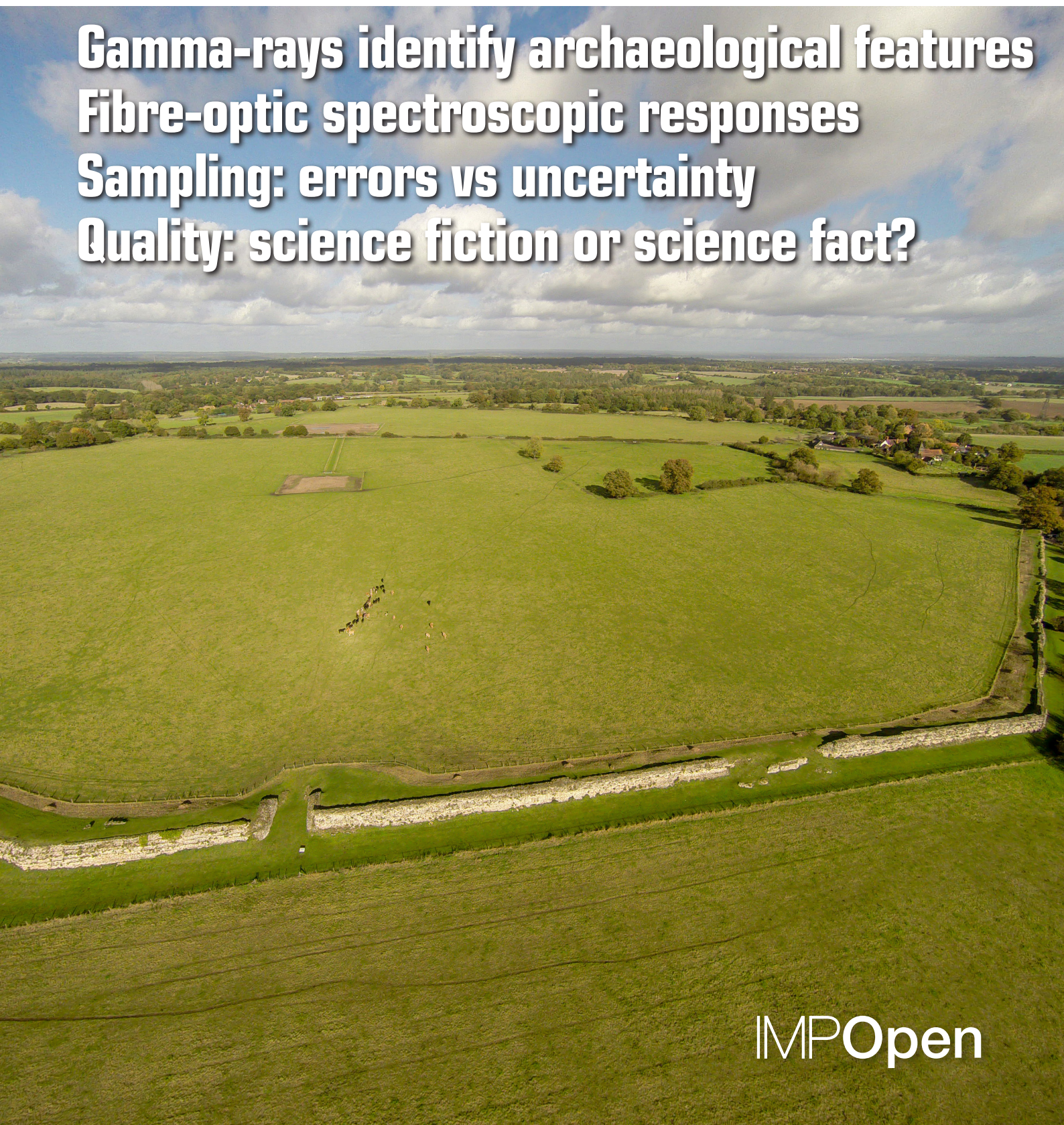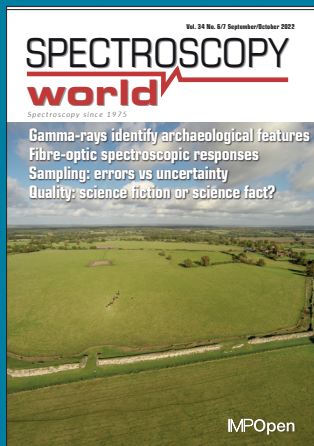# SPECTROSCOPY world

Spectroscopy since 1975

Gamma-rays identify archaeological features
Fibre-optic spectroscopic responses
Sampling: errors vs uncertainty
Quality: science fiction or science fact?

IMPOpen

This is an aerial photo of the Roman settlement at Silchester in Hampshire, UK. The article, starting on page 20, explores the use of gamma-ray spectroscopy to study archaeological structures below ground at various parts of the site.

CONTENTS

Spectroscopy World is a digital magazine, published eight times a year and available free-of-charge to qualifying individuals outside Europe and the Middle East.

IMPOpen

EDITORIAL

Six or seven years ago I was able to return to mountain (off-road) biking thanks to discovering electric mountain bikes (e-MTBs). This immediately increased the distance I could cover on a ride and encouraged exploring. No longer was taking a wrong turn and finding that I had to bike back up a steep hill the "horror" it used to be! An unexpected benefit was that I found that a large number of Stone Age sites remain around the area of the South Downs in which I live. I pass tens of barrows (burial mounds), and many cross dykes and other pre-historic boundary indicators on a typical ride. This has sparked an interest in our early history and archaeology in general. This is a rather lengthy introduction to our first article that uses gamma-ray spectroscopy to investigate more recent buried historic sites. "Atomic archaeology? Using portable gamma surveying techniques to identify buried archaeological features"

by Victoria Robinson describes how gamma-ray spectroscopy can reveal new features in buried archaeological sites. In her case at the Roman settlement of Silchester in Hampshire, UK, and other sites.

A second, sponsored, article from Jamie Foss, Cole Strattman and Simonetta Tumbiolo of PerkinElmer looks at "Overcoming the challenges of reducing background interference for LC/MS/MS trace PFAS analysis". This provides a thorough overview of what you should consider if you need to analyse per- and polyfluoroalkyl substances without contamination from your instrumentation. I am sure there are applications to other similar analytes and analysis methods.

In the Tony Davies column, Tony and Wieland Hill look into "Exploiting fibre-optic spectroscopic responses". Here, artefacts that are normally avoided in spectroscopy can be used to provide information about the environment a

fibre optic is exposed to. Applications include border security and leak detection.

Kim Esbensen and Francis Pitard use their many decades of representative sampling experience to consider "Errors vs uncertainty vs measurement uncertainty". "Error" and "uncertainty" are being used interchangeably and confusingly. This is "a scientific flaw of the first order"! However, Kim and Francis will put you right.

John Hammond finishes his *magnum opus* on "Four Generations of Quality" with a look at what is science fiction and what is science fact. He considers what may turn out to be "fact" in the future for each of the preceding eight articles in the series. Predicting the future is always a dangerous game. Feel free to use the online commenting facility to add your own thoughts.

NEWS

All-attosecond pump–probe spectroscopy

An international team of researchers has demonstrated attosecond-pump attosecond-probe spectroscopy to study non-linear multi-photon ionisation of atoms.

An international team of researchers from the Max Born Institute in Berlin, University College London and ELI-ALPS in Szeged, Hungary, has demonstrated attosecond-pump attosecond-probe spectroscopy to study non-linear multi-photon ionisation of atoms. The obtained results provide insights into one of the most fundamental processes in non-linear optics.

Femtosecond pump-probe spectroscopy has revolutionised the understanding of extremely fast processes. For instance, the dissociation of a molecule can be initiated by a femtosecond laser pump pulse, and can then be observed in real time using a time-delayed femtosecond probe pulse. The probe pulse interrogates the evolving state of the molecule at different time delays, making it possible to record a movie of the molecular dissociation. This powerful technique was recognised with the Nobel Prize in Chemistry in 1999.

Some processes in nature, however, are even faster and take place on attosecond timescales. It would, therefore, be ideal to initiate an ultrafast process using an attosecond pump pulse, and to interrogate the system under investigation using an attosecond probe pulse. So far, attosecond-pump attosecond-probe spectroscopy has been demonstrated for relatively simple processes involving the absorption of two photons. However, since all-attosecond pump–probe spectroscopy is very challenging, most experiments in attosecond science use only one attosecond (pump or probe) pulse in combination with one femtosecond pulse.

The researchers have now been able to demonstrate a pump–probe experiment, in which complex multi-photon ionisation processes
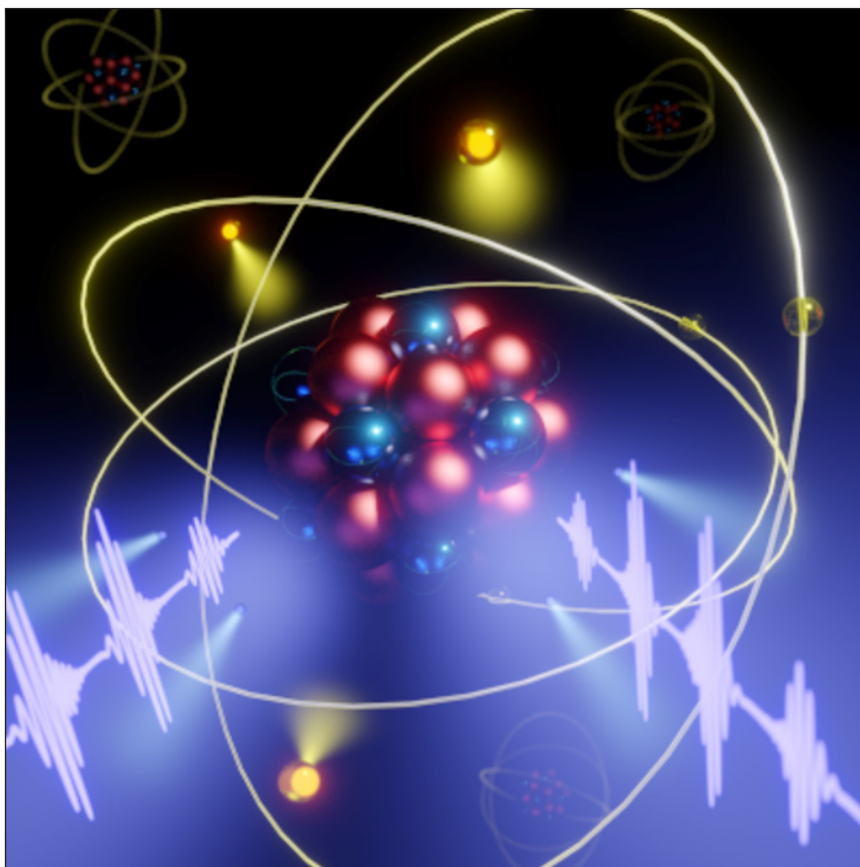
Figure 1. Two intense attosecond pulse trains (white) interact with an atom, resulting in the emission of three electrons (yellow). During this process four photons (blue) are absorbed. The probability of this process can be controlled by varying the temporal and the spatial overlap between the two attosecond pulses. Credit: Balázs Major

were studied using two attosecond pulse trains. This experiment required the generation of very intense attosecond pulses, for which a large laser system was used. Consequently, the researchers performed the experiment in the largest laboratory that is available at the Max Born Institute. At the same time, the two attosecond pulses had to be overlapped with attosecond temporal and nanometre spatial stability. This explains why these experiments are so challenging.

The artistic visualisation of the experiment (Figure 1) shows two attosecond pulse trains interacting with an argon atom. Following the absorption of four photons from the attosecond pulses, three electrons were removed from the atom. There are many possible ways in which this multi-photon absorption may take place. To find out in detail how the electrons were removed from the atom, the researchers varied the time delay between the two attosecond pulses and observed how many ions were generated.

As shown in Figure 2, the yield of the doubly-charged Ar^{2+} ions (red curve) was almost independent

Introduction to the Theory and Practice of Sampling

Kim H. Esbensen

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

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

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

"I recommend this book to all newcomers to TOS"

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

"One of the book's major advantages is the lavish use of carefully designed didactic diagrams"
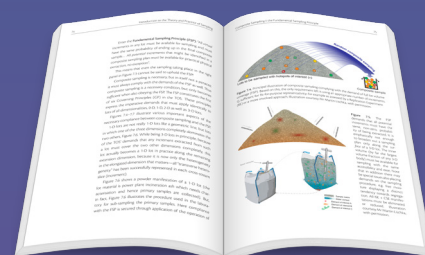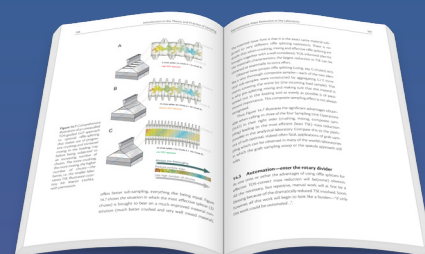
impopen.com/sampling

IMPublicationsOpen

of the time delay. In contrast, the yield of the triply-charged Ar^{3+} ions (blue curve) shows pronounced oscillations when varying the time delay between the two attosecond pulses. The researchers were able to conclude that the multiphoton absorption occurred in three steps: In each of the first two steps a single photon was absorbed, whereas in the third step two photons were absorbed at the same time. These results were confirmed by computer simulations that were carried out at University College London and at ELI ALPS.

