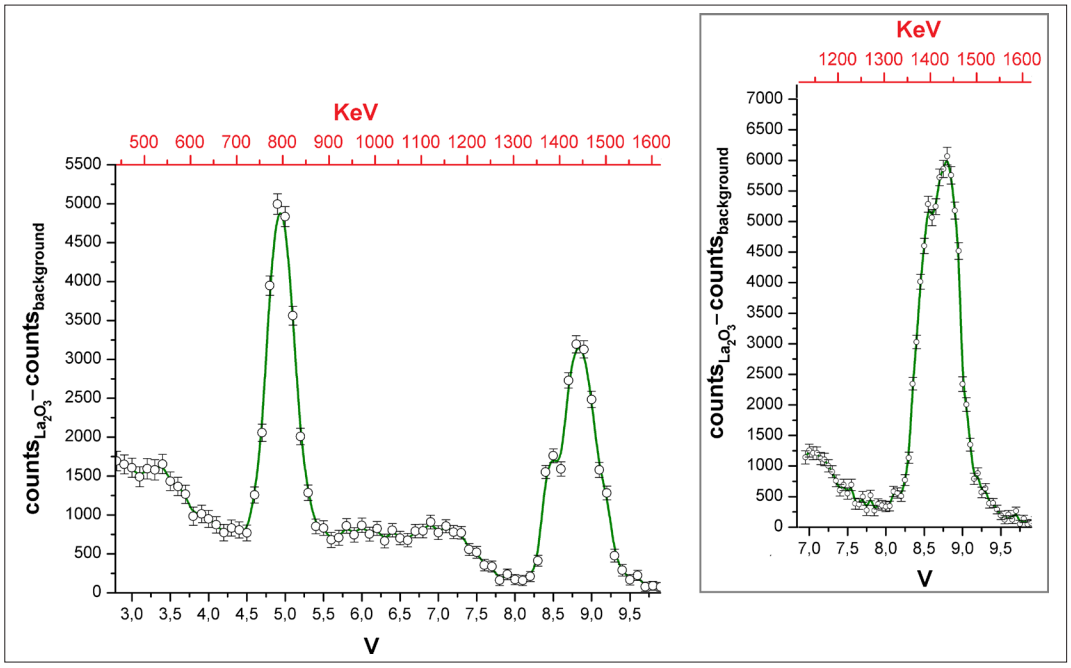


Figure 3. Gamma spectrum of La_2O_3. Inset: portion of the spectrum collected with improved resolution.

spectrum is limited and the points are separated by about 17keV. To verify the existence of the shoulder, that region of the spectrum was collected again with doubled abscissa resolution and longer counting time (see the inset of Figure 3). The shoulder was confirmed, and its energy suggests that it could be related to the escape of an iodine X-photon.

Undergraduate laboratory experiments

The experiment previously described with a SCA analyser requires too much time to be carried out in an undergraduate laboratory. On the basis of the results described, a gamma spectrum with sufficient resolution can, however, be collected overnight using a commercially available multi-channel analyser. If only a SCA is accessible, the gamma activity of the sample in the 450–1600 keV range can be unambiguously distinguished from the background in two hours (one hour measurement for the sample, another one for the background).

Besides La_2O_3, another source that can be safely used by students with the same experimental setup is a potassium salt such as KCl, in order to detect the gamma emission related to ^{40}K EC decay. A useful exercise related to ^{40}K is the calculation of the gamma activity of a potassium source (see Exercise 1). KCl can be used by the students for the rough calibration of the amplifier (see Exercise 2).

In all the cases, it is worth noting that there are no safety hazards related to radioactivity, and La_2O_3 and KCl are non-toxic powders.

Exercise 1

Consider a source constituted by *m* grams of a potassium salt having formula weight *W*. Search on the web for ^{40}K data concerning half-life, natural abundance and relative probability of the EC decay. Use these data and Equation 4 to calculate the gamma activity in Bq and μCi.

Exercise 2

The potential range to collect the gamma spectrum is determined by the high-voltage supply of the PMT and the setting of the amplifier. A rule of thumb for most phototubes is that a 10% variation of the high-voltage will change the gain by a factor of two. To calibrate the amplifier use more than 300g of KCl as source and set a counting time around 5min. Consider a sufficiently large potential window, e.g. 2V. Depending upon the working range of the analyser, set the centre of the potential window at a suitable value (e.g. 8V if the analyser works in the 0–10V range). Start the first measurement with low voltage supply and good amplification, for example use half of the maximum values for both the settings. Record the counts and repeat the measurement by progressively increasing (about 10% each time) the high-voltage supply. Do not exceed the PMT limit! Counts vs high-voltage supply should show a maximum. Starting from the corresponding high-voltage value, halve the amplification and carry out a new series of measurements, increasing the high-voltage supply by about 2–3% each time. Find the new high-voltage value corresponding to the maximum counts and repeat the procedure until restricted by the PMT and amplifier limits.

Conclusions

Gamma spectroscopy on non-enriched sources is a powerful tool for safely exploring several concepts related to radioactivity, in particular:

- Basic concepts: decay modes, radioisotope half-life, units in radioactivity, activity of a source.

continued on page 19

ARTICLE

Letter to the Editor: Importance of citing full details of spectral library and search program

O. David Sparkman
Adjunct Professor of Chemistry and Director of the Pacific Mass Spectrometry Facility, Chemistry Department, University of the Pacific, Stockton, CA, USA. E-mail: osparkma@pacific.edu

A collection of mass spectra of known compounds and the program used to search a spectrum of an unidentified compound are two different entities. The results obtained depend on both. Two different compounds can be found as the first Hit when a search of the same spectrum is performed using the NIST Mass Spectral (MS) Search Program (National Institute of Standards and Technology, Gaithersburg, MD, USA) to search the NIST/EPA/NIH EI Mass Spectral Library (NIST 20) or the *Wiley Registry of Mass Spectral Data*, 12th Edn (John Wiley & Sons, Hoboken, NJ, USA). Two different compounds can be found as the first Hit when the NIST/EPA/NIH EI Mass Spectral Library (NIST 20) is searched using the NIST MS Search Program v.2.4 or the Probability Based Matching (PBM) search program, which is a part of the Agilent Technologies (Santa Clara, CA, USA) ChemStation Data Analysis program. Even when the same mass spectrum is searched against two different versions of the NIST/EPA/NIH EI Mass Spectral Library (NIST 14 and NIST 20) using the same search program (NIST 20's MS Search v.2.4) two different compounds can be found as the first Hit.

There is not one root cause for these differences. In the example of a difference between the search of the NIST 14 and NIST 20 EI libraries using the same search program, it can be as simple as the addition of the spectrum of the unidentified compound to the library between editions. Spectra for approximately ~60 K new compounds were added between these two releases. Or, the different first Hits could be due to complex search algorithm differences in the case of the NIST MS Search program vs the PBM search of ChemStation.

The point is that it is not only necessary to be specific about the publisher of the library but also the edition of the mass spectral library as well as the search program used. Because of my involvement with NIST, I have subscribed to two search strings in Google Scholar Alerts since 5 October 2014 ([NIST Mass Spectral Database] and [nist "mass [spectral | spectra | spectrum]"]). During this period, I have received list of citations for both of these strings every two to five days. Each list contains 5–20 citations to articles that make mention of one of the two NIST Mass Spectral Libraries (NIST/EPA/NIH Library of EI Spectra or the NIST Tandem Library of Production Mass Spectra). Many of these citations also reference the *Wiley Registry* and sometimes other smaller libraries like the flavor and fragrance EI library of Robert P. Adams (Diablo Analytical, Antioch, CA, USA), the Maurer/Pfleger/Weber *Mass Spectral Library of Drugs/Poisons/Pesticides/Pollutants and Their Metabolites*, 2011 Edn (John Wiley & Sons), the Designer Drugs 2020 EI mass spectral library by Peter Rösner (John Wiley & Sons), SWGDRUG *Mass Spectral Library* etc., to mention just some of the many small mass spectral libraries.

Too often citations to the search of a mass spectral library are simply. "The identity of unknowns was confirmed using the NIST library." Unfortunately, not more than 20% of the articles cite the version (edition) of the mass spectral library, and nearly every article is missing any mention of the program used to search the library. When someone purchases an instrument, they will purchase it with a mass spectral library. Today, this is truer of gas chromatography/mass spectrometry (GC/MS) instruments than tandem mass spectrometers used with liquid chromatography, but with the introduction of the NIST Tandem Library and the fact that ThermoFisher is providing a copy of that Library with every tandem instrument that they sell, this will change.