The developed experimental technique, reported in *Optica* (doi.org/h9b6) can be used in the future to study complex processes not only in atoms, but also in molecules, solids and nanostructures. An exciting question that the researchers hope to answer is how several electrons interact with each other. This could help to understand the most fundamental processes on the shortest timescales. ↗
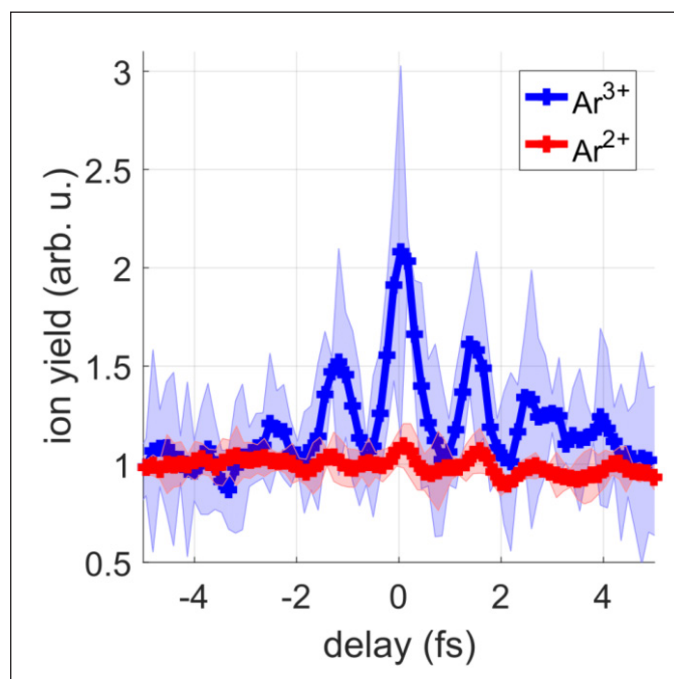
Figure 2. Ar^{2+} and Ar^{3+} ion yields as a function of the time delay between two attosecond pulse trains. (a) The Ar^{2+} ion yield (red curve) is only weakly modulated as a function of the XUV-XUV time delay, whereas clear oscillations with a period of 1.3 fs are observed in the delay-dependent Ar^{3+} ion yield (blue curve). These results indicate that Ar^{2+} is generated via the sequential absorption of two photons. Subsequently, two additional photons are simultaneously absorbed to form Ar^{3+}. Credit: MBI

Common factor-based methods for hyperspectral image exploration

A new Open Access paper in *JSI—Journal of Spectral Imaging* compares common factor-based methods for hyperspectral image exploration: PCA, MAF, MNF and MDF.

A new Open Access paper in *JSI—Journal of Spectral Imaging* (doi.org/h9b7) compares common factor-based methods for hyperspectral image exploration: principal components analysis (PCA), maximum autocorrelation factors (MAF), minimum noise factors (MNF) and maximum difference factors (MDF). These can all be posed as a maximisation problem that ultimately yields a symmetric eigenvalue problem (SEP) for each model.

The SEPs allow a simple comparison of the models for analysis of hyperspectral images using a PCA metaphor with MAF, MNF and MDF describable as weighted PCA. The examples in the paper showed how the different methods
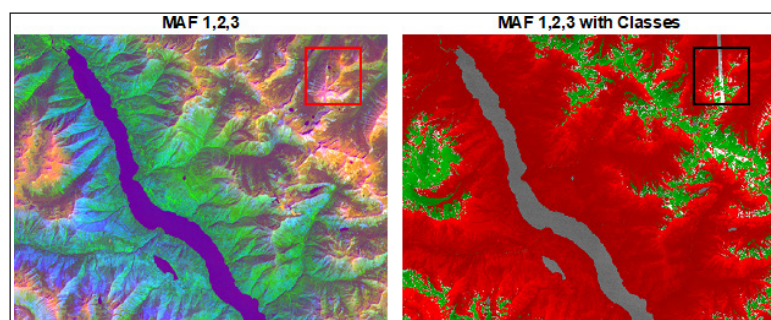
captured different signals in the images and the scores from each method can be combined synergistically allowing for additional modelling and extended visualisation. MDF is a factor-based edge detection model that not only allows for additional visualisation but the opportunity to identify and

exclude (or highlight) edge signal in the images. The third example showed that models can also be used synergistically for finding and elucidating anomalies. In the example, MDF showed the highest sensitivity of the models studied for anomaly detection. ↗

MS imaging makes ingredients, additives and contaminants of food visible

Mass spectrometry imaging provides highly precise information on the spatial distribution of substances. Researchers at the University of Bayreuth have succeeded in making visible an additive in dairy products and a production-related contamination in baked goods.

Mass spectrometry imaging (MSI) provides highly precise information on the spatial distribution of substances in many areas. Researchers at the University of Bayreuth have succeeded in making visible an additive in dairy products and a production-related contamination in baked goods. Special ingredients that influence food quality can be detected in fruit, vegetables and meat products. The study, which was conducted in cooperation with the Bavarian Health and Food Safety Authority (LGL) and published in *Food Chemistry* (doi.org/gpqnwg), shows the great potential of this method, not least in terms of consumer protection.

Natamycin in cheese
To protect cheese wheels or smoked sausages from mould infestation, the surfaces are often treated with the fungicide natamycin. An EU regulation sets a limit of one milligram per square decimetre for this and also stipulates that natamycin must not penetrate deeper than 5 mm into a treated cheese wheel. However, this penetration depth cannot be described in detail using the food analysis methods commonly in use. However, the Bayreuth research team led by Prof. Dr Andreas Römpp has been able to use MSI to show for the first time where and in what quantities the fungicide occurs in different types of Gouda. The penetration of the natamycin molecules can be tracked from the rind to the inside of the cheese wheel. The scientists collaborated with the LGL in these investigations. Based on the results obtained, they have developed methodological standards for

The fungicide natamycin (green) penetrates inward from the coating (left) in some locations. Credit: © Julia Kokesch-Himmelreich

the identification of natamycin in cheese", says Prof. Römpp.

Acrylamide in gingerbread
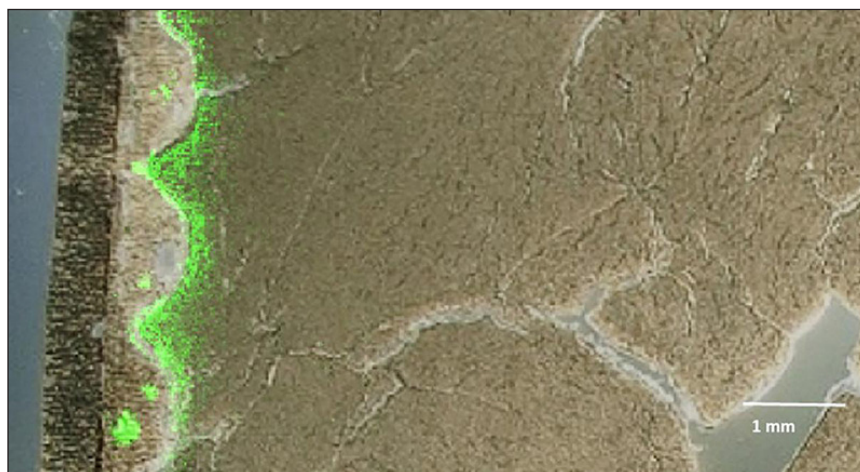An EU regulation also sets limits for the presence of acrylamide in food. It is a cancer-promoting substance that is formed from sugar and asparagine at low humidity and temperatures above 120°C. A method developed in Bayreuth, Germany, based on MSI, visualises acrylamide distribution in traditional German gingerbread. "To do this, we had to cool the gingerbread samples to less than –60°C and then use an electric microsaw to produce gingerbread slices of 2 mm thickness. This was the only way we could detect very small amounts of acrylamide", reports Prof. Römpp.

Studies of veal sausages
The new study also shows that MSI is equally suitable for analyses of processed meat products. In veal sausages, water-soluble and fat-soluble components become visible, so that low-fat and high-fat regions can be clearly distinguished.

Likewise, it becomes visible where substances of plant origin are found that come from admixed herbs. "However, MS imaging not only enables the localisation of ingredients in meat products, but also helps, for example, in investigations of 'sticky meat' or so-called hydrolysate additives, which are supposed to feign higher quality when they are not declared on the packaging. It could therefore be useful in detecting consumer deception in meat products and better protect consumers in this respect as well", says Prof. Römpp.

Kiwifruit and carrots
The application potential in the field of fruits and vegetables is demonstrated by studies on kiwifruit and carrots. The "mini kiwi" (*Actinidia arguta*) is not only sweet, but also has numerous health-promoting bioactive ingredients. Using sample slices that were only a few hundredths of a millimetre thick and cooled down to a temperature of –40°C, the Bayreuth bioanalysts visualised the distribution of
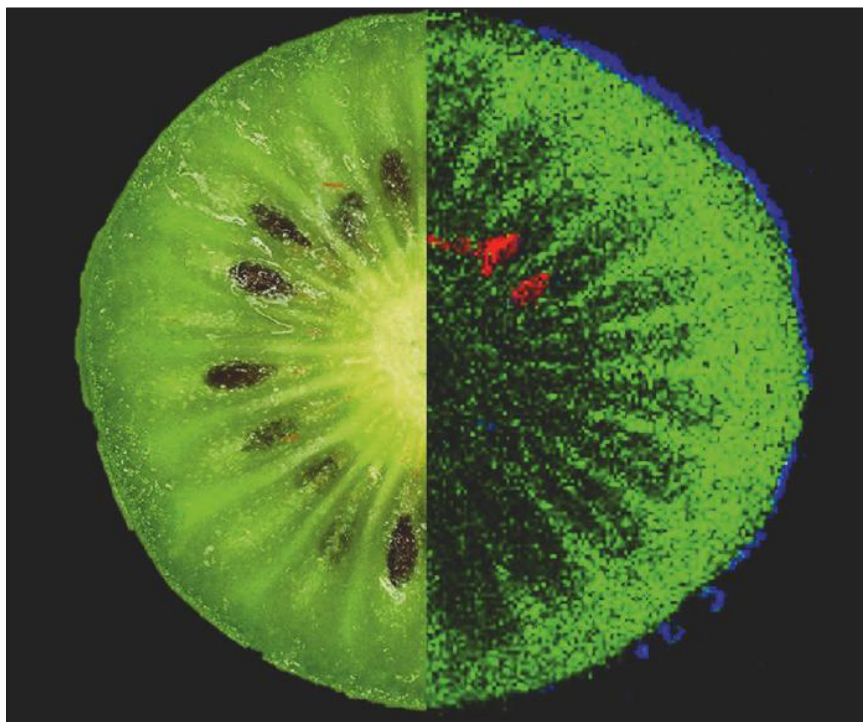
Ingredients of a kiwi: green = sugar, blue = polyphenol, red = typical kiwi lipid. Credit: © Oliver Wittek

several substances in the skin and flesh: sugar molecules (disaccharides), antioxidant polyphenol and a fat (lipid) characteristic of kiwis. In carrots, in turn, molecules of beta-carotene, a precursor of vitamin A, were detected. In addition, it was also possible to identify the spatial distribution and typical molecular structures of different dyes (anthocyanins) that give carrots an orange, yellow or violet coloration.

"Our study makes it clear that MS imaging is a valuable addition to already established food analysis methods: it offers new insights into the spatial distribution and relative proportions of ingredients. It has the great advantage that the molecules of the ingredients do not have to be labelled with dyes or other labelling methods. At the University of Bayreuth—within the newly established Faculty VII of Life Sciences: Food, Nutrition and Health—we will continue to work in the future on refining the analytical capabilities of imaging mass spectrometry, combining it with other food analysis tools,

and applying it to ingredients not previously studied. In this way, we at the University of Bayreuth can make important contributions to consumer protection", says Prof. Römpp. 〽

Mass spectrometry reveals action of drug compounds in tissue

Native ambient mass spectrometry can analyse how drug molecules bind to proteins in tissue samples and could offer an improved route to drug discovery and development.

Researchers at the University of Birmingham, in collaboration with AstraZeneca, have developed a technique that uses native ambient mass spectrometry (MS) to detect intact protein–drug complexes directly from liver tissue.

Part of the early stages of drug discovery takes place in cell cultures, enabling the effects of different compounds to be tested on specific biological targets involved in various diseases. Although this allows researchers to assess how the compounds act against the target, it does not

capture the full effects of the physiological environment.

This new technique enables researchers to use real tissue samples to assess which proteins the drug will bind to in the body and, therefore, how effective it is likely to be against the target. Being able to pinpoint the interaction between the drug and the protein can provide valuable insight to guide drug discovery.

"Identifying the drug–protein interaction at an early stage is incredibly hard. Using MS on proteins is often compared to making an elephant fly. What

we've done is add an unsecured hat—the drug molecule—to the elephant, and measured the whole process", said Professor Helen Cooper, School of Biosciences at the University of Birmingham, UK. "Usually in early-stage drug discovery, measurements are taken outside of the physiological environment, so when researchers move onto testing drugs in tissue, they can fail because they have interactions that were not expected."

In the study, published *Angewandte Chemie* (doi.org/gqhgjf), in the researchers used

Targeted proton transfer charge reduction (tPTCR) nano-DESI mass spectrometry imaging of liver tissue from orally dosed rat (Animal 3). a) optical image of a blood vessel within liver tissue. b) Composite ion image of charge-reduced haeme-bound α-globin (7+ and 6+ charge states; *m/z* 2259.9 and *m/z* 2636.3 respectively, red) and the charged-reduced [FABP+bezafibrate] complex (7+ and 6+ charge states; *m/z* 2097.5 and *m/z* 2446.9 respectively, blue). c) Ion image composed from charge-reduced haeme-bound α-globin (7+ and 6+ charge states) showing abundance in blood vessels. d) Ion image composed from charge-reduced [FABP+bezafibrate] complex (7+ and 6+ charge states) showing abundance in bulk tissue and absence in the blood vessel. Reproduced from https://doi.org/10.1002/ange.202202075 under a CC BY licence.

tissue taken from the livers of rats dosed with bezafibrate, a drug commonly used to treat high cholesterol. They used MS on thin sections of tissue to detect the drug molecule and the specific fatty acid binding protein to which it attaches to form a complex.

Further, they undertook MS imaging of a dosed liver section by use of native nanospray desorption electrospray ionisation (nano-DESI). This enabled visualisation of the distribution of the [FABP1+bezafibrate] complex throughout the bulk liver tissue and the haeme-bound α-globin in the blood vessels (see image).

Next steps for the research will include improving the sensitivity of the technique, and extending it to other types of drug compounds. Looking further ahead, the team hopes it can be developed for use in human tissue, taken from biopsies. This would yield a greater understanding of why drugs work differently in different patients. ↳

Raman spectroscopy overcomes an additive manufacturing constraint

Two-photon-polymerisation lithography structures examined via Raman spectroscopy and nanoindentation.

Additive manufacturing (AM) using two-photon polymerisation lithography (TPP) has increased in usage in industry and research. Currently, a major constraint of TPP in general and specifically of the material IP-Q (Nanoscribe GmbH, Germany) is the users' limited access to knowledge about material properties. Due to the nature of the process, the elastic properties in particular depend not only on the utilised material but also on structure size, process and fabrication parameters. For example, no degree of conversion (*DC*) and Young's modulus (*E*) values for IP-Q had been reported.

Due to the nature of the process, the elastic properties in particular depend not only on the utilised material but also on structure size, process parameters and hatch strategy. A common approach uses a combination of Raman spectroscopy and nanoindentation to characterise the *DC* of monomer to polymer measurable via Raman spectroscopy, which can then be related to the mechanical behaviour of the material, measurable via nanoindentation.

Ongoing research on acoustic metagratings and metamaterials fabricated on MEMS would benefit from optimised elastic parameters

to provide adjustability of the acoustic behaviour, as they affect the characteristic acoustic impedance directly. AM encompasses processes with which objects can be created three-dimensionally from a technical drawing. The data are sent to an AM system, which then performs the fabrication. AM via TPP is based on selective curing of a liquid precursor to create solid structures inside a drop of monomer. Afterwards, the leftover liquid is washed away. Well known TPP applications are optical sub-micron structures, where the photoresist IP-Dip (Nanoscribe GmbH, Germany) is commonly utilised. The

NEWS
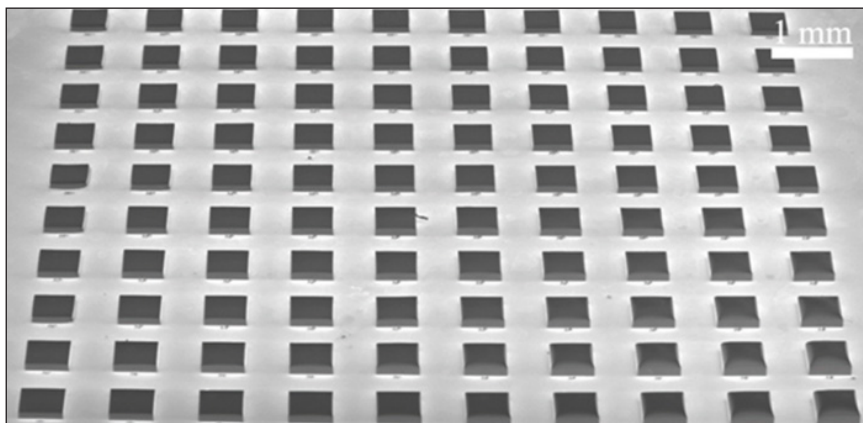
SEM micrograph of a cuboid parameter sweep comprising 10×10 elements fabricated via TPP from the photoresist IP-Q. Credit: The Authors, doi: 10.1117/1.JOM.2.3.033501

more recently developed photoresist IP-Q was designed by the same manufacturer for larger applications, e.g. mounts, moulds and structural metamaterials. Sample structures from each of the two photoresists were produced in parameter sweeps. This allows the comparison of process parameters to the resulting characteristics. Raman spectroscopy was employed.

The reflection comprises not only the irradiated wavelength but also Raman scattering. The characteristic peaks of the Raman scattering spectrum can be used for the identification of chemical substances. In our work, it was used to determine the ratio of monomer to polymer, or DC, in the TPP samples.

Micro- and nanoindentation were used to test the mechanical properties of the samples. A hard tip whose mechanical properties are known is pressed into the sample whose properties are unknown. From the slope of the load vs displacement curve, E values were computed.

Finally, parameter sweeps of cuboid sample structures fabricated using TPP were investigated across the parameters laser power and scan speed to find dependent properties. The employed photoresists were examined using Raman spectroscopy to find the DC of monomer to polymer, and subsequently micro- or nanoindentation were used to find E.

For IP-Dip, the attained DC and E ranged from 20% to 45% and 1 GPa to 2.1 GPa, respectively. The results were compared to reports found in the literature. For IP-Q, the attained DC and E ranged from 53% to 80% and 0.5 GPa to 1.3 GPa, respectively. The characterised properties of IP-Q manifest as the current state of knowledge of the material.

"By this means, offering an approach to optimise elastic parameters of TPP-fabricated structures will be beneficial for various ongoing research topics. A promising application for this method is the characterisation of the elastic parameters of acoustic metagratings and metamaterials fabricated on MEMS. These devices can subsequently be implemented beneficially in life science, mobility and industrial applications", said Severin Schweiger of Fraunhofer Institute of Photonic Microsystems and Brandenburg University of Technology in Germany.

Making it easier to differentiate chiral molecules

Using a new method, helical dichroism in the X-ray domain, scientists are better able to distinguish between chiral substances.

Chirality is especially relevant in biological molecules, since it can cause different effects in the body. Thus it is essential in biochemistry and toxicology, as well as in drug development, to separate enantiomers from each other so that, for example, only the desired variant gets into a drug. Now researchers from PSI, EPFL and the University of Geneva have jointly developed a new method that enables

enantiomers to be better distinguished, and thus better separated, from each other: helical dichroism in the X-ray domain.

The currently established method for distinguishing between enantiomers is circular dichroism (CD). In this approach, light with circular polarisation is sent through the sample. This light is absorbed to a different extent by the enantiomers. CD is

widely used in analytical chemistry, in biochemical research, and in the pharmaceutical and food industries. In CD, however, the signals are very weak: the light absorption of two enantiomers differs by just under 0.1%. There are various strategies for amplifying the signals, yet these are only suitable if the sample is available in the gas phase. Most studies in chemistry and biochemistry,
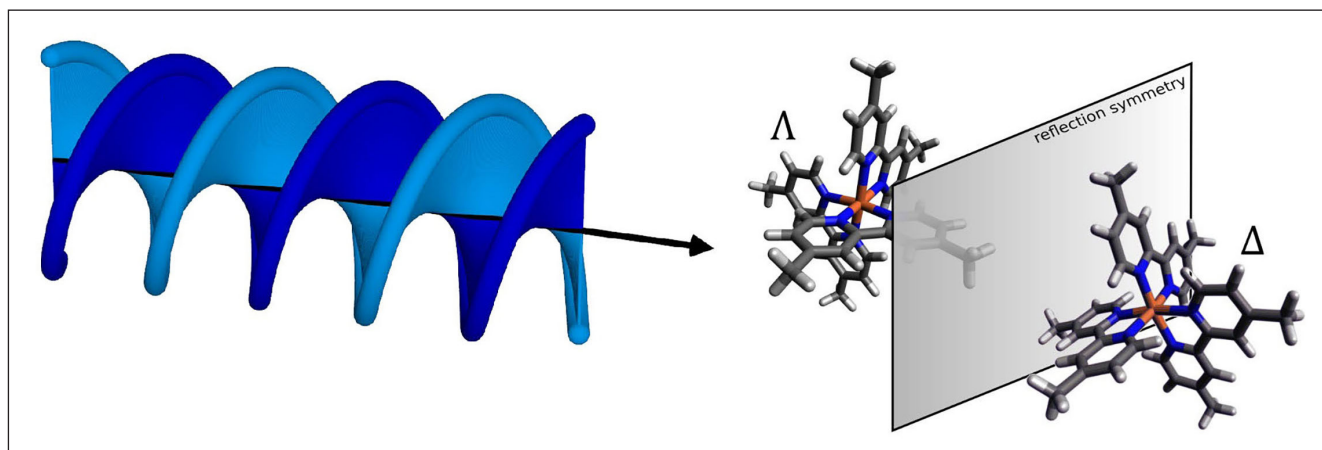
At the Swiss Light Source SLS at PSI, researchers have successfully shown that enantiomers can be distinguished from one another using helical X-ray light. Enantiomers are molecules that are mirror images of each other. Separating such molecules is relevant in biochemistry and toxicology, as well as in drug development. Graphic: Paul Scherrer Institute/Benedikt Rösner

however, are carried out in liquid solutions, mainly in water.

In contrast, the new method, published in *Nature Photonics* (doi. org/gqf4vs), exploits so-called helical dichroism (HD). The effect underlying this phenomenon is found in the shape of the light rather than its polarisation: the wavefront is curved into a helical shape.

At the Swiss Light Source SLS at PSI, the researchers were able for the first time to show successfully that enantiomers could also be distinguished from each other using helical X-ray light. At the cSAXS beamline of SLS, they demonstrated this on a sample of

the chiral metal complex iron-tris-bipyridine in powdered form, which the University of Geneva researchers had made available. The signal they obtained was several orders of magnitude stronger than what can be achieved with CD. HD can also be used in liquid solutions and thus fulfils an ideal prerequisite for applications in chemical analysis.

It was crucial for this experiment to create X-ray light with precisely the right properties. The researchers were able to accomplish this with so-called spiral zone plates, a special kind of diffractive X-ray lenses through which they sent the light before it hit the sample.

"With the spiral zone plates we were able, in a very elegant way, to give our X-ray light the desired shape and thus an orbital angular momentum. The beams we create in this way are also referred to as optical vortices", says PSI researcher Benedikt Rösner, who designed and fabricated the spiral zone plates for this experiment.

Jérémy Rouxel, an EPFL researcher, explains further: "Helical dichroism provides a completely new kind of light–matter interaction. We can exploit it perfectly to distinguish between enantiomers."

ICP-MS reveals use of recycled glass in ancient Greek villa

ICP-MS of glass tesserae from a villa from late antiquity indicates that some were made from recycled glass, as well as which elements were used for colouring.

Kaare Lund Rasmussen from University of Southern Denmark with Thomas Delbey from Cranfield University in the UK and the classical archaeologists Birte Poulsen and Poul Pedersen from Aarhus University and University of Southern Denmark have performed archaeometric analysis using inductively coupled plasma mass spectrometry (ICP-MS) of

19, approximately, 1600-year old mosaic tesserae.

The tesserae originate from an excavation of a villa from late antiquity, located in Halikarnassos (today Bodrum in Anatolia, Turkey). Halikarnassos was famous for King Mausolus' giant and lavish tomb, which was considered one of the seven wonders of the world.

The villa was laid out around two courtyards and the many rooms were adorned with mosaic floors. In addition to geometric patterns, there were also motifs of various mythological figures and scenes taken from Greek mythology; e.g. Princess Europa being abducted by the god Zeus in the form of a bull and Aphrodite at sea in her seashell. Motifs from the stories of

NEWS

Detail of mosaic floor. Credit: Kaare Lund Rasmussen/University of Southern Denmark

the much younger Roman author Virgil are also represented.

Inscriptions in the floor have revealed that the owner was named Charidemos and that the villa was built in the mid-fifth century.

Mosaic flooring was a costly luxury: expensive raw materials like white, green, black and other colours of marble had to be transported from distant quarries. Other stone materials, ceramics and glasses also had to be imported.

"I received 19 mosaic tesserae for analysis in my lab in Denmark. Of these, seven were of glass in different colours; purple, yellow, red and deep red. My conclusion is that six of them are probably made of recycled glass", says Kaare Lund Rasmussen.

This conclusion is based on chemical analysis with ICP-MS. With it, the research team has determined the concentrations of no less than 27 elements, some of them at a concentration of billionths of a gram. Their work has been published in *Heritage Science* (doi.org/gqjvbz).

"We were able to distinguish between base glass from Egypt and base glass from the Middle East and, also, we could determine which elements were added by the ancient craftsmen to colour the glasses and to make them opaque, which was preferred at the time", he says.

It is, of course, difficult to extrapolate from only seven glass mosaic tesserae, but the new results fit very well with the picture of Anatolia in late antiquity. As the power of the Roman Empire waned, trade routes were closed or rerouted, which probably led to a shortage of goods in many places—including raw materials for glass production in Anatolia.

This, together with the stories depicted on the floors, allows the classical archaeologists to put together a more detailed picture of what was fashionable in late antiquity and what the possibilities were for the artistic unfolding. ↰

Fusion of LIBS and acoustic data improves accuracy

Fusion of LIBS and acoustic data to analyse minerals under Terrestrial and Martian atmospheric conditions improves the performance.