It is also unfortunate that people are publishing an article in 2020 in which the NIST EI library released in 1998 is being used.

As these instrument age, the researchers using them, especially those that are not mass spectrometrists, don't realise that easily and inexpensive upgradable tools, like libraries of mass spectra and search programs that can greatly expand their existing instrument's usefulness, are available.

The main reason journal article authors need to include not only the name and edition of the mass spectral library

used to confirm or identify unidentified compounds but also which programs were used to search these libraries, is so their reader can evaluate the validity of the results.

It is not enough to say, "The identity of the components was confirmed by searching their mass spectra, separately, against the NIST and Wiley libraries". That sentence should read, "The identity of the components was confirmed by searching their mass spectra, separately against the NIST 17 and Wiley 8 libraries using the internal library search algorithm for the Shimadzu GC/MS Solutions V.4.5 software". (The Shimadzu GC/MS Solutions software, like that of most other GC/MS Data Systems, allows only one mass spectral library at a time to be searched.) PerkinElmer, Waters, Agilent's ChemStation (GC/MS and LC/MS) and Agilent's MassHunter (Qual and Quan), Sciex, Bruker and others all have their completely separate proprietary library search routines even through many provide the NIST MS Search Program and libraries in the MS Search format as well as their proprietary format. These proprietary programs and formats can change with changing version of their instrument's software that they accompany; this is why the version number of the data analysis software should be a part of the citation. With the availability of both the NIST MS Search and other third-party search programs as well as the software's priority search, it is absolutely mandatory that the search software be specified.

It should be remembered that the NIST MS Search Program has both an Identity Search (to be used when it is suspected that a spectrum of the unidentified compound is in the libraries being searched) and a Similarity Search (to be used when it is suspected that there is no spectrum of the unidentified compound in the searched libraries). In the NIST MS Search Program (v.2.4), accompanying NIST 20, there are four different Identity Searches (EI Normal, EI Quick, MS/MS and In-Source HiRes) and five different types of Similarity Searches (EI Hybrid, EI Simple, EI Neutral Loss, MS/MS in EI and MS/MS Hybrid, for use with product-ion mass spectra from atmospheric pressure ionisation produced precursor-ions). Therefore, it is best that not only the version being used is stated but, also, which algorithm is being used when reporting the use of the NIST MS Search Program. Another unique feature of the NIST MS Search, unlike the search routines provided as part of most data analyses' software routines, is that up to 127 different libraries can be searched, simultaneously. Hits are listed according to quality, not according to search order.

The following are examples of proper citations to use when working with one of the two NIST Mass Spectral Libraries and the NIST MS Search Program.

Search of an electron ionisation spectrum

Identification of an unidentified compound's mass spectrum was accomplished using the NIST Mass Spectral Search Program's, v.2.4, EI Normal Identity Search of the NIST 20 NIST/EPA/NIH EI Mass Spectral Library (mainlib and/or replib) and [name(s) of any other library(ies) searched]. If any Search Constraints were used, these should also be listed; especially if the Retention Index Database was used.

Search of a product-ion mass spectrum

Search of the NIST 20 Tandem Library (hr_msms_nist, lr_msms_nist, apci_msms_nist or (biopep_msms_nist) and [name(s) of any other product-ion mass spectral library(ies) searched] was done using the NIST MS Search Program's (v.2.4) MS/MS Hybrid Similarity Search. If any Search Constraints were used, these should also be listed.

Again, it can't be over emphasised that the library publisher, library edition and software used should be cited in detail.

This Letter to the Editor is being submitted to multiple journals in order to obtain as much coverage as possible on this topic.

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continued form page 17

- Advanced concepts: gamma photons, interaction with matter, Compton scattering.

The measurements suggested here give insight into questions related to natural radioactivity in the environment. The instrumental equipment offers the possibility to work with PMTs, amplifiers and analysers that may also be encountered during other undergraduate laboratory experiments, such as those related to luminescence measurements.

Acknowledgements

Università Ca' Foscari Venezia is gratefully acknowledged for financial support. We sincerely thank Enrico Trave and Paolo Calvelli for support.

References

1. S.G. Hutchison and F.I. Hutchison, "Radioactivity in everyday life", *J. Chem. Educ.* **74,** 501–505 (1997). https://doi.org/10.1021/ed074p501
2. H. Domenech, *Radiation Safety*. Springer, Basel (2017).
3. *Table of Radioactive Isotopes*. http://nucleardata.nuclear.lu.se/toi/nuclide.asp?iZA=570138 (accessed April 2020).
4. W.D. Loveland, D.J. Morrissey and G.T. Seaborg, *Modern Nuclear Chemistry*, 1st Edn. Wiley, Hoboken (2006).
5. *Table of Radioactive Isotopes*. http://nucleardata.nuclear.lu.se/toi/nuclide.asp?iZA=190040 (accessed April 2020).
6. *Table of Radioactive Isotopes*. http://nucleardata.nuclear.lu.se/toi/nuclide.asp?iZA=560437 (accessed April 2020).
7. F. El-Daoushy and F. Hernández, "Gamma spectrometry of ^{234}Th (^{238}U) in environmental samples", *Analyst* **127,** 981–989 (2002). https://doi.org/10.1039/b203012h
8. G.R. Gilmore, *Practical Gamma-ray Spectrometry*, 2nd Edn. Wiley, Chichester (2008).
9. J. Prekeges, *Nuclear Medicine Instrumentation*. Jones & Bartlett Publishers, Sudbury (2010).

TONY DAVIES COLUMN

COVID-19 #2: Compliant data processing from your home office

Antony N. Davies[a] and Mohan Cashyap[b]
[a]SERC, Sustainable Environment Research Centre, Faculty of Computing, Engineering and Science, University of South Wales, UK
[bc]MASS Informatics, Harpenden, UK

This column continues our theme of supporting working whilst unable to freely or safely access the analytical laboratory. We want to look at what advances have been made in systems allowing spectroscopic data processing from your home office. This has always caused particular problems for those working in highly regulated environments, such as the pharmaceutical industry, and their supplier and support contractors.

Definitions of Open and Closed systems, blockchain

In general, regulated industries have tried to avoid their IT environments falling into the "Open" category due to the increased requirements to ensure data is not capable of being tampered with. Within a well-protected company network this should not be a problem, as they are classical "Closed" environments under the definition. "Closed system means an environment in which system access is controlled by persons who are responsible for the content of electronic records that are on the system." In contrast, cloud provision can fall under the Open system definition. "Open system means an environment in which system access is not controlled by persons who are responsible for the content of electronic records that are on the system."[1]

Now, if you refer to the introductory columns on use of Cloud Computing,[2,3] we discussed various types of Cloud systems which were more or less likely to be able to meet compliance criteria. The FDA Open system adds additional burdensome requirements on the IT infrastructure and software solutions, such as encryption of the data (not just when in transit) and full electronic signatures. But the problems don't stop there, as the requirements for full training records for all systems staff to prove they are GxP compliant and up-to-date doesn't vanish when you outsource your IT infrastructure in an Open system… it just transfers the responsibilities to your external host/provider.

The big cloud hosting organisations claim to have regulatory compliant offerings, but if you approach them you need to know exactly what your strategy will be. For regulatory inspectors, the focus is more and more on data integrity. Who has had access to what and what did they do in your compliant environment is key to demonstrating data integrity and that your security features have been correctly installed and are operating fit-for-purpose. Where some cloud providers have issues is in making their system audit trails open for inspection, so this is a key area you, as customer, need to ensure you have what you need.

Advances in blockchain technologies is one area where we may be able to steal innovations driven by other sectors. Our requirements on automated audit trails cover all actions mandated for audit and signoff by our different regulators. These must remain secure even when data moves outside our immediate internal IT environment, a common critical underlying functionality in blockchain systems. Here the chain of data and the audit trail can be proven to be tamper-proof. But to exploit this we need not only better software systems but also improvements in our hardware environments to ensure no data leakage. Things to consider, for example, when allowing remote working include ensuring no password sharing, IP tracking can easily be spoofed if you want to beat the system, so you need better systems to detect data leakage, maybe something like the anomaly detection capabilities used by banks with similar problems.

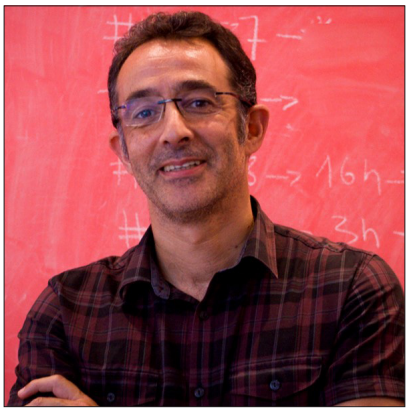
One of the essential tools for ensuring better data integrity is extensive automation of the data transmission and processing functions. This also leads to opportunities to support the work of the Quality Person in a regulated environment through the deployment of some levels of artificial intelligence to support the checking of, for example, study documentation. Scanning documentation for "obvious" or formal errors such as unlikely, incorrect or missing date, out-of-tolerance results, missing fields etc. can easily be automated. This does not replace the work of the Quality Person, but assists them by focusing their work on the anomalies in documentation they would normally have to find themselves. Taking this a step further, you can imagine working on the supporting analytical data and, for example, being able to identify where two spectra were so "identical" as to effectively mean it was impossible that they had come from two

separate QC measurements; thereby flagging possible errors or attempts at fraud.

Deployment examples of compliant cloud solutions

So where are we in terms of moving our systems safely into a cloud environment? Santi Dominguez, CEO of MestreLab, had some interesting comments which also reach back into the previous column[4] on making use of the extra time we have working from home to upskill: they have been running a series of additional free training workshops.[5] They saw that many people taking part were already using their software but were looking for additional training in more advanced spectroscopic data processing in areas they did not currently exploit. After years of this column complaining that the chromatographers are well ahead of the spectroscopists in the advanced level of IT system support with their chromatography data systems (CDS), it was interesting and nice to hear from him a feeling of obligation to develop similar advanced data handling and analytical workflow oriented support for spectroscopists…

"At Mestrelab we have been moving towards allowing our users to work remotely and freeing them from geographical restrictions. We see this access to data anytime from anywhere as being a critical part of the Lab of the Future. The design of our tools and solutions has had this idea at its heart for several years, and we are either there or getting there with most of the tools. The coronavirus pandemic has illustrated this by making remote work compulsory rather than desirable, and the amazing attendance we have had to our COVID19 workshops has shown the interest in the community in the value that this geographical flexibility offers."



Santi Dominguez

With LIMS systems being very much focused on standardised procedures or biased to handling chemical structures at their core, it would be great if spectroscopists would finally have a cloud-based enterprise application to support our work. Santi and his colleagues have taken this on board and are producing a system which can automate large parts of the "request>measure>capture_data>retrieve>process>report>archive" workflows we all use. He commented…

"With the technology available today, there is no reason why you should not be able to continue to progress your research and work just because you are travelling, at a conference or because a global pandemic prevents you from going to your workplace. It is up to us, as solution developers, to allow our users to transcend those geographical limitations, and this is at the core of our philosophy as a company."

We also had a really useful discussion with Heather Longden, a former colleague (of TD) who has a role as Senior Marketing Manager at Waters for Pharmaceutical Regulatory Intelligence, is a specialist in compliance to e-record regulations and an active member of ISPE GAMP Community of Practice, where she is called on as an expert in Data Integrity. In light of the working from home challenges today, Heather acknowledged that the Empower Cloud CDS has been adopted by a number of highly regulated laboratories. As Steve Bird, former Director of Informatics Strategic Marketing included in an Amazon Web Services (AWS) whitepaper...

"Users can sign on to Empower Cloud from any online computer or device, inside or outside of their organisation's network, using the same Empower credentials they would use at their desks or in their laboratories. This change significantly enhances their business continuity and data security capabilities while also ensuring their compliance and validation requirements are met."

Heather was very positive about putting scientific data processing systems into the cloud and had some positive stories where the deployment to the cloud used in an IAAS (Infrastructure as a Service) can actually greatly improve the compliance position of a company. Here I must apologise for citing a CDS system, but it does show what is now possible. Clearly there are additional challenges to solve for SaaS (Software as a Service) applications for regulated laboratories, but the IaaS model allows scientists to run dedicated individual single tenant solution on cloud infrastructure. In Waters' case, they have partnered with AWS as a cloud provider, and leverage automated AWS provided scripts to "install" the application, which is more reliable and consistent than an IT expert deploying applications on inhouse developed, on-premise infrastructure.

Heather did point out that when auditing or verifying your cloud providers understanding and delivery of GxP compliance requirement, be prepared to phrase questions in a way that IT provider's understand, discussing security and authentication, consistent installation etc, rather than IQ, OQ PQ, audit trails and data approval or batch release. Key to this is to ensure that you not only have understood the additional risks, and noting the mitigated risk, but that you also have clear documented agreement laying out who is responsible for what.

"... this is what the cloud provider is responsible for…".

She has been looking at the difference between US and European compliance, which is normally very closely aligned. However, an additional requirement in Annex 11 of the European regulations[6] is to "regularly review" audit trails (and consequently to have documented somewhere that this activity has been carried out).

The annex 11 is not a clear departure from Part 11. It explicitly clarified an expectation of both agencies that ALL critical data and meta data is reviewed.

TONY DAVIES COLUMN

Especially during the pandemic, a compliance trap has opened up with vendors being supportive by making additional licenses of their products available to people working from home. Although it might be obvious, just because the software is the same release version as what you have installed on your desktop computer in your laboratory, it will still need to be a validated installation on a validated computer system. So beware of trying to install your scientific data processing software onto the family's ultra-fast gaming PC… you might produce those 2-million datapoint surface plots really quickly, but you will not be able to use the results in a compliant manner!

I would like to finish off with an example Heather cited of the use of the cloud, not just to reduce costs in your IT environment but to exploit it to produce a much stronger compliant position where companies are working with external third parties such as contract research organisations (CROs) or contract manufacturing organisations (CMOs). Here, the contracting organisation uses an IaaS cloud deployment of their own to be the SaaS provider to their subcontracting CROs and CMOs (Figure 1). This reduces the worries about setting up and ensuring rock-solid Chinese walls with your subcontractors especially around data leakage.

Essentially, the subcontractors are carrying out the work for the contracting company in their own laboratories, but the instrumentation is run through the cloud software deployment of the contracting organisation. Again, a clear case where everything must be very well documented, but does eliminate many of the compliance hurdles associated with out-sourcing much of your new product development activities while maintaining an overall strong compliance position. Waters have a funny short video explaining all this much better than we can, which I would recommend watching if you have a spare four minutes.[7]

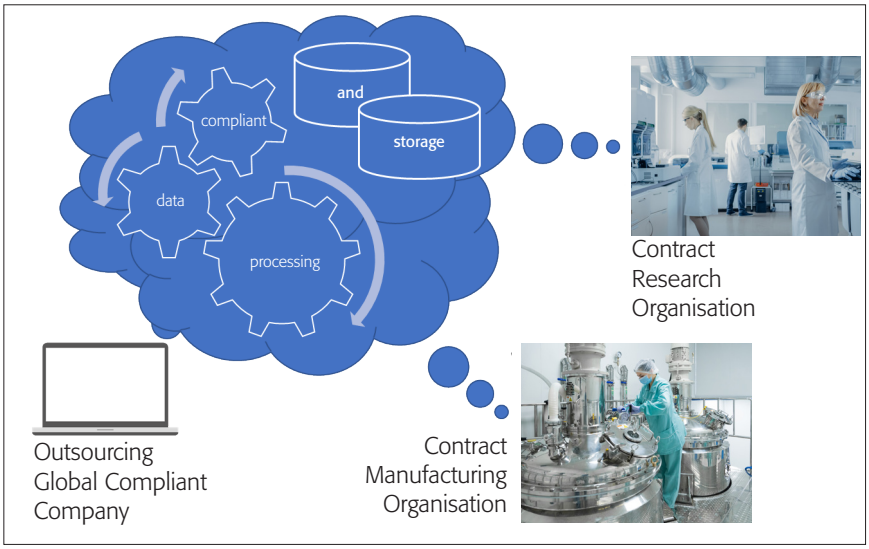


Figure 1. CxO organisations creating data which is acquired directly into the cloud-hosted enterprise application and owned by the outsourcing company.

Conclusions

So, thankfully, it seems that we, as a community, have moved substantially forward since our earlier articles on the introduction of cloud-based solutions. The solutions have addressed the compliance issues and seem to have started to actually deliver more flexible enhanced compliance positions over conventional deployments. If you have any good examples of such innovation yourself, please let us know and we will see if we can feature them in future columns.

Thanks!

Special thanks to Heather Longden at Waters and Santiago Dominguez at MestreLab for some very useful discussions and inspiration when putting this column together!