A research team from the University of Málaga has validated the use of a system for more accurate detection of compounds in rocks by fusing different types of data obtained with laser-induced breakdown spectroscopy (LIBS). The research has been carried out in the laboratory simulating the atmospheric conditions on Earth and Mars and reported in *Analytical Chemistry* (doi.org/gqhw83).

LIBS uses a laser to transform the state of matter from solid to plasma. In just one millionth of a second, the system captures the emission of the elements that make up the sample. At the same time as the change of matter occurs, an acoustic wave originates from the detonation of the mineral. The experts involved in this study have fused spectral information and that provided by the propagation of sound to obtain more reliable data. They confirm that this model for the analysis of materials achieves a better definition of the compounds in less time and at a scale of analysis approaching the attogram.

In comparison with the results obtained with LIBS or the acoustic

NMR PRIMER:

AN HSQC-BASED APPROACH

(with vector animations)

by Steven M. Pascal

This book has one aim: to explain the key two-dimensional protein NMR experiment, the ^{1}H,^{15}N-HSQC, along with variants and extensions, in a generally accessible manner. Vector diagrams of one-, two- and three-dimensional pulse sequences are provided, along with accompanying animated versions. The animations allow the evolution of net magnetisation during the course of the experiments to be visualised and directly compared with the corresponding spin operator terms.

First, a brief introduction to spins, populations, the NMR experiment and relaxation is provided. Evolution due to J-coupling is next described and used to explain magnetisation transfer in the HSQC experiment and several variants. The extraction of structural, sequential and dynamic information is then illustrated via various extensions of the HSQC. Extensive footnotes and appendices introduce several more advanced concepts, such as sensitivity enhancement and the TROSY effect.

ANIMATIONS

The animations were originally created in Flash, which is no more. The animations have been converted to animated GIFs which enable them to be viewed easily with any browser. Control of these animations works best in Google Chrome using the GIF SCRUBBER extension: this allows pause/restart/reverse/speed control/etc.

BUY THE BOOK

NMR Primer: An HSQC-Based Approach costs just £24.95, plus postage & packing. This includes online access to the vector animations via an access code and password provided in each copy.

NMRPRIMER.IMPOPEN.COM
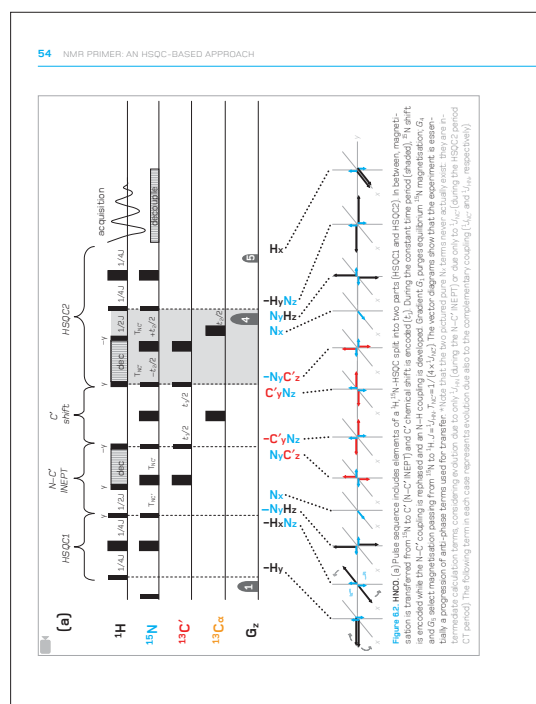
dataset separately, the results provided by the new system improve the information from 90 % and 77 %, respectively, to 92 % for Earth atmospheric conditions, and from 85 % and 81 % to 89 % for Mars.

"We demonstrate for the first time that the acoustic wave generated by the laser on the sample can be used to create a statistical descriptor and to improve the capacity of LIBS for rock differentiation", Javier Laserna, a researcher at the University of Málaga says. ⋏

Javier Laserna. Credit: Fundación Descubre
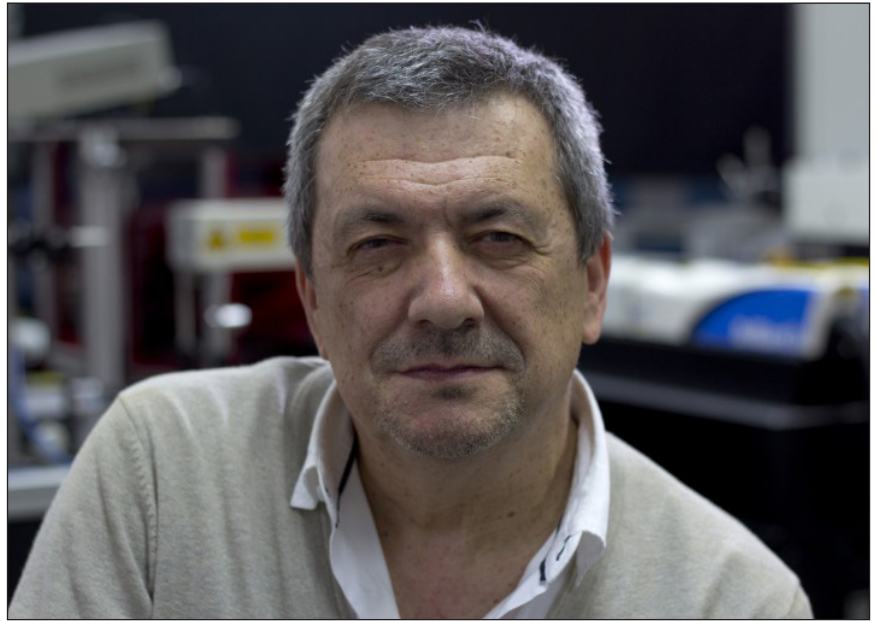
First NIR spectrum from the James Webb Space Telescope

The European Spave Agency (ESA) has released the first transmission spectrum made from a single observation using Webb's Near-Infrared Imager and Slitless Spectrograph (NIRISS) reveals atmospheric characteristics of the hot gas giant exoplanet WASP-96 b.

A transmission spectrum is made by comparing starlight filtered through a planet's atmosphere as it moves across the star, to the unfiltered starlight detected when the planet is beside the star. Each of the 141 data points (white circles) on this graph represents the amount of a specific wavelength of light that is blocked by the planet and absorbed by its atmosphere.

In this observation, the wavelengths detected by NIRISS range from 0.6 µm (red) to 2.8 µm (in the near infrared). The amount of starlight blocked ranges from about 13,600 parts per million (1.36 %) to 14,700 parts per million (1.47 %).

The grey lines extending above and below each data point are error bars that show the uncertainty of each measurement, or the reasonable range of actual possible values. For a single observation, the error on these measurements is remarkably small. The blue line is a best-fit model that takes into account the data, the known properties of
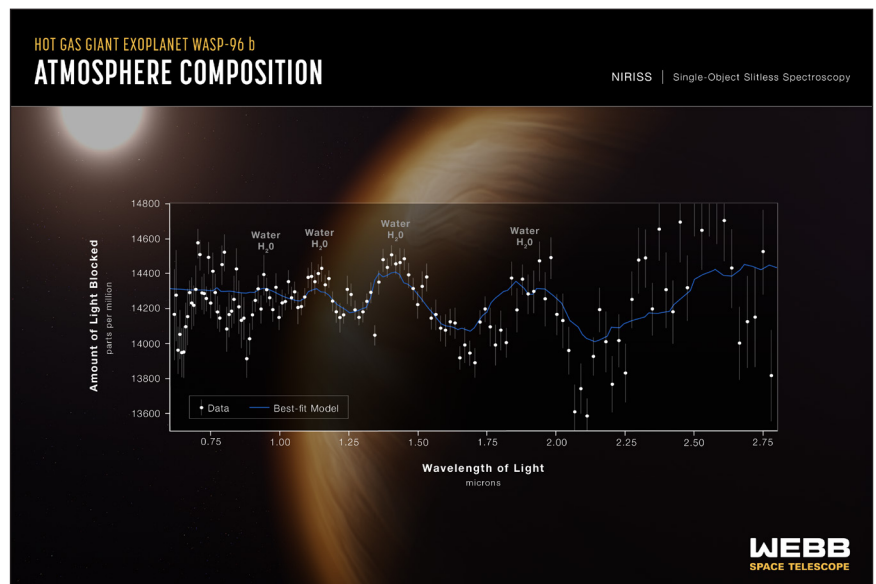
Image credit: NASA, ESA, CSA, STScI, and the Webb ERO Production Team

WASP-96 b and its star (e.g., size, mass, temperature) and assumed characteristics of the atmosphere. Researchers can vary the parameters in the model—changing unknown characteristics like cloud height in the atmosphere and abundances of various gases—to get a better fit and further understand what the atmosphere is really like. The difference between the best-fit model shown here and the data

simply reflects the additional work to be done in analysing and interpreting the data and the planet.

Although full analysis of the spectrum will take additional time, it is possible to draw a number of preliminary conclusions. The labelled peaks in the spectrum indicate the presence of water vapour. The height of the water peaks, which is less than expected based on previous observations, is evidence for the presence of clouds that suppress the water vapour features. The gradual downward slope of the left side of the spectrum (shorter wavelengths) is indicative of possible haze. The height of the peaks along with other characteristics of the spectrum is used to calculate an atmospheric temperature of about 725 °C.

This is the most detailed infrared exoplanet transmission spectrum ever collected, the first transmission spectrum that includes wavelengths longer than 1.6 μm at such high resolution and accuracy, and the first to cover the entire wavelength range from 0.6 μm to 2.8 μm in a single shot. The speed with which researchers have been able to make confident interpretations of the spectrum is further testament to the quality of the data.

The observation was made using NIRISS's Single-Object Slitless Spectroscopy (SOSS) mode, which involves capturing the spectrum of a single bright object, like the star WASP-96, in a field of view.

WASP-96 b is a hot gas giant exoplanet that orbits a Sun-like star roughly 1150 light years away, in the constellation Phoenix. The planet orbits extremely close to its star (less than 1/20th the distance between Earth and the Sun) and completes one orbit in less than 3½ Earth-days. The planet's discovery, based on ground-based observations, was announced in 2014. The star, WASP-96, is somewhat older than the Sun, but is about the same size, mass, temperature and colour.

The background illustration of WASP-96 b and its star is based on current understanding of the planet from both NIRISS spectroscopy and previous ground- and space-based observations. Webb has not captured a direct image of the planet or its atmosphere.

NIRISS was contributed by the Canadian Space Agency. The instrument was designed and built by Honeywell in collaboration with the Université de Montréal and the National Research Council Canada.

Highly-sensitive SERS probes detect the PD-L1 biomarker

Ultrasensitive biosensors based on SERS have been developed to detect the cancer metastasis related programmed death ligand (PD-L1) biomarker.

Recently, a team led by Prof. Huang Qing at the Institute of Intelligent Machines, Hefei Institutes of Physical Science (HFIPS) of the Chinese Academy of Sciences (CAS) has reported the fabrication of ultrasensitive biosensors based on surface-enhanced Raman spectroscopy (SERS) to detect the cancer metastasis related programmed death ligand (PD-L1) biomarker. In this research, scientists fabricated highly sensitive and specific aptamer-functionalised probes based on Au/TiO$_2$/Fe$_3$O$_4$ (shell/core) magnetic nanocomposites and Ag/4-ATP/Au (shell/core) SERS nanotags.

Using the "sandwich" approach, they captured the malignant exosomes between magnetic
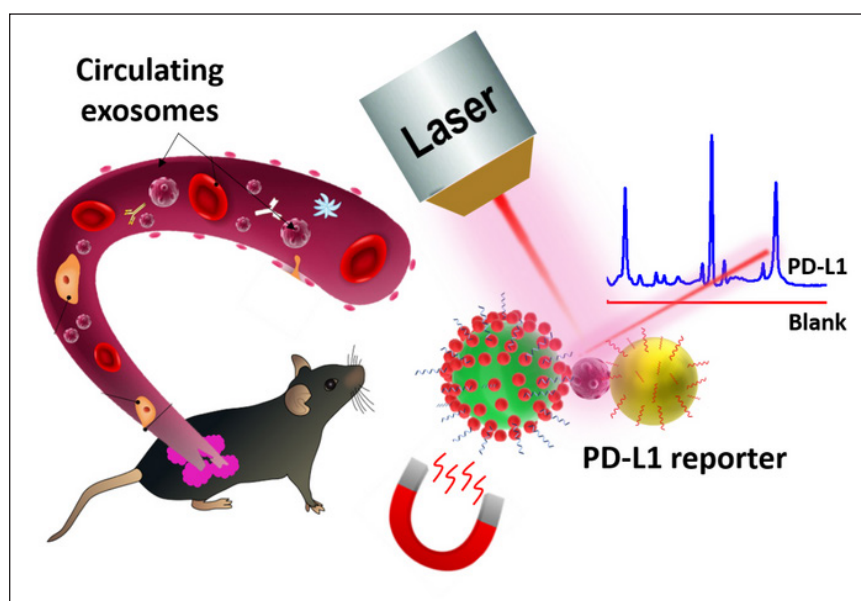
Schematic representation of circulating exosomal PD-L1 detection using SERS-sandwich. Credit: Muhammad

NEWS

nanocomposites and SERS nano-tags with which they could quantitatively measure the PD-L1 biomarker as low as $4.31\,ag\,mL^{-1}$ by analysing the Raman report signals.

In the mice model, the researchers confirmed that the proposed technique could be useful in analysing time dependent growth of tumours by analysing enhancement in PD-L1 expression in the tumour. Moreover, the researchers demonstrated the applicability of their work by integrating nanoparticles probes with a portable Raman spectrometer to realise the PD-L1 measurement with 95 % sensitivity.

Overall, the outcome of this work, published in *Biosensors and Bioelectronics: X* (doi.org/gqfn2d), demonstrated the great clinical significance of PD-L1 biomarker diagnosis which in future would be helpful in monitoring patients' health who undergo PD-L1/PD-1 immunotherapy. ↗

ANISG Tony Blakeney Award

Professor Kerry Brian Walsh is to receive the prestigious ANISG Tony Blakeney Award for 2022.

Professor Kerry Brian Walsh is to receive the prestigious Tony Blakeney Award for 2022 from the Australian Near Infrared Spectroscopy Group (ANISG). Kerry has been a pioneer in the development and application of NIR spectroscopy. As an academic, he has shown leadership in teaching and mentoring students at undergraduate and post graduate levels and productive collaboration with Australian and international scientists. His research is wide ranging and cutting edge and includes contributions to the understanding of the basic principles relating to NIR spectroscopy. Of equal importance, are his contributions to rural industries by pioneering the introduction of NIR spectroscopy in the horticultural areas of Australia.

The benefits of Kerry's work have international impact through contributions at major international conferences and international aid programs. In his own words, *"I believe that my R&D effort should result in a gain to society..."*. A current research project led by Kerry is *"Feasibility and commercial viability*

Professor Kerry Walsh is to receive the 2022 ANISG Tony Blakeney Award.

of voluntary carbon standards and methods for farmers" indicates that Kerry tackles current issues.

Kerry's leadership and many contributions to students, colleagues and fellow scientists and his ability to apply his scientific knowledge to the good of rural industries and food security are attributes which align very closely with those of the late Tony Blakeney whom this award honours. ↗

NEWS

ICP-MS reveals use of Bruker launches NMR-based research tool for Long COVID

An NMR-based Molecular Phenomics Clinical Research Tool for "Long COVID" multi-organ risk assessment uses a multiplexed combination of biomarkers.

Bruker has launched PhenoRisk PACS™ RuO, a research-use-only NMR test for molecular phenomics research on "Long COVID" patients' blood samples, using a multiplexed combination of biomarkers discovered at the Australian National Phenome Center (ANPC) at Murdoch University, under Director Jeremy Nicholson. The PhenoRisk PACS™ RuO test is promising for research on early-stage risk factors, on longitudinal recovery monitoring and on potential secondary organ damage in cardiovascular disease, type II diabetes, kidney dysfunction and inflammation. It consists of an Avance™ IVDr NMR spectroscopy system that tests several biological mechanisms of action simultaneously in a 20-minute blood-test from SARS-CoV-2 acute infection survivors who suffer long-term sequelae, termed either Post-Acute COVID Syndrome (PACS), or commonly known as "Long COVID" syndrome.

Recent publications have demonstrated that NMR is playing a pivotal role in measuring COVID-19 progression based on phenomic molecular signatures, and it is also providing new insights into various PACS mechanisms of action, as well as into Long COVID clinical management and treatment options. The PhenoRisk PACS™ RuO test characterises metabolic and proteomic biomarkers of SARS-CoV-2 pathobiology, even in asymptomatic acute COVID cases, thereby enabling multi-organ risk assessment, recovery and therapeutic research in multiple Long COVID dysfunctions.

This new NMR test has the potential to quantitatively discriminate PACS patients from healthy or fully recovered individuals by studying COVID-triggered pheno-conversion, defined as transient or persistent systemic change of the molecular signatures in human plasma samples after acute infection. Subsequent pheno-reversion of metabolic signatures detected by PhenoRisk PACS™ RuO may indicate PACS recovery.

In addition to metabolism analytes, PhenoRisk PACS™ RuO quantifies composite signals for groups of glycoproteins and phospholipids, which can indicate inflammation and cardiovascular disease risk, as well as a new NMR biomarker called supramolecular phospholipid composite (SPC). These markers show excellent discrimination of COVID-19 from controls, while the Glyc/SPC ratio has been proposed as a useful molecular marker for Long COVID, which could significantly augment current clinical and therapeutic research.〰

NIR spectroscopy algorithm to identify authenticity of crop varieties

A Chinese team has developed a new algorithm for near infrared spectroscopy, suitable for high-throughput identification of the authenticity of crop varieties.

Recently, the crop quality intelligent perception team of Hefei Institute of Intelligent Machinery, Hefei Institutes of Physical Science (HFIPS) of Chinese Academy of Sciences (CAS) has developed a new algorithm for near infrared (NIR) spectroscopy, suitable for high-throughput identification of the authenticity of crop varieties.

The authenticity of crop varieties is of great significance in variety protection and seed breeding. Traditional methods for authentic identification of crop varieties, such
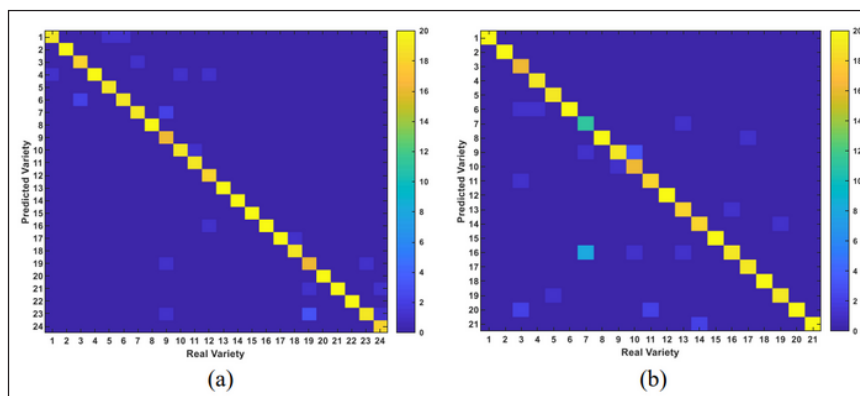
Heat maps of the confusion matrices of wheat (a) and rice (b) sample sets identified by the InResSpectra neural network. Credit: XU Zhuopin

as DNA molecular identification, isoenzyme identification and field identification, have the disadvantages of complicated operations, time-consuming, sample damage, environmental pollution and slow detection results, so an effective method is urgently needed to realise the authenticity identification of crop varieties.

As a rapid detection technology, NIR spectroscopy, has many advantages. It is environmentally friendly, sensitive and non-destructive. In this research, a self-developed high-throughput seed quality sorting instrument based on NIR spectroscopy, made by the team, can achieve rapid identification and sorting of individual seeds. Based on this instrument, researchers proposed an improved convolution neural network (CNN)—the InResSpectra network—to help achieve more accurate seed variety identification. This is an optimised Inception network and successfully removed the 1×1 convolution branch to reduce the complexity of the model, and increased the residual element of the ResNet network, which accelerated the training of the neural network and improved accuracy.

Researchers applied the developed system and the InResSpectra network for the identification of 24 wheat varieties and 21 rice varieties, and achieved 95.35 % and 93.07 % accuracy, respectively, which provided an effective method for the spectroscopic identification of the authenticity of crop varieties. Their work was published in *Infrared Physics* (doi.org/gqfjdc).↗

Nominations for IRDG Norman Sheppard Award 2022

Nominations for the IRDG's 2022 Norman Sheppard Award are invited.

The Norman Sheppard Award is made by the Infrared and Raman Discussion Group (IRDG) to honour scientists who have significantly advanced the field of vibrational spectroscopy while working in academia or industry. Recipients will have made a sustained and distinguished contribution to the field, and will also have strongly supported the aims and activities of the IRDG.

Nominations for the 2022 Award are invited and should be e-mailed to neil.everall@btinternet.com, and copied to awards@irdg.org, by 31 October 2022. Any current member of the IRDG may nominate a candidate for this award. Full details of the nomination procedure can be found at http://www.irdg.org/awards/norman-sheppard-award/details/.↗

Norman Sheppard FRS receiving the first Award from Geoff Dent.

ARTICLE

Atomic archaeology? Using portable gamma surveying techniques to identify buried archaeological features

Victoria Robinson

School of Archaeology, Geography and Environmental Science, University of Reading, Reading, UK

The nuclear industry and the field of archaeology could hardly be described as "two sides of the same coin". Perhaps not even the same currency. However, early research suggests that technologies widely used in the nuclear industry could offer valuable support to the non-intrusive identification of buried archaeological features which provide a brand-new insight into historic sites and ways of life.

A collaborative project between the University of Reading and Nuvia Limited is investigating the application of portable gamma radiation survey methods at sites of historical interest, which has to date yielded some interesting results.

The story so far

Gamma radiation surveys at several key locations within the Roman Town of Silchester, Hampshire, UK, was successfully completed[1] using Nuvia's Groundhog® system (Box 1). The site was selected due to the extensive geophysical data already available for the site, against which the radiation survey data

could be compared. Further, it was recognised that Silchester offered a range of target types on which to test the Groundhog® system.

The purpose of this preliminary investigation was to explore whether radiation surveying could form an additional useful "tool" in the toolbox of existing geophysical technologies used in archaeological exploration.

Four areas were surveyed, including areas of urban and industrial activity, a temple complex and an area containing cremations and suspected inhumations. The most exciting results relate to a survey of the Temple Complex at the site. Figure 1 compares the results from a traditional geophysical survey (Figure 1a) against the gamma survey data (Figure 1b) from the same area. The figure shows that the gamma survey was capable of measuring subtle differences in the radiochemical composition of
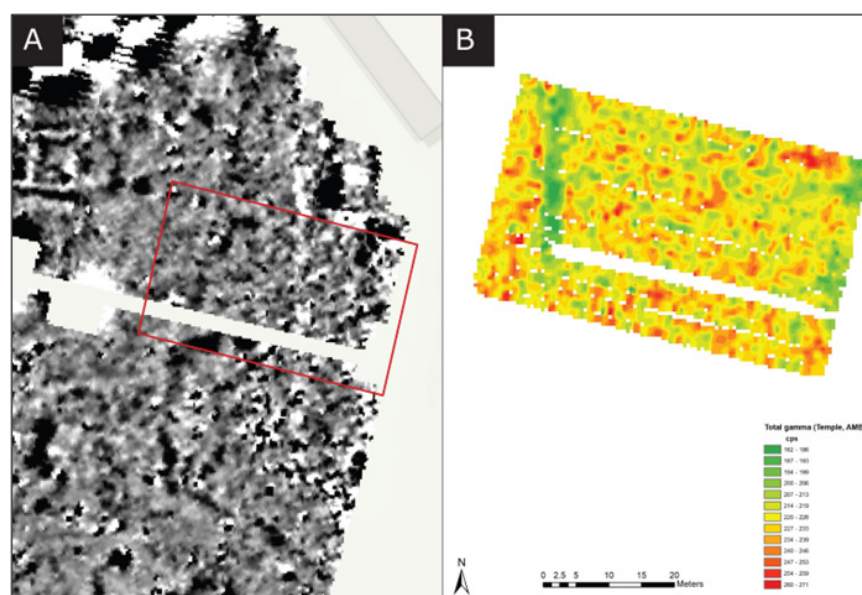
Figure 1. Comparison of fluxgate gradiometry data (± 5 nT) (a) against total gamma radiation data (b) collected at the Temple Complex. Uncollimated survey data are displayed in cps. A clear linear anomaly of depleted radioactivity can be seen in the left-hand side of the survey area. This aligns with a feature visible in the fluxgate gradiometry data, which is known to be a Temenos wall. Reproduced from Reference 1 under a CC BY licence. Geophysics data from Reference 2.
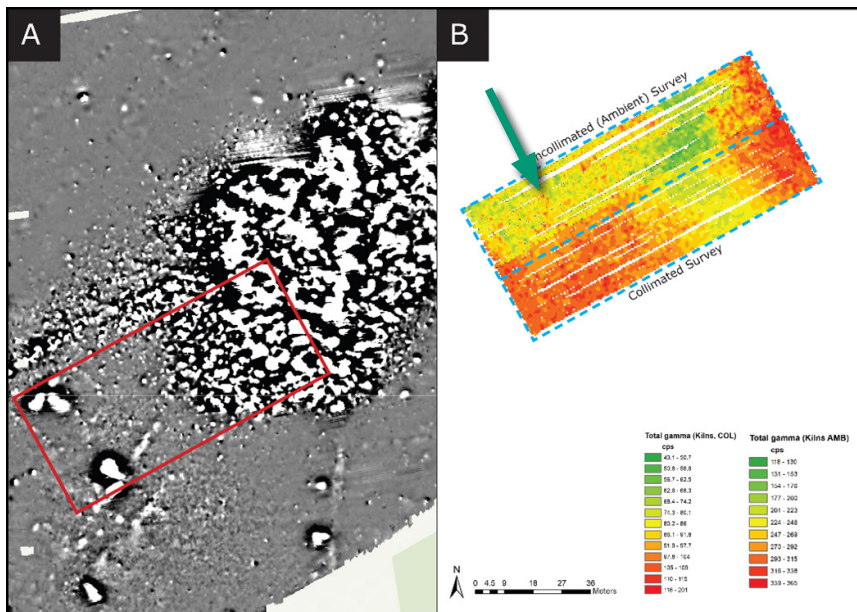
Figure 2. Comparison of caesium magnetometry data (± 7 nT) (a) against total gamma radiation data (b) collected at Little London. Both collimated and uncollimated measurements are presented. Radiation data are displayed in cps. An area of depleted radioactivity in the upper half of the Groundhog® survey area aligns with the clear anomaly present in the geophysics data. A "P"-shaped anomaly (arrowed) in the top-left corner of the survey area broadly aligns with one of the kilns but is assumed to be naturally occurring or a feature of interpolation. Reproduced from Reference 1 under a CC BY licence. Geophysics data from Reference 3.

the Temenos (temple) wall bounding the Temple Complex and the surrounding substrate. The feature, which appears as a linear area of depleted radioactivity on the left-hand side of the image, aligns with similar anomaly in the flux-gate gradiometry survey data from the same area, suggesting that this method could indeed support archaeological surveys.