References

1. *Electronic Code of Federal Regulations, Title 21: Food and Drugs, Part 11—Electronic Records; Electronic Signatures*, e-CFR data is current as of 14 May 2020. https://www.ecfr.gov/cgi-bin/text-idx?SID=10b1b35c3a3ca71cf54d46dde50ab704&mc=true&node=pt21.1.11&rgn=div5
2. A.N. Davies and M. Cashyap, "A head in the clouds?—Part one", *Spectroscopy Europe* **26(3),** 21–22 (2014). https://www.spectroscopyeurope.com/td-column/head-clouds-part-one
3. A.N. Davies and M. Cashyap, "Who's ahead in the clouds?—Part three", *Spectroscopy Europe* **27(1),** 18–21 (2015). https://www.spectroscopyeurope.com/td-column/whos-ahead-cloud-part-three
4. A.N. Davies and H.-J. van Manen, "COVID-19: Lock-down and up-skill", *Spectroscopy Europe* **30(2),** 19–21 (2020). https://www.spectroscopyeurope.com/td-column/covid-19-lock-down-and-skill
5. COVID-19 Mestrelab workshops: https://resources.mestrelab.com/webinars/
6. EudraLex, *The Rules Governing Medicinal Products in the European Union, Volume 4, Good Manufacturing Practice, Medicinal Products for Human and Veterinary Use*. Annex 11: Computerised Systems (2010). https://ec.europa.eu/health/sites/health/files/files/eudralex/vol-4/annex11_01-2011_en.pdf
7. https://www.waters.com/waters/en_US/Empower-Cloud-with-AWS/nav.htm?locale=101&cid=134950285

SAMPLING COLUMN

Theory of Sampling—an approach to representativity offering front line companies added value and potential substantial savings

Fritz Rendeman,[a] Jørgen Riis Pedersen[b] and Kim H. Esbensen[c]
[a]Scion-DTU, Diplomvej 373, Ø2, 2800 Kgs. Lyngby, Denmark. fritz@rendemann.dk, www.intoscandinavia.com, www.rendemann.dk
[b]Business Consultant, Sense Leadership Coaching. jriispedersen@gmail.com
[c]Owner, chief consultant, independent researcher at KHE Consulting, Aldersrogade 8, Copenhagen DK-2100, Denmark; guest-, associate-, visiting professor (Denmark, Norway, Puerto Rico, Quebec). khe.consult@gmail.com

Previous Sampling Columns have dominantly focused on the technical issues of representative sampling. This column addresses sampling from the complementary point of view: "What is the economic and commercial impact from non-representative sampling on management decisions and in boardrooms?" We have invited two experienced business consultants to help scope out an outline indicating powerful opportunities for added value and for substantial savings.

In medias res

Sampling is the process of selecting and extracting a small part of material suitable for analysis under the critical demand that it is a guaranteed representation of the much larger original lot.

A lot is the sampling target material residing in, for example a shipload, a railroad carriage, a truckload… or constituting a process flow, a moving stream of matter.

Sampling is a technical operation influencing the validity of decision making: sampling affects the bottom line in major sectors of world trade…

Sampling lies behind a significant number of claims and disputes related to commercial transactions…

Sampling leads to a fundamental uncertainty about material, product or goods characterisation, which, if ignored, may translate into economic consequences in the form of *hidden* value losses…

The principal reason for the above uncertainties is material *heterogeneity*. There are huge adverse risks associated with heterogeneous materials, which must be sampled according to codified procedures that specifically *counteract* heterogeneity in appropriate ways.

Sampling must always be carried out in a *representative* fashion, by a *competent* legal person, i.e. a properly trained sampling technician, process engineer, supervisor, or a certified department, institution or agency.

Sampling matters greatly—from the point of view of providing a reliable

Figure 1. Sampling targets (lots) come in a great variety of shape, form and size, and apparently with no common traits. However, the TOS stipulates how it is only necessary to take their degree of heterogeneity into account.

SAMPLING COLUMN

basis for decision making in all of science, technology, industry and society. Without a minimum of proper knowledge, competence and experience, there is no guarantee that a "repeated sampling operation" will result in a "duplicate sample" with the same analytical result, precisely *because* of material heterogeneity.

It is only the specific sampling process with which a sample was extracted that can be designated as *representative*, or *not*, according to certain criteria which are codified in the Theory of Sampling (TOS). Material extracted without a documented TOS-basis can never be designated as representative samples—only as worthless small lumps of matter without a meaningful provenance w.r.t. the original lot. Such non-representative extracts, "specimens", must be discriminated against and never relied upon. Specimens are not worth processing in the analytical laboratory, and far less analysed, as their analytical results will be fraught with uncontrollable uncertainties of quite unacceptable magnitudes. Sampling uncertainties are 10–25–50 times *larger* than the traditional analytical uncertainties which are usually the only determinants in contractual specifications. This misunderstanding is the source of many contractual disputes, but which are wholly avoidable and therefore unnecessary. The TOS is the only competence basis with which to address all these issues.[1–4]

Impact on critical decision making

This issue is not always fully recognised and acknowledged, and may not always be passed on to all decision making levels, CEO, boardroom. Yet all decisions with critical economic consequences are made here. If there is a fundamental lack of understanding of the magnitudes of such "hidden" uncertainties, there is a lack of due diligence by those responsible for producing correct and reliable documentation upon which to base management decisions on the operative level, or strategic deliberations at the company board room level. This is why there must be at least a minimum core understanding of such "technical sampling issues" at management and board room level as well.

In a commercial transaction, sampling produces the basis for a *reference* to an agreed product specification, or acts as a quality reference both at pre-shipment inspections as well upon arrival control at the destination. Documented samples are often used as reference material and deposited at the Chamber of Commerce for use in case of legal dispute. Furthermore, if payments of goods are conditioned on a "Letter of Credit" transaction, the sampled reference material (with its certified analytical result) is the decisive factor for payment approval and for the go-ahead process of the shipment in question.

But what if the supposed representative samples are, in fact, worthless specimens?

The contractual apparatus can then be fundamentally undermined with easily imaginable adverse consequences. **Sampling matters greatly**—at all levels in any company, corporation and organisation.

Sampling—minimum technical knowledge

All materials are heterogeneous on one scale or another, it is only a matter of degree. Sample representativity is the imperative criterion that must be honoured in order to draw valid conclusions about the true characteristics of an original lot, of materials, of material processes. The force of the TOS becomes clear when it is realised that the TOS is applicable to *all* types of material; exactly the same principles and practical sampling rules need to be invoked.

Internally in a company, choosing and using *only* correct sampling procedures, and knowing how to choose *only* correctly designed equipment, is a critical facility for being able to reach a desired Quality Control/Quality Assurance (QC/QA) level. Using *only* representative sampling allows a company to optimise the quality of the processing taking place, or of the quality of the products produced. There is a plethora of "hidden" sampling necessary to be able to *document* QC/QA appropriately. Are all companies or other stakeholders fully equipped for this task? The TOS is a critical success factor for proper monitoring of quality.

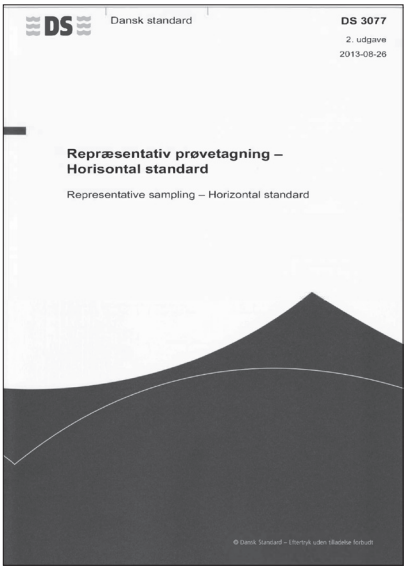


Figure 2. DS3077 (2013): the *de facto* international standard for representative sampling.

Proper sampling and laboratory analysis become critically important competences. There has been a growing historical usage of proper sampling concepts, methods and equipment over the last 50 years, but even today this is far from universally applied. This historical development is fully documented, indeed available at all levels of appreciation.[1–4] First and foremost, there is documentation in the form of a *de facto* international standard devoted to the general principles, equipment design characteristics and, perhaps most important, the necessary and sufficient rules for *practical representative sampling* based exclusively on the TOS.[1]

Summing up—cardinal points

Analysing a non-representative specimen is pointless! If a sample cannot be documented to be representative of the lot/target material from where it was taken, it is a waste of time, effort and money to analyse it.

Erroneous sampling is, every year, responsible for:

- waste in production,
- sub-optimised product quality levels,

Introduction to the Theory and Practice of Sampling

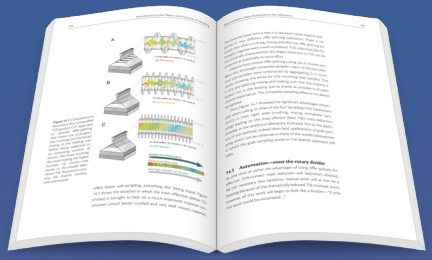
NEW BOOK

Kim H. Esbensen

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

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

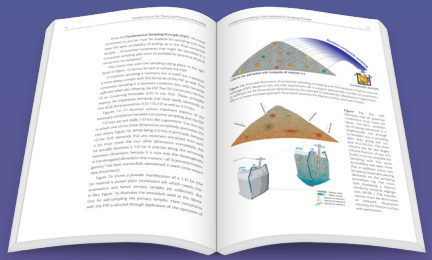
This book presents the Theory and Practice of Sampling (TOS) starting from level zero in a novel didactic framework without excessive mathematics and statistics. The book covers sampling from stationary lots, from moving, dynamic lots (process sampling) and has a vital focus on sampling in the analytical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



"I recommend this book to all newcomers to TOS"

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

"One of the book's major advantages is the lavish use of carefully designed didactic diagrams"



impopen.com/sampling

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- erroneous QC/QA documentation,
- a volume of unwanted complaints, claims, legal disputes and lawsuits,

but all this is largely unnecessary! Non-representative sampling also results in damage to companies' reputations and may, at worst, translate into lost business opportunities.

Sampling, although strictly a technical operation, nevertheless deserves the full attention of management. Ignorance of the TOS results in unacceptably high risks in decision making. If a "sample" does not qualify as representative (in reality a "specimen"), it may be part of the reason for significant production loss, commercial disputes, disruption of a commercial partnerships and, potentially, high costs for third parties too.

The importance of sampling *must* be understood and acknowledged by all stakeholders. The person, department or other entity responsible for sampling *must* possess a minimum of relevant TOS competence, practical know-how, proper training and *integrity*, so as professionally to be able to fulfil this vital role in reaching the quality objectives and contractual obligations committed to at higher levels in a company or corporation. It is necessary to establish an entity with a *unified* responsibility for sampling "from-lot-to-analysis", which has undisputed *carte blanche* to perform the necessary QC/QA of all sampling processes throughout and across all department borders.

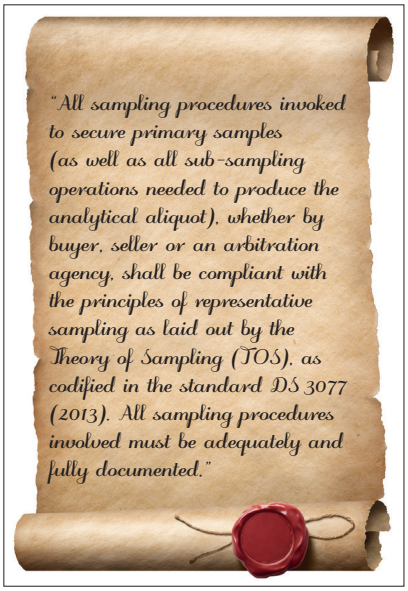


Figure 3. Example of a generic text that would eliminate all sampling vs analysis uncertainties in commercial contracts.

Globalisation and increased volumes of international trade produce a need for relevant rules and regulations. The scenarios depicted here will be the same for all international economic activity levels. From pre-corona prosperity to post-corona recovering economies, all technical and business issues are identical. Therefore, standards and certification bodies have established common guidelines and legal references for cross border transactions and safety regulations.

What has been missing up to now, the *missing link*, is one comprehensive, universal standard for sampling: DS3077 (2013) provides this.[1] A unanimously adopted horizontal standard for representative sampling is the logical guardian, which all stakeholders and parties should be able to agree upon. An example of its deployment in the generic buyer–seller scenario is shown in Figure 3.

References

1. *DS 3077. Representative Sampling—Horizontal Standard*. Danish Standards (2013). http://www.ds.dk
2. K.H. Esbensen, *Introduction to the Theory and Practice of Sampling*. IM Publications Open, Chichester, UK (2020). impopen.com/sampling
3. F.F. Pitard, *The Theory of Sampling and Sampling Practice*, 3rd Edn. CRC Press (2019). ISBN: 978-1-138476486
4. K. Esbensen and G. Lyman, "'Critique of Gy's Sampling Theory"': Misplaced expectations of Wikipedia's democratic intentions", *TOS forum* **1**, 28–30 (2013). https://doi.org/10.1255/tosf.11

Fritz Rendemann
- MBA, IMI, Geneva, Switzerland
- Business Management
- International Business Development
- Corporate Restructuring & Turnaround Management
- Strategic Planning

Jørgen Riis Pedersen
- Ph.D. Sociology, EHESS, University of Paris
- Change Management and Strategy
- Corporate Rightsizing
- Organisational Development

Kim H. Esbensen
- M.Sc. University of Aarhus, Denmark
- PhD Technical Univ. Denmark (DTH)
- Geology, metallurgy, Data Analysis, Chemometrics
- Theory and Practice of Sampling (TOS)
- Science education outreach

PRODUCTS AT ASMS 2020

In response to the COVID-19 pandemic, the American Society for Mass Spectrometry (ASMS) changed their physical annual conference into a combined on-demand and live virtual event.

A number of mass spectrometry manufacturers that use ASMS to launch their latest products have followed ASMS' lead and have held virtual press conferences to announce these. Since the renamed ASMS 2020 Reboot doesn't start until this issue has gone to press, our coverage will be less than complete. However, we have included products announced before our deadline, and plan to include others that we learn about in the next issue.

New Orbitrap high-resolution mass spectrometer

The new Thermo Scientific Orbitrap Exploris 120 mass spectrometer has fast scanning modes and rapid polarity switching. It is compatibile with Thermo Fisher's latest software, including Compound Discoverer 3.2, providing extensive characterisation capabilities, access to multiple mass spectral fragmentation library and structural database sources, and TraceFinder 5.1 software, which simplifies and optimises the high-throughput screening and quantitation of compounds with improved integration of difficult peaks and streamlined reporting.

Thermo Fisher Scientific

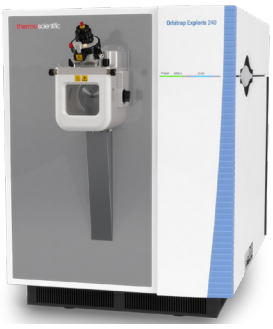
http://link.spectroscopyeurope.com/32-048

New Exploris Orbitrap mass spectrometer

The Thermo Scientific Orbitrap Exploris 240 mass spectrometer expands the Orbitrap Exploris platform with mass accuracy, sensitivity and resolving power across a wide dynamic range. New-generation system architecture and instrument control software provide simple yet powerful data acquisition capabilities and the system offers positive/negative mode switching. It is also compatible with the Thermo Scientific FAIMS Pro interface to achieve enhanced identification of proteins and peptides.

Thermo Fisher Scientific

http://link.spectroscopyeurope.com/32-049



Triple quadrupole LC/MS system

Agilent Technologies have introduced the Agilent 6470B Triple Quadrupole LC/MS (6470 LC/TQ) system, which includes enhancements that boost system uptime and improve overall system performance for low-level detection in routine, high-throughput environments. Customers running a single chromatographic method for applications such as multi-residue screening will see greater data fidelity, reproducibility and sensitivity. The system integrates seamlessly into existing analytical labs and customer workflows—and it offers quicker routine maintenance through the addition of VacShield technology, designed to easily remove ion injectors without venting.

Agilent Technologies

http://link.spectroscopyeurope.com/32-058

High-throughput sample purification and injection for MS

Agilent Technologies have introduced the high-throughput RapidFire 400 system, which provides results with cycle times as short as 2 s/sample and adds optional sample cooling for greater sample stability. The system includes barcode reading capabilities for sample tracking, now supports 1536 well plates and allows unattended operation for the analysis of more than 98,000 samples. It is designed to integrate with the Ultivo LC/TQ system, thereby reducing lab space requirements.

Agilent Technologies

http://link.spectroscopyeurope.com/32-059

MALDI-2 post-ionisation source

Bruker have launched the MALDI-2 post-ionisation (PI) source, which is now available as an option on the timsTOF fleX™ ESI/MALDI mass spectrometer. MALDI-2 technology can offer one or two orders of magnitude higher sensitivity for many small molecules and lipids. MALDI-2 increases the applications range of MALDI mass spectrometry and imaging even further, as well as both the sensitivity and range of applications of MALDI. MALDI-2 requires a second laser (266 nm) fired orthogonally into the expanding MALDI plume that is generated by Bruker's proprietary primary *SmartBeam*™ 3D (355 nm) laser. An optimised *flexMatrix*™ formulation is recommended for MALDI-2.