Surveys at the other areas targeted at Silchester yielded mixed results. A radiation survey completed at nearby Little London, known to be an area of industrial activity during the Roman period, was able to identify the location of a modern infilled claypit (Figure 2b). Faint linear features present in a section surveyed with the detector in a collimated configuration may be attributable to kilns identified in the geophysical data (Figure 2a). However, this is uncertain and may be attributable to interpolation of the data or a natural occurrence.

Radiation surveys at the burial site and area of urban activity did not generate any anomalies as identified in the geophysical data, as had been expected.

How does radiation surveying work in an archaeological context?

Typical geophysical surveying methods look for and measure differences in the physical properties of potential features of interest and the surrounding substrate. This includes for example magnetism and electrical resistance and dielectric permittivity. Similarly, when completing radiation surveys of archaeological sites, we are looking to measure subtle variations in the concentrations of naturally occurring radioactivity present in potential targets and the surrounding soil. Due to their abundance in Earth's crust, long half-lives, ability to accumulate within materials of interest and ability to generate gamma rays of sufficient energy and intensity to support gamma ray mapping, this research has identified ^{40}K, ^{238}U and ^{232}Th as the radionuclides of interest.

There are multiple mechanisms that can cause measurable differences in the concentrations of radioactivity within targets. These are summarised in Figure 3. Of particular interest in the field of archaeology is the historic use of imported construction materials, and the concentration of radioactivity in these materials. Construction materials have throughout history, been transported over significant distances to a desired location or settlement. This is observed in many iconic historical monuments such as the Welsh blue stones of Stonehenge or the Dorset-sourced marbles

The author surveying

ARTICLE

Box 1

What is Groundhog®?

Groundhog is a family of proprietary radiation detection systems designed, owned and operated by Nuvia Limited. These systems are well established within the nuclear industry. They are used for detecting and quantifying the nature and extent of radioactive material in support of various tasks including reassurance surveys, site clearance, remediation planning and particle detection. The basic system comprises a sodium iodide detector, gamma spectrometer, GPS system, supporting electronics and hand-held data logger/ultra-mobile PC processing unit.

The system can be scaled to suit the nature and size of the project, with hand-held systems suitable for smaller areas of tens of square metres through to vehicle mounted systems capable of surveying several hectares in a day. Both methods of application are capable of achieving a high density of measurements: >15,000 measurements per day for hand-held systems and >50,000 per day for vehicle mounted system.

Radiation and GPS data are continuously recorded and processed to generate high quality outputs including spectral data and visual outputs that support interpretation of data and rapid identification of areas of interest.

of Westminster Abbey. These imported materials typically have a different geochemical composition to the local geology. Where imported materials are present in sufficient quantities, the difference in gamma signatures should be measurable. The process of making construction materials such as brick can also lead to the concentration of naturally occurring radioactivity. For example, following the removal of water, air and other volatile components during the forming and firing of clay bricks. Sufficient accumulation of these bricks could also lead to a measurable difference in radioactivity concentrations. Historic industrial activities such as the mining and processing of ores has been and continues to be a notable source of technologically enhanced naturally occurring radioactive material. Finally, the ability of uranium, in the form of uranyl to exchange with calcium within the apatite structure of bone during fossilisation can also lead to the accumulation of radioactivity within fossilised bone material.
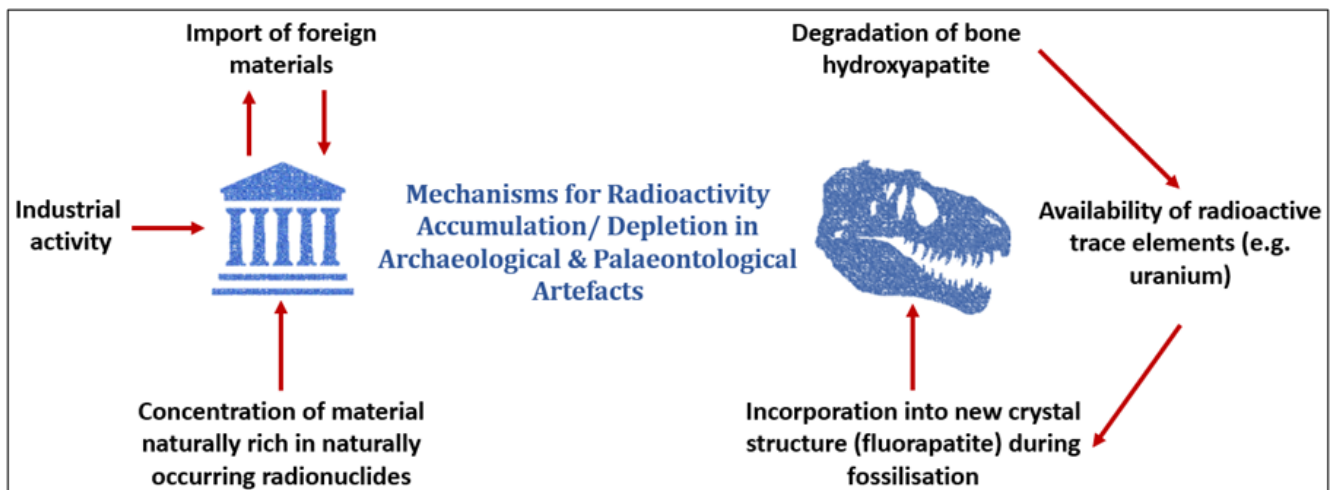
Figure 3. Mechanisms for accumulation/depletion of naturally occurring radioactivity in buried archaeological and palaeontological artefacts.

Why could this approach add value to archaeological prospection?

The effectiveness of current geophysical techniques is dependent on the ability to measure clear differences in the physical properties of potential targets and surrounding substrate. This ability can be influenced by several factors including the presence of nearby metallic structures, presence of salt water or a high water-table, local geology and the type of target suspected. In consequence, there is no geophysics panacea. Rather, consideration must be given to the physical and chemical properties of the suspected target(s) and surrounding substrate, target size, amount of overburden and nearby infrastructure to inform optimal selection of an optimal geophysical method for each site. Application of a limited range of contrasting techniques at a site can offer multiple benefits including: mitigating the effects of interference, improving data fidelity and subsequent interpretation.

In consequence, having a "toolbox" of available techniques to apply to a site of archaeological interest can be a useful mechanism for overcoming known vulnerabilities. Current research is, therefore, continuing to explore whether portable gamma radiation surveying could offer a valuable contribution to this toolbox.

It is recognised that this technique has its own set of limitations. For example, at sites where archaeological features are constructed from very local materials offering no contrast in radionuclide composition. It is also acknowledged that any concentrations of naturally occurring radioactive material could be readily shielded by overburden, particularly when present at depths of more than 40–50 cm,

or high soil moisture content. Despite this, completion of gamma radiation surveys at archaeological sites would offer unique advantages and limitations that contrast to those observed for more traditional geophysical techniques. For example, gamma surveying techniques will not be susceptible to interference from metallic structures and electrical lines. Further, the spectrometric capability of the system deployed within this research project supports identification of key radionuclides present within features of interest which could support site characterisation.

Conclusion

The results from this preliminary study are exciting and have raised some interesting questions. For example, why clear anomalies were observed at some sites and not others, and how the surveying method could be revised to improve data quality—such as through increasing sampling density. A number of fieldwork exercises are planned in the coming months to trial alternative survey methods and collect additional data through the sampling and analysis of soil and artefacts. This will help us address these questions and better understand what, if any, role radiation surveys could play in archaeological

exploration. This work has also highlighted the importance and value of cross-industry knowledge sharing—including the potential for opening up entirely new fields of research.

For more information on the Silchester study, the original paper[1] is open access.

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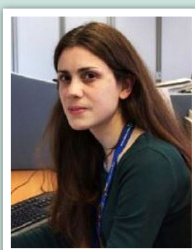
Victoria is a Chartered Environmentalist who graduated from Oxford Brookes University with an MSc in Environmental Management in 2006. She then pursued a career with Nuvia as a Radioactive Waste and Environmental Management Consultant, accruing over 15 years' nuclear industry experience. In this time, she has worked within multi-disciplinary teams for a diverse range of projects in the UK and overseas. In 2018 Victoria embarked on a part-time PhD with the University of Reading. Her research project explores the efficacy of gamma-radiation surveying for detecting artefacts of potential archaeological/palaeontological interest. It has provided a unique opportunity to combine elements of her day job with a life-long interest in natural history.

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Overcoming the challenges of reducing background interference for LC/MS/MS trace PFAS analysis

The path to achieving reliable results

Jamie Foss, Cole Strattman and Simonetta Tumbiolo

PerkinElmer, Inc.

Per- and polyfluoroalkyl substances (PFASs) represent a large group of thousands of anthropogenic compounds that have been produced and widely used in industrial applications and consumer products since the 1950s. Some of the major industry sectors using PFASs include aerospace and defence, automotive, aviation, food contact materials, textiles, leather and apparel, construction and household products, electronics, firefighting, food processing, and medical articles.

These compounds have unique physical and chemical characteristics: they all contain carbon–fluorine bonds (among the strongest chemical bonds in organic chemistry), that means they are highly stable and resistant to degradation. All PFASs are highly persistent in the environment. In fact, they are known to persist in the environment longer than any other man-made substance. This, along with their ubiquitous use, have led to the accumulation of PFAS in the environment, with growing concern

of human exposure to these chemicals.[1–3] Most PFASs are also easily transported in the environment covering long distances from the source of their release.

Many PFAS are found in human and animal blood and are present at low levels in a variety of food products and in the environment all over the globe. PFAS are found in soil and a number of water resources, including drinking, surface, ground- and wastewater.[1–6]

Among PFASs, perfluorooctane-sulphonic acid (PFOS) and perfluorooctanoic acid (PFOA) have been the most prevalent in the environment and, having attracted the most attention, they have been included in many advisory guidelines. For example, the United States Environmental Protection Agency (US EPA) issued a health advisory of 70 parts per trillion (ppt, equivalent to $70\,ng\,L^{-1}$) for PFOA and PFOS in drinking water.[7]

The European Chemicals Agency (ECHA) and the European Commission have, since 2014, screened data on PFASs contained in ECHA's registration database and coordinated group-based regulatory work. The Drinking Water Directive, which took effect on 12 January 2021, includes a limit of $0.5\,\mu g\,L^{-1}$ for all PFAS.[8]

The optimisation of analytical methods for identification and quantification of PFASs is essential for risk assessment. Because of its high sensitivity, selectivity and robustness, the most widely used analytical method for PFAS detection is based on Liquid Chromatography coupled with tandem Mass Spectrometry (LC/MS/MS). Many agencies have developed methods for the analysis of PFAS in environmental matrices. Among them:

- **US EPA Method 537.1** for the determination of 18 PFASs in drinking water. The LC/MS/MS method is based on isotopic internal standards with reversed-phase solid phase extraction (SPE) sample preparation.[9]

- **US EPA Method 8327**, designed to measure a group of 24 PFAS compounds in ground, surface and wastewater samples, uses LC/MS/MS with external calibration.[10]

- **US EPA Method 533** addresses some shorter chain and more polar PFAS compounds. It contains a list of 25 PFAS compounds (C_4–C_{12}) comprising the majority of those in 537.1, with the addition of some polar fluorotelomers and ether carboxylic acids. The LC/MS/MS method uses isotopic dilution and ion exchange SPE sample preparation.[11]

- **US EPA Draft Method 1633** was introduced in 2021 and is

for use in the Clean Water Act (CWA). It is a composite method for a broad range of PFAS in multiple matrices. The method encompasses 40 targeted PFAS compounds in various matrices, including aqueous, solids, biosolids and tissues.[12]

- **ISO 25101:2009** is utilised for the determination of PFOS and PFOA in unfiltered samples of drinking, ground- and surface water by coupling SPE with LC/MS/MS.[13]

For the determination of low levels of PFAS, it is necessary to utilise either a highly sensitive mass spectrometer, or a sample preparation technique that includes a concentration step. Coupling SPE with LC/MS/MS has been one of the most popular approaches to PFAS analysis in aqueous samples, and has been employed in EPA Method 537.1, as well as ISO 25101.

Sample preparation techniques differ according to the sample type. However, the key challenge of measuring ppt levels of PFAS is that these compounds are ubiquitous throughout the environment and accumulate everywhere, including the laboratory equipment and accessories. In fact, many of the components used in liquid chromatographs, mass spectrometers and SPE systems are made of polytetrafluoroethylene (PTFE) or PTFE copolymers, which leach PFAS compounds and cause background interference during a sample measurement. Even the use of glass sample containers can generate additional challenges, the glass in fact adsorbs PFAS compounds. Special care is required, and alternate materials must be used throughout the laboratory in order to reduce the absorption of these chemicals.

Reducing PFAS background

To reach accurate ultra-trace levels, every step of the analytical protocol must be free of PFAS materials, from sample collection to sample preparation, analysis and measurement. Table 1 summarises the necessary steps for reducing background contamination during sample preparation and analysis.