Bruker now also offers a MALDI-2 compound reference library for its MetaboScape® metabolomics software, which was created during various academic and pharma collaborations.

PRODUCTS AT ASMS 2020

MetaboScape provides automatic analyte annotation within the SCiLS™ Lab MALDI imaging software, including CCS-algorithms that improve the confidence of annotation for many metabolites, glycans and lipids directly in tissue images.
Bruker
http://link.spectroscopyeurope.com/32-061

prm-PASEF mode for translational quantitative 4D proteomics

Bruker's timsTOF™ Pro has been enhanced by combining PASEF® with parallel reaction monitoring (PRM) for label-free quantitative proteomics. This mode of prm-PASEF takes advantage of the fourth dimension of separation using TIMS to improve selectivity and sensitivity, combined with the speed of PASEF to increase the number of precursor targets. Skyline software can now analyse the prm-PASEF data and produce quantitative reports.
Bruker
http://link.spectroscopyeurope.com/32-062

Real-time search on timsTOF Pro 4D data

Bruker has announced the availability of the proteomic pipeline (IP2) with a GPU-based search engine incorporating the ProLuCID database search tool from the laboratory of Professor John Yates at The Scripps Research Institute. Due to the ability of graphical processing units (GPUs) to execute many parallel instruction threads simultaneously, this GPU-based IP2 software allows timsTOF Pro 4D data to be searched in real-time during acquisition, with search results available at the end of the run.
Bruker
http://link.spectroscopyeurope.com/32-063

Ultra-high sample throughput MS system

SCIEX's Echo® mass spectrometry system is now commercially available. The system uses acoustic ejection mass spectrometry with the Open Port Interface for ultra-high sample throughput. It can analyse up to three samples per second and provide quantitative results. This enables projects previously requiring weeks of analysis to be completed in days. Echo also enables high standards of quantitation with consistent and precise reproducibility, even in challenging matrices.
SCIEX
http://link.spectroscopyeurope.com/32-064

High-throughput screening and quantitation software

The high-throughput screening and quantitation of compounds is a crucial process performed in routine analyses across forensic, environmental and food safety laboratories. The new Thermo Scientific TraceFinder 5.1 software helps scientists simplify and optimise quantitation with improved integration of difficult peaks, delivering consistent results between users. The software features the ability to search both a Thermo Fisher library and a custom library in the same method, providing access to an increased number of compounds. Laboratory scientists and technicians also benefit from streamlined reporting, with easily customisable reports that avoid complex formatting and formulae.
Thermo Fisher Scientific
http://link.spectroscopyeurope.com/32-055

GC-MS software for small molecule analysis

The new Thermo Scientific Compound Discoverer 3.2 software is for users of Thermo Scientific Orbitrap GC-MS systems, providing advanced sample characterisation with HRAM GC-MS data for easy identification of biomarkers and other compounds, and the ability to perform statistical analysis with the software's statistical processing engine. Two additional software nodes support chromatographic deconvolution, peak grouping and identification by spectral library search. These new nodes use an automated method for unknown compound identification with both electron ionisation and positive chemical ionisation data on the GC Orbitrap systems.
Thermo Fisher Scientific
http://link.spectroscopyeurope.com/32-051

Mass spectrometry software for development of biopharmaceuticals

The latest Thermo Scientific BioPharma Finder 4.0 software, for scientists in the development of new biopharmaceuticals, introduces an advanced solution for the characterisation of both protein biotherapeutics and oligonucleotide therapeutics, providing enhanced flexibility to biopharmaceutical laboratories. Using a novel MS^2 prediction algorithm, automatic identification and annotation using MS^2 data, the software offers increased confidence in results with reduced operator effort. The updated Sequence Manager supports DNA and RNA and allows users to

PRODUCTS AT ASMS 2020

create their own oligo sequences for a new level of adaptability for a wide range of study.
Thermo Fisher Scientific
http://link.spectroscopyeurope.com/32-053

Thermo Scientific Xcalibur 4.4 software

The Thermo Scientific Xcalibur 4.4 software is for small molecule researchers using the Thermo Scientific Orbitrap Exploris mass spectrometer platform. It provides structural identification and characterisation to automate processes and save user time. The latest upgrade broadens the AcquireX intelligent data acquisition workflow across a wider range of instrumentation. The new software enables the fully automated collection of high-quality MS/MS data on components of interest in a sample, reducing manual input and the need for multiple runs.
Thermo Fisher Scientific
http://link.spectroscopyeurope.com/32-052

Mass spectral data visualisation software

The qualitative analysis of mass spectral data is enhanced with the release of Thermo Scientific FreeStyle 1.7 data review application for Xcalibur software, which enables mass spectrometry users to easily visualise data in multiple formats. The latest version features access to Multiple Mass Defect Filtered chromatograms for a mass and mass defect range, editable isotope abundance and analytics histogram views. The new solution also offers the same familiar interface and easily accessible tools that FreeStyle users are accustomed to.
Thermo Fisher Scientific
http://link.spectroscopyeurope.com/32-054

Ion mobility system

MOBILion Systems has introduced its first ion mobility separations product, which is based on Structures for Lossless Ion Manipulation (SLIM™). The SLIM technology was invented by Dr Richard Smith and has been licenced exclusively to MOBILion Systems. SLIM provides rapid, high-resolution separations in the gas phase on printed circuit boards, effectively digitising separations prior to mass spectrometry analysis. A 13-m serpentine ion path length can be achieved in a component that is 14 × 18 inches. The new product can be integrated with LC-MS workflows and can, for some applications, replace the liquid chromatograph.
MOBILion Systems
http://link.spectroscopyeurope.com/32-065

Mass spectrometry proteomics software

The newest release of Thermo Scientific Proteome Discoverer 2.5 software provides higher confidence peptide identification, more accurate quantification and higher throughput data analysis than previous versions. The intelligent software uses deep learning facilitated by a Prosit-derived neural network, licensed in collaboration with MSAID GmbH, to provide more accurate prediction of fragmentation mass spectra. The technology enables more confident results in applications such as human leukocyte antigen (HLA) or metaproteomic analysis, which require large search spaces. Additionally, the new release includes a new enrichment service that helps provide biological meaning for differential analyses and support of targeted workflows. The software features tools to help deploy Thermo Scientific SureQuant Targeted Mass Spectrometry Assay Kits and custom SureQuant IS-triggered acquisition assays in new proteomics laboratories.
Thermo Fisher Scientific
http://link.spectroscopyeurope.com/32-050

Web content

There is a lot more content published on spectroscopyeurope.com than we have space for in the print edition. You may like to browse the news section in particular. From time to time, we highlight particular content that you can only read online. The bit.ly shortlinks just point to the story on our website.

- News: WITec announces 2020 Paper Award winners: https://bit.ly/sewitecawards
- News: Bruker partners with ANPC to combat the COVID-19 threat: https://bit.ly/seanpc
- Application: Enhanced solar cell performance with Pt-based "Roller Wheel" molecules, https://bit.ly/sesolarapp
- Application: EDXRF analysis of carbon black, https://bit.ly/secarbonapp

NEW PRODUCTS

ATOMIC

Quad-quadrupole ICP-MS instrument

PerkinElmer's NexION® 5000 multi-quadruple ICP-MS instrument has four quadrupoles. The first, Quadrupole Ion Deflector (Q0), directs ions to the entrance of the first mass filter. The second, Transmission Analyser Quadrupole 1 (Q1, full-sized for <0.7 amu mass resolution), acts as a mass filter or as an ion guide to direct ions to the Quadrupole Universal Cell. The third, Quadrupole Universal Cell (Q2), using dynamic bandpass tuning, prevents side reactions with residual gases in the cell. The fourth, Transmission Analyser Quadrupole 2 (Q3, full-sized for <0.7 amu mass resolution), acts as a mass filter or as an ion guide to direct ions to the detector.

The Extended Dynamic Range (EDR) feature increases linear dynamic range to 10^{12}, allowing both high and low concentration analytes to be handled in a single analytical run, resulting in fewer re-runs. The instrument also has a number of improvements to reduce maintenance for greater uptime. A Triple Cone Interface with patent-pending OmniRing™ combined with a quadrupole ion deflector allow no maintenance beyond the cones, for continual operation and improved stability. The

34-MHz free-running RF generator is air cooled (no water or gas cooling), so is maintenance-free, eliminating need to replace plasma load coils.

PerkinElmer

http://link.spectroscopyeurope.com/32-041

INFRARED

Spectrum 3 FT-IR spectrometer

PerkinElmer's new Spectrum™ 3 FT-IR spectrometer covers the near, mid and far-IR ranges (11,000–30 cm^{-1}). There is automatic switching between beamsplitters without manual user intervention and the sources can be switched from IR to NIR at the touch of a button. Two sampling accessories can be installed simultaneously, avoiding the need to switch accessories between measurements. A fully integrated TG-IR (EGA4000) solution can be added, and the spectrometer is upgradeable to IR microscopy and imaging with automated switching of the beam to any PerkinElmer microscopy or imaging system. Experiments can be run directly from the SmartPanel on the instrument without returning to the PC, and IR data can be accessed from anywhere with cloud connectivity.

PerkinElmer

http://link.spectroscopyeurope.com/32-042

LUMINESCENCE

New generation Fluorolog research spectrofluorometer

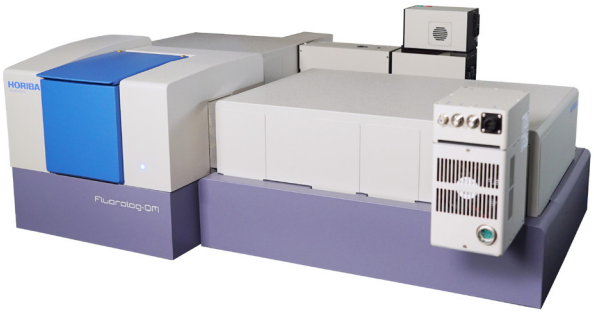
The Fluorolog-QM is the fourth generation of the Fluorolog, all reflective, modular research spectrofluorometer. The Fluorolog-QM is a lens free, all reflective spectrofluorometer covering all wavelengths from the deep UV (180 nm) to the IR (5500 nm). The Fluorolog-QM has a guaranteed sensitivity of 32,000 : 1 signal-to-noise ratio for the Raman band of water using the FSD (Square Root) method. It also has focal length of 350 mm for single monochromators, and 700 mm for double monochromators. The instrument is controlled with HORIBA's newest fluorescence software, FelixFL, for all acquisition and analysis of spectral and time-resolved data.

The Fluorolog-QM can be configured to suit a broad range of luminescence research applications, ranging from a simple steady state configuration with a single light source and single cooled PMT housing, to a versatile configuration with four different light source options and six different detectors, all connected

to the same instrument and all controlled automatically with FelixFL software.

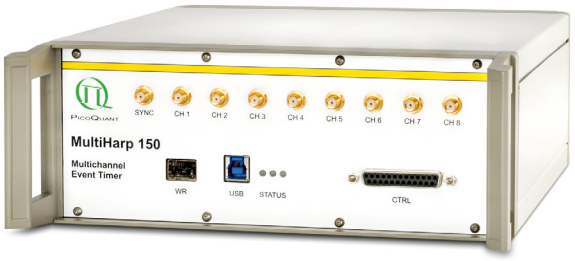
Horiba Scientific

http://link.spectroscopyeurope.com/32-046



PicoQuant combines high timing precision with ultrashort dead time

PicoQuant has released three new models of the high-throughput multichannel event timer MultiHarp 150: MultiHarp 150 4P, 8P and 16P. The units have either 4, 8 or 16 detection channels and offer improved timing precision with 10 ps minimum bin width, along with jitter better than 45 ps (root mean square). The new MultiHarp 150 models are PicoQuant's latest TCSPC electronics for fast and high-resolution fluorescence lifetime imaging (rapidFLIM) and multichannel photon correlation. All detector channels and the common SYNC input of a MultiHarp 150 are all independent and synchronised. The SYNC input can also be used as either an additional detection channel or for a periodic reference signal with repetition rates up to 1.2 GHz. Depending on the configuration of the computer, the MultiHarp 150 can sustainably capture and transfer 80 million time-tags per second with single channel peak rates of up to 1.5 Gcps for short times. Thanks to its hardware-integrated 65536 bin histogrammer,



count rates up to 166 million counts per second (Mcps) in total for 4 or 8 channel versions, and up to 332 Mcps for the 16 channel version can be achieved in lifetime measurements, while keeping both the data transfer to the PC and the CPU utilisation low.

PicoQuant

http://link.spectroscopyeurope.com/32-040

MASS SPEC

MCPD system

Shimadzu and Axel Semrau have developed a solution to detect production-related contaminants [monochloropropane diol (MCPD) fatty acid ester, glycidyl fatty acid ester and free MCPD] in food containing fats and oils. The system consists of a triple quadrupole mass spectrometer and an automated sample management system. Since the direct detection of MCPD fatty acid ester is not yet possible due to the unmanageable number of isomers and the lack of suitable standards, the new solution uses an indirect method of determining the content of 2-MCPD, 3-MCPD and glycidol. This includes the transesterification of MCPD and glycidyl ester as well as the extraction of the resulting free MCPD and its derivatisation. There is a choice of the GCMS-TQ8040 NX or the GCMS-TQ8050 NX as part of the solution, and complete databases for food analysis are available for both models.

Shimadzu

http://link.spectroscopyeurope.com/32-060

NEW PRODUCTS

SAMPLING

Spectroscopy autosamplers

The S20 series of autosamplers is the latest generation designed specifically for PerkinElmer's spectroscopy platforms, atomic and molecular. It comprises the S23 and S25, which are equipped with proprietary crash detection, programmable intelligent acceleration and deceleration speed in three axis, dual rinse station and LED status light. This series is also designed with removable sample trays for easy switching between running aqueous and organic matrices.

PerkinElmer

http://link.spectroscopyeurope.com/32-045

New features for Spectrum Two+

PerkinElmer's Spectrum™ Two+ FT-IR spectrometer is an update to the Spectrum Two instrument. New features are a rugged touchscreen and secure, cloud connectivity. The UATR (universal attenuated total reflectance) accessory has a new improved design.

PerkinElmer

http://link.spectroscopyeurope.com/32-044

RAMAN

Raman system for forensic analysis

Renishaw has introduced the inVia™ InSpect, a new version of the inVia confocal Raman microscope optimised for use in forensic laboratories for trace evidence analysis. Forensic science covers many disciplines because evidence can take many different forms. This means the typical forensic laboratory is home to a range of analytical systems. Renishaw has designed the inVia InSpect Raman microscope to complement these technologies. It can be used in isolation to analyse samples that cannot be examined fully using other techniques, and it can also be used with them to obtain more detailed chemical information. The inVia InSpect can identify materials that may be difficult or time consuming to prepare. For example, hard crystalline powders, ceramic shards and glass chips can all be easily analysed with virtually no preparation. The InSpect's confocal Raman capability allows the analysis of complex structures containing layers, voids or inclusions. Upgrade options are available for more specialist forensics applications.

Renishaw

http://link.spectroscopyeurope.com/32-047

New generation alpha300 *apyron* Raman microscope

WITec has introduced a new generation of their alpha300 *apyron*, the model with high levels of automation and user-friendliness. The alpha300 *apyron* can self-align and self-calibrate, enhancing reproducibility and eliminating potential sources of error. This is made possible by a complete set of WITec's AutoBeam opto-mechanical components, a new and versatile tool kit that can be configured to create the optimal experimental setup for every investigation. New functionalities provided by AutoBeam modules include polarisation-dependent measurements with motorised polariser and analyser rotation, push-button spectrometer connection and signal maximisation, and automated adjustment of both iris diaphragms. Software-driven automation also allows the alpha300 *apyron* to be operated completely remotely, whether in an environmental enclosure such as a glove box, or from another location.

The alpha300 *apyron* microscopes are available with a wide variety of excitation wavelengths from the ultraviolet through the

visible into the near infrared, and can be equipped with up to three ultra-high throughput WITec UHTS spectrometers and their accompanying range of detectors. Raman imaging and correlative measurements are defined, executed, saved and evaluated with WITec's Suite FIVE software and EasyLink handheld controller.

WITec

http://link.spectroscopyeurope.com/32-043

X-RAY

Handheld XRF analyser with light element detection

The new Vanta Element-S handheld X-ray fluorescence (XRF) analyser provides fast light element detection at an affordable price, adding to the family of entry-level Vanta Element XRF instruments. The S model is equipped with a silicon drift detector (SDD) to analyse light elements such as magnesium (Mg), aluminium (Al), silicon (Si), sulfur (S) and phosphorus (P) in alloys. It is suitable for scrap recycling, basic PMI, metal manufacturing and precious metals, measuring ferrous metals, aluminium, copper, stainless steel, nickel and gold karats. The analyser offers on-screen grade ID and comparison for the light elements Mg, Al and Si in seconds. Its SDD detector can distinguish similar alloy grades such as 303 stainless steel from 304, and aluminium 6061 or 6063 from 1100.