Mobile phase and HPLC pump

High-quality mobile phases and blank runs are important aspects of the analysis. Additionally, instead of utilising conventional glass vials with PTFE-lined septa, polyethylene vials and caps are necessary to reduce the possibility of contamination. The HPLC pump, autosampler and SPE system all contain PFAS components that require mitigation as well.

Customarily, the pump of an HPLC system has PTFE parts that can leach PFAS compounds. Moreover, contamination is likely in all but the highest grades of reagents. To combat interference from these sources, a delay column may be installed in the flow path between the pump and the autosampler, as shown in Figure 1.

A delay column captures PFAS contaminants coming from the mobile phase, the solvent lines or the pump before they reach the autosampler. As a result, the captured compounds elute via the gradient later than the analyte peak in the sample.

The chromatograms to the right of the schematic show examples of the delay peak eluting after the analyte peak. Thus, installation of a delay column allows clear separation of PFAS contaminants from the analytes of interest, enabling more authentic measurements of PFAS in the sample.

Table 1. Steps to reduce PFAS contamination.

Source of contamination	Mitigation
Mobile phases	Purchase LC/MS grade solvents Use a delay column
PFAS parts and tubing in HPLC pump	Use a delay column
PFAS tubing in HPLC autosampler	Replace with PEEK tubing
Vials and PTFE-lined caps	Use only polyethylene vials and caps
PFAS tubing in SPE apparatus	Replace with polyethylene tubing

Figure 1. Reducing background from pump and mobile phases.

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Autosampler

In many cases, the HPLC autosampler contains fluoropolymer tubing which will introduce contamination upon injection of the sample. It is recommended to replace all tubing with high performance polyether ether-ketone (PEEK) to eliminate the possibility of PFAS contamination during sample injection. For convenience, PerkinElmer offers a kit specifically developed to replace the tubing in their autosamplers for PFAS applications.

Solid Phase Extraction (SPE) system

SPE extraction configurations normally include an abundance of fluoropolymers. The tubing connecting sample bottles to the SPE cartridges can be a significant source of PFAS contamination. Replacement of all transfer tubing with linear low-density polyethylene (LLDPE) or PEEK tubing is necessary to avoid PFAS leaching. In addition, some of the valving on the manifold may be constructed of PTFE; substitution with polyethylene stopcocks is recommended. Finally, sample collection during SPE extraction should employ polyethylene centrifuge tubes.

After proper mitigation of all possible PFAS sources, the LC/MS/MS system will be ready to analyse PFAS at low parts per trillion levels.

Example: Validation study using EPA 537.1

A recent study validated PerkinElmer's PFAS mitigative steps by employing EPA Method 533 and EPA Method 537.1 on a QSight® 220 LC/MS/MS system (Figure 2). First, a 250 mL drinking water sample was collected in a polyethylene bottle. Next, the method involved fortification with surrogates to monitor the extraction efficiency. The sample was then concentrated by SPE using a polystyrenedivinylbenzene (SDVB) stationary phase. In this step, the sample was loaded onto the SPE

Figure 2. PerkinElmer QSight® 220 LC/MS/MS triple quadrupole system.

tube and eluted with methanol. The extract was then evaporated to dryness under nitrogen and reconstituted in 1 mL of 96 % methanol. This concentrated the sample by a factor of 250, thereby enabling quantification of the low levels necessary for the analysis. Internal standards were added after reconstitution of the sample.

Subsequently, 10 µL of sample was injected onto a C18 column in the LC/MS/MS instrument. The analytes were separated in the LC column and eluted into the mass spectrometer, which was used in Multiple Reaction Monitoring (MRM) mode. The retention times for the calibration standards enabled identification of the compounds and the MRM

transitions, for both quantifier and qualifier ions.

Separation

EPA Method 537.1 describes a chromatographic technique that takes approximately 37 minutes to separate the 18 analytes, surrogates and internal standards. However, improvements to the chromatographic method made by PerkinElmer scientists achieved a run time of about 10 minutes. This represented significant time savings while maintaining excellent chromatographic resolution, and excellent separation of the linear and branched isomers. Examples of their separation are shown in Figure 3.

Calibration

Calibration curves were run for all 18 analytes and the surrogate standards, encompassing the range necessary to include the lower limits of detection (LOD) from EPA regulations. The full method ranged from 0.02 ppt to 120 ppt. As demonstrated in Table 2, excellent linearity was observed, with all correlation coefficient (R^2) values for the calibration curves of 0.99 or better.

Sensitivity

In terms of instrument sensitivity, the limits of quantitation (LOQ) and LOD were estimated based on signal-to-noise ratios.
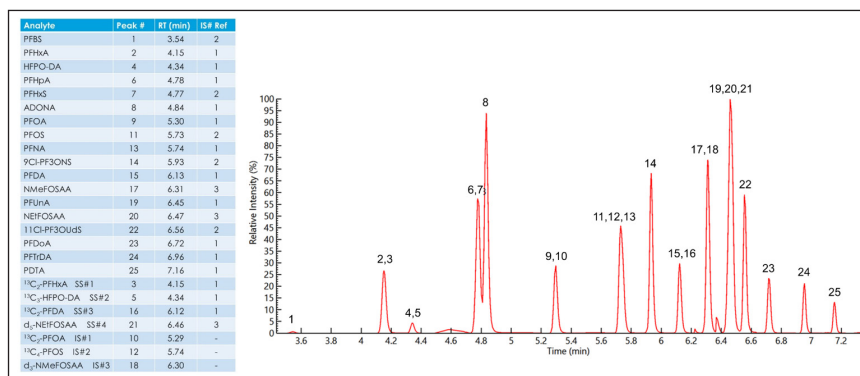
Figure 3. Total ion chromatogram of an 80 ng L^{-1} extracted fortified laboratory field blank sample containing all method analytes, surrogates and internal standards.

Table 2. Instrument and method calibration ranges and linearity (R2) for eight-point calibration curves of all EPA Method 537.1 analytes and surrogates.

Compound	Instrument calibration range (ng L^{-1})[a]	Method calibration range (ng L^{-1})[b]	$R^{2\,c}$
PFBS	16.4–26287	0.07–105.1	0.9994
PFHxA	5.5–29703	0.02–118.8	0.9987
$^{13}C_2$-PFHxA	4.6–24752	0.02–99.0	0.9989
$^{13}C_3$-HFPO-DA	67.5–24752	0.27–99.0	0.9992
HFPO-DA	18.5–29703	0.07–118.8	0.9985
PFHpA	5.5–29703	0.02–118.8	0.9984
PFHxS	5.2–28218	0.02–112.9	0.9998
ADONA	5.2–28218	0.02–112.9	0.9990
PFOA	5.5–29703	0.02–118.8	0.9998
PFOS	5.3–28515	0.02–114.1	0.9974
PFNA	18.5–29703	0.07–118.8	0.9993
9Cl-PF3ONS	5.1–27772	0.02–111.1	0.9998
PFDA	81.0–29703	0.32–118.8	0.9990
$^{13}C_2$-PFDA	4.6–24752	0.02–99.0	0.9988
NMeFOSAA	5.5–29703	0.02–118.8	0.9998
PFUnA	18.5–29703	0.07–118.8	0.9968
NEtFOSAA	5.5–29703	0.02–118.8	0.9968
d5-NEtFOSAA	18.3–99010	0.07–396.0	0.9962
11Cl-PF3OUdS	5.2–28069	0.02–112.3	0.9997
PFDoA	18.5–29703	0.07–118.8	0.9963
PFTrDA	5.5–29703	0.02–118.8	0.9959
PFTA	5.5–29703	0.02–118.8	0.9967

[a]Instrument calibration range is the actual concentration range of calibration standards used to determine calibration curves.
[b]Method calibration range is determined by multiplying the instrument calibration range by 1/250 to account for the SPE sample preparation/concentration.
[c]R^2 values are the average of triplicate calibration curves.

Data reported in Table 3 confirm that the QSight® 220 LC/MS/MS system is highly capable of performing the method successfully. With the 250-to-1 sample concentration from the SPE extraction step, the limits were well below the current requirements for all compounds, even those at extremely low levels.

Experiments were conducted in order to define the method detection limits of all target analytes for EPA Method 537.1. The lowest concentration minimum reporting limits (LCMRLs) as well as the experimental minimum reporting limits (MRLs) were also determined. Results are tabulated in Table 4. Experimental MRLs are at acceptable levels to meet the current requirements for all the targeted PFAS compounds.

Recovery

Recovery studies were completed for all 18 analytes by spiking fortified laboratory field blanks at four different levels, ranging from 0.3 ppt up to 80 ppt. Figure 4 shows the recoveries for each analyte at each of the four concentrations. EPA Method 537.1 requires recoveries between 70 % and 130 % of the known spiking level. The developed method using the QSight® 220 LC/MS/MS met requirements for recovery across all four concentrations evaluated.

Conclusion

LC/MS/MS analysis of PFAS at ultra-trace levels requires mitigation to both liquid chromatograph and mass spectrometer to eliminate the leaching of fluorochemicals from components within the systems. Manual SPE configurations also require mitigative steps to eliminate any components constructed of PTFE to minimise or eliminate any PFAS contamination. PerkinElmer offers kits and knowhow to streamline remediation. The use of high-grade reagents and PFAS-free laboratory accessories are also critical. By implementing steps to remove or reduce background contamination and appropriate sample preparation, PerkinElmer's highly sensitive QSight® 220 LC/MS/MS system has proven to be extremely capable of meeting the challenging demands of low-level PFAS analysis in drinking water. Validation studies demonstrated that the instrument easily meets stringent requirements of EPA 537.1 and 533 regulations for all targeted analytes.

Table 3. Instrument sensitivity (LOQ and LOD) for all target analytes in EPA Method 537.1.

Analyte	Instrument (ng L^{-1})[a]		Method (ng L^{-1})[b]	
	LOD	LOQ	LOD	LOQ
PFBS	2.00	6.68	0.008	0.027
PFHxA	2.31	7.70	0.009	0.031
HFPO-DA	6.70	22.35	0.027	0.089
PFHpA	2.10	6.99	0.008	0.028
PFHxS	0.38	1.28	0.002	0.005
ADONA	0.24	0.79	0.001	0.003
PFOA	2.57	8.56	0.010	0.034
PFOS	0.92	3.07	0.004	0.012
PFNA	2.52	8.40	0.010	0.034
9Cl-PF3ONS	0.60	2.00	0.002	0.008
PFDA	2.17	7.24	0.009	0.029
NMeFOSAA	0.29	0.96	0.001	0.004
PFUnA	3.50	11.67	0.014	0.047
NEtFOSAA	0.25	0.85	0.001	0.003
11Cl-PF3OUdS	0.44	1.48	0.002	0.006
PFDoA	2.02	6.73	0.008	0.027
PFTrDA	1.55	5.16	0.006	0.021
PFTA	4.29	14.30	0.017	0.057

[a]Instrument LOD/LOQ was determined using the signal-to-noise ratio (S/N) of the peak from the lowest detectable calibration standard (5–18 ng L^{-1}) and extrapolating to the concentration at which the S/N = 3 or 10 for LOD or LOQ, respectively. This is an estimate to demonstrate expected LOD/LOQ and can vary from lab to lab. [b]Method LOD/LOQ is calculated by multiplying the instrument LOD/LOQ by 1/250 to account for the 250 to 1 sample concentration from the SPE extraction. LOD/LOQ cannot be used as MRLs but provide an estimate of instrument sensitivity.

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Table 4. Method detection limits (DL) and lowest concentration minimum reporting limits (LCMRL) and minimum reporting levels (MRL) determined experimentally on the QSight® LC/MS/MS system and compared to reference values report in EPA Method 537.1.

Analyte	Experimental DL (ng L^{-1})[a]	EPA 537.1 DL (ng L^{-1})[b]	Experimental LCMRL (ng L^{-1})[c]	EPA 537.1 LCMRL (ng L^{-1})[d]	Experimental MRL (ng L^{-1})[e]
PFBS	1.1	6.3	0.72	1.8	1.4
PFHxA	1.5	1.7	0.93	1.0	0.30
HFPO-DA	1.5	4.3	0.57	1.9	1.6
PFHpA	1.6	0.63	0.10	0.71	1.6
PFHxS	1.2	2.4	0.60	1.4	0.29
ADONA	1.4	0.55	ND	0.88	0.28
PFOA	1.3	0.82	0.34	0.53	0.30
PFOS	1.4	2.7	1.0	1.1	0.29
PFNA	1.6	0.83	0.50	0.70	1.6
9Cl-PF3ONS	1.1	1.8	0.68	1.4	1.5
PFDA	1.1	3.3	0.40	1.6	0.30
NMeFOSAA	1.2	4.3	0.22	2.4	0.30
PFUnA	1.3	5.2	0.30	1.6	1.6
NEtFOSAA	1.2	4.8	0.73	2.8	1.6
11Cl-PF3OUdS	0.66	1.5	0.39	1.5	0.28
PFDoA	1.2	1.3	0.19	1.2	0.30
PFTrDA	1.0	0.53	0.82	0.72	4.0
PFTA	0.86	1.2	1.5	1.1	4.0

[a]Experimental DL was determined from ten LFB replicates fortified at ~4.0 ng L^{-1} measured over three days and calculated according to section 9.2.8 in EPA Method 537.1 rev 2.0.

[b]Reference DL values from EPA Method 537.1 rev 2.0 (Table 5) determined from seven LFB replicates fortified at 4.0 ng L^{-1} measured over three days and calculated according to section 9.2.8.

[c]Experimental LCMRLs were determined from ten replicates each at five fortification levels ranging from ~0.2 ng L^{-1} to 80 ng L^{-1} using the EPA LCMRL Calculator.[11]

[d]Reference LCMRL values from EPA Method 537.1 rev 2.0 (Table 5).