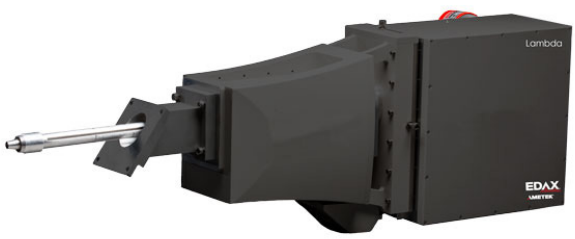
The Vanta Element-S weighs 1.32 kg and is IP54-rated to resist dust and moisture and built to pass a 1.2 m drop test (MIL-STD-810G) to help protect from the occasional drop or jostle. Other protective features include a stainless-steel faceplate and a Prolene® window with Kapton® mesh support that easily sticks on and peels off for tool-less window changes in the field. Optional wireless connectivity is available, and users can connect to the Olympus Scientific Cloud™ for wireless data sharing and access to convenient fleet management tools, as well as the Olympus mobile app or the company network. The analyser also has a 1-GB microSD™ card to store results and two USB ports to export data.

Olympus IMS

http://link.spectroscopyeurope.com/32-056

WD X-ray spectrometer

EDAX has announced the new Lambda wavelength dispersive spectrometry (WDS) series. This utilises a combined hyperbolic–parabolic HCO optical design with a polycapillary transmitter to optimise X-ray optics across a wide energy range. It is supplied with a complete and fully customised set of diffractors. Lambda spectrometers do not require a dedicated WDS port and, having a compact geometry, they are compatible with additional detectors installed on the same SEM. The spectrometers are fully integrated with Energy Dispersive Spectroscopy and Electron Backscatter Diffraction detectors using the same software interface. The new WDS product platform includes the Lambda Plus and Lambda Super WDS spectrometers, covering up to 10 keV and beyond 15 keV energy ranges, respectively. Based on the sealed gas counter detector design, Lambda spectrometers are fully compatible with high vacuum applications that are especially important for SEMs with FEG emitters. The sealed gas detector design and extended energy range opens additional application domains for Lambda products, such as *in situ* and environmental WDS measurements.



EDAX

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- ☐ Polarimetry
- ☐ Process spectroscopy
- ☐ Raman spectroscopy
- ☐ Sampling
- ☐ Separation Science
- ☐ Spectroradiometry
- ☐ Surface Analysis
- ☐ Terahertz Spectroscopy
- ☐ UV/Vis
- ☐ X-Ray Diffraction
- ☐ X-Ray Spectrometry
- ☐ None

Area of employment:
(please tick ONE only)

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- ☐ Independent Lab
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- ☐ Hospital
- ☐ Government
- ☐ Research Inst/Foundation
- ☐ Other_____

Job function: *(please tick ONE only)*

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Notes on the Diary

As you can imagine, many events have been cancelled or postponed due to COVID-19. Where we have been notified about changes, these have been updated. We have decided to leave others in for two reasons. First, a visit to the website will quickly tell you the status of the event. Second, the presence of an event here will remind you that it takes place, and you can keep track of any new dates from the website.

It must be a particularly difficult time for meeting organisers and we want to do all we can to support them and their meetings. If you have updates, do let us know: circ@spectroscopyeurope.com.

Conferences

2020

23–28 August, Boston, MA, United States. **XXIX International Conference on Magnetic Resonance in Biological Systems (ICMRBSXXIX).** John Markley, jmarkley@wisc.edu, http://www.icmrbs.org/.

20–26 September, Aachen, Germany. **17th International Symposium of Trace Elements in Man and Animals (TEMA17).** Prof. Dr. Lothar Rink, immunologie@ukaachen.de, https://www.ukaachen.de/kliniken-institute/institut-fuer-immunologie/institut.html.

20–25 September, Kyoto, Japan. **11th International Conference on Laser-Induced Breakdown Spectroscopy (LIBS2020) (Web Conference).** 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/.

11–16 October, Reno, NV, United States. **47th Annual Conference of Federation of Analytical Chemistry and Spectroscopy Societies (SciX2020) (Possible Virtual Event).** scix@scix-conference.org, https://www.scixconference.org/event-3326054.

6–10 December, Bilbao, Spain. **European Congress on Magnetic Research (EUROMAR 2020).** euromar2020@kenes.com, https://www.euromar2020.org/.

2021

14–16 January, Amsterdam, Holland. **Hyperspectral Sensing Meets Machine Learning and Pattern Analysis (HyperMLPA).** http://www.spectro-expo.com/hypermlpa/.

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

15 January Amsterdam, Holland. **2nd Symposium on Short Wave Infrared Imaging and Spectroscopy (Swllms).** http://www.spectroexpo.com/swiims/.

31 January–5 February, Ljubljana, Slovenia. **2021 European Winter Conference on Plasma Spectrochemistry.** Johannes T. VanElteren, http://www.ewcps2021.ki.si/.

Spring, Ivine, United States. **RamanFest 2020.** https://www.ramanfest.com/.

28–30 June, Manchester, United Kingdom. **The 20th Biennial National Atomic Spectroscopy Symposium (BNASS 2020).** Dr Phil Riby, philip.riby@manchester.ac.uk, http://www.rsc.org/events/detail/40623/bnass-2020-the-20th-biennial-national-atomic-spectroscopy-symposium.

Summer, Courmayeur, Italy. **18th Chemometrics in Analytical Chemistry Conference (CAC2020).** ludovic.duponchel@univ-lille.fr, https://cac2020.sciencesconf.org/.

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

18–21 October, Trondheim, Norway. **2nd Nordic Metabolomics Conference.** mila.knoff@ntnu.no, https://www.ntnu.edu/isb/nmc2020.

Exhibitions

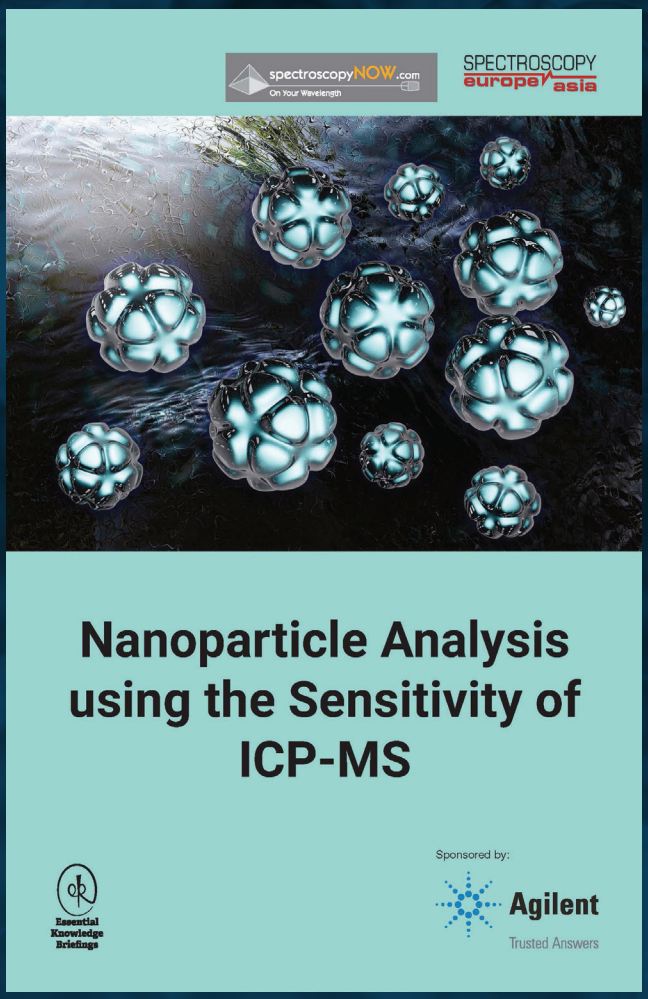
2020

14–16 January, Amsterdam, Netherlands. **Spectro Expo 2021.** http://www.spectroexpo.com

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

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

20–24 June, Düsseldorf, Germany. **51st International Symposium on High Performance Liquid Phase Separation and Related Techniques.** Michael Lammerhofer, michael-laemmerhofer@uni-tuebingen.de, https://www.hplc2021-duesseldorf.com/.



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