[e]Experimental MRLs were determined from seven LFBs fortified at concentrations ranging from ~0.2 ng L^{-1} to 4.0 ng L^{-1} according to section 9.2.6 of EPA Method 537.1 rev 2.0 using the Half Range prediction interval method with confirmed upper and lower Prediction Interval Results (PIR) ≤150 % and ≥50 %, respectively.

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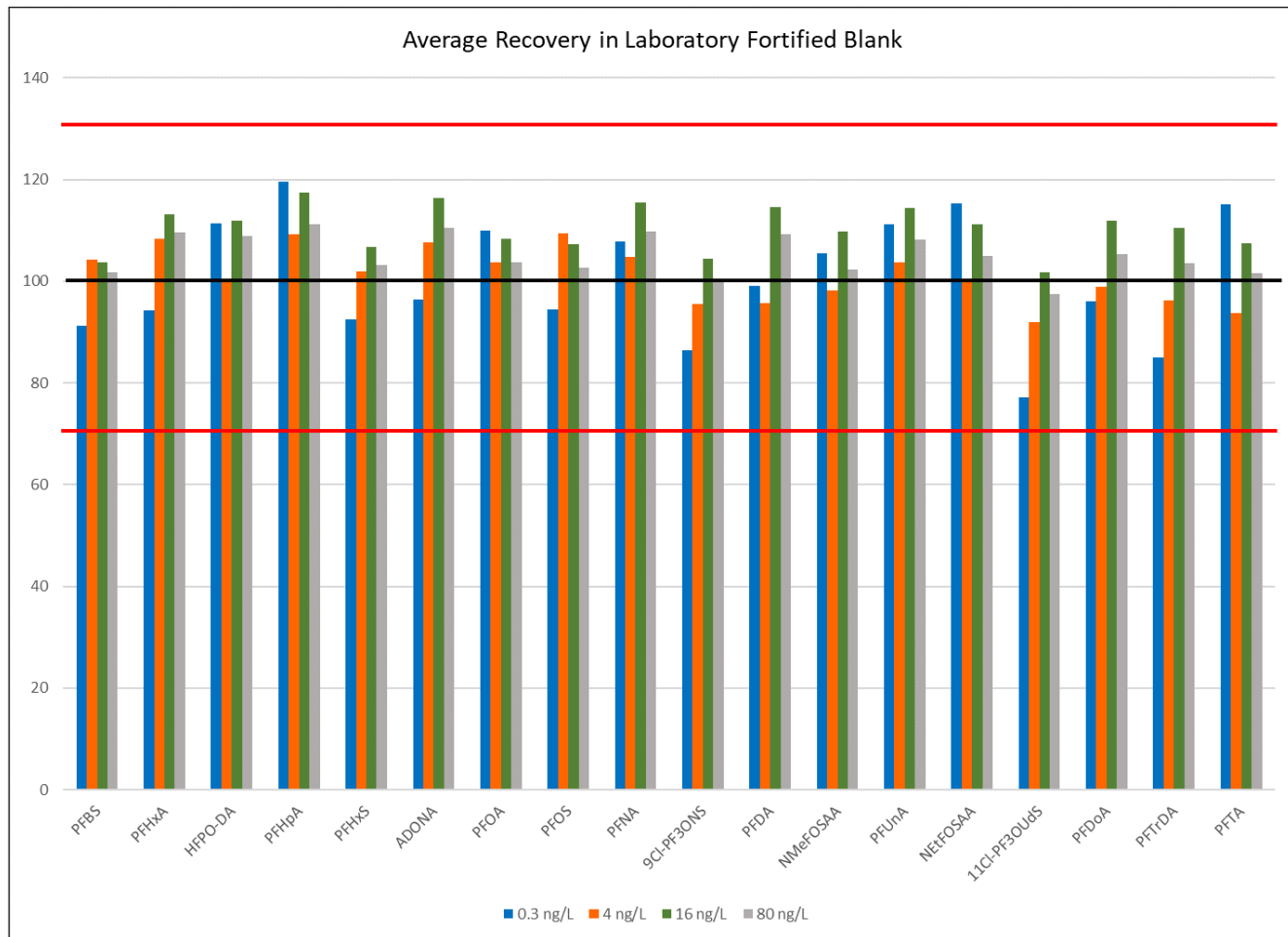
Figure 4. PFAS recovery precision and accuracy summary.
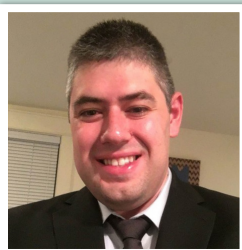
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Cole Strattman joined PerkinElmer in the role of Field Applications Scientist, supporting the Northeast US. Cole will be working with the Applied segments with a focus on the markets related to LC and LCMS instrumentation. Cole comes from a 14-year career at Rhodes Technologies where he was working as a scientist responsible for research and development. Cole has a Bachelor's in Chemistry from University of RI and is experienced in method development & validation for GC, HPLC and LCMS using different instruments along with various sample preparation techniques.

SPONSORED ARTICLE

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TONY DAVIES COLUMN

Exploiting fibre-optic spectroscopic responses

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Social Media is an interesting source of information on the whereabouts and activities of former colleagues. I was prompted about a change of role for Wieland Hill, a Raman spectroscopist and part of our Molecular Spectroscopic Methods research team back in Dortmund before we all went our separate ways. I congratulated Wieland on his new role as a systems architect and looked forward to seeing his first new building! Wieland responded that he was working on novel Raman system architecture design rather than blocks of flats or fancy museums. This work seemed to be a really interesting use of spectroscopic data processing from optical fibre cables. Looking at his papers this is certainly something we have not covered in this column in the past and, therefore, suitable for this issue!

Distributed sensing using spectroscopic signal from optical fibres

Reading through some of Wieland's papers in this area, it was amusing to realise that what Wieland and his colleagues have been exploiting in

their work would, in "conventional" spectroscopy using optical fibres, be regarded as annoying disturbing artefacts coming from the optical fibres themselves! As we find ourselves currently in an unexpectedly unstable world, exploiting these signals can, for example, give better border security. This is especially true where long borders, in often remote areas, do not lend themselves to frequent manned patrols or surveillance.[1]

So, what properties of these optical fibres can be exploited? Well let's go back to some basics of laser light scattering as observed using optical fibres. The underlying principle of a distributed fibre optic sensing system is to send laser pulses down the fibre and analyse the back-scattered radiation.

Rayleigh scattering

Rayleigh elastic scattering of light results from the polarisability of the medium the light is passing through (no change in wavelength).[2] The amount of scattering is inversely proportional to the fourth power of the wavelength (try using that explanation to your children of why the sky is blue![3]). Usually unwelcome in optical fibres as it gives rise to attenuation of the intensity of the laser light passing through the fibre.

Brillouin scattering

An inelastic scattering process resulting in quite small changes in wavelength of the scattered light. Brillouin scattering is created through the interaction of the incident photons with periodic vibrations (phonons) of, for example, the matrix the light is passing through. Can be a linear or non-linear effect induced by the incident laser beam itself or interaction with counter propagating light (spontaneous or stimulated Brillouin scattering).[4]

Raman scattering

Probably the most widely used light-scattering effect in spectroscopy.[5] Whereas Brillouin scattering is driven by the interaction of the laser photon electromagnetic field with acoustic phonons in the media it is travelling through, Raman scattering requires the interaction of the electromagnetic waves with optical phonons or vibrations of molecular structures of that medium. Raman scattering is a few orders of magnitude weaker than Rayleigh scattering.

These three effects yield scattered light at either the same frequency as the incident laser light (Rayleigh) or at shifted frequencies with Raman scattering showing the greatest change in frequency from the incident radiation. Figure 1 shows (badly not to scale) the frequencies of the different scattering phenomena relative to that of the original incident laser photon. The different colours in Figure 1 represent a change to the steady state signal observed due
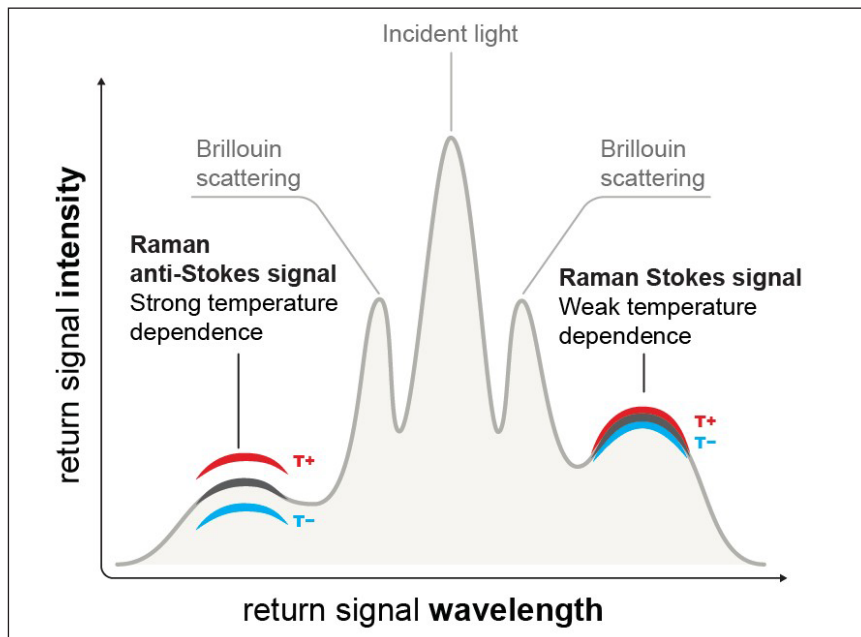
Figure 1. Relative frequencies of the inelastic scattered radiation from the Rayleigh, Raman and Brillouin scattering relative to the laser frequency. The blue and yellow lines represent the changes to the respective scattered light when used in a fibre-optic sensor. © 2012 AP Sensing

to physical effects which will be explained in the next section.

Remote monitoring of physical properties

With the correctly designed equipment, there are three different phenomena giving rise to scattered radiation that can be measured. Each phenomenon is susceptible to changes in physical parameters/environment of the optical fibre. Now, Figure 1 shows both the Stokes and Anti-Stokes scattering frequencies indicating that energy is lost to the media or gained from it during the scattering process. All three scattering processes are impacted by changes in the temperature of the system and two of them are also sensitive to strain.

Raman scattering lends itself to distributed temperature sensing as the relative intensities of the Stokes and Anti-Stokes Raman signals. They are proportional to the populations of the ground and first excited vibrational states of the molecules responsible for the scattering. As these population differences are quite large at ambient temperature, the variations in temperatures away from ambient are capable of being monitored by corresponding changes in the relative intensities of the Stokes and Anti-Stokes Raman signals.

Brillouin scattering is also susceptible to temperature, but also to strain in the system, resulting in both intensity and frequency changes for the scattered light. Having two potential sources for changes in a metrology signal is a complication which needs addressing to be certain which effect, and in what proportion, is responsible for the intensity changes observed.

Rayleigh scattering doesn't change frequency, but does exhibit changes in intensity and phase of the scattered photons which are derived from temperature again and dynamic strain. By now I am sure you are beginning to realise why a company involved in designing robust and novel systems in this area has need for a systems architect such as Wieland! Apart from

presenting some difficult challenges in the production of complex spectrometers to measure the respective signals, what use are such physical parameters in distributed sensing over long distances?

Applications of distributed fibre-optic sensing

Tiny changes in the length of the optical fibre cause changes in the interference patterns from multiple Rayleigh scattering centres. Any acoustic events occurring in the vicinity of the optical fibre cable are "heard" by the cable and can be measured by the changes in the Rayleigh interference patterns. You can imagine that being able to detect somebody walking along the outside of your security fence by the impact of their feet on the ground or attempts to tunnel under your security wall by the impact of spades in the earth.

With appropriate signal processing to filter out false alarms this operates as an excellent distributed warning system. Using communications-grade optical fibres, it is possible to monitor many kilometres distant from the location of the spectrometer. Thus, providing not only warning but also location of ongoing incidents. If we take an example that has been in the news a lot during this record-breaking year for drought and water shortage in Europe. A distributed fibre optic sensing system could easily warn of and locate a breach in a water supply pipeline. Saving not only costs in locating the breach, but also enabling a much more rapid response time before too much resource had been lost and minimising the damage to the environment around the breach.

Brillouin scattering has been adopted for distributed strain sensing for major infrastructure projects where long-term environmental changes such as ground shifting around a pipeline, for example, could pose serious problems for the deployed infrastructure

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investment.[6] Brillouin scattering has also proven to be capable of the longest optical fibre cable length deployments of hundreds of kilometres when combined with Raman amplifiers and remotely pumped optical amplifiers.[7] Reaching the theoretical limits of distance range, spatial and temperature resolution is the main challenge of designing advanced distributed sensing systems. You cannot just add another amplifier when the signal vanishes in its shot noise, so you need to think carefully about the right architecture.

Raman scattering can deliver robust temperature monitoring over tens of kilometres of infrastructure, such as power distribution cables, the inside of road tunnels or chemical plant. In power cables, the detecting optical fibre cable can actually be integrated into the cable screen itself making deployment a much simpler affair.

Conclusions

I am very pleased to see that Social Media can not only be used for organising college reunions and promoting bullying between school children, but has also enabled me to spot Wieland's new appointment. This has led to reading some interesting articles about a field of spectroscopy and spectroscopic data processing about which I knew next to nothing. For further reading and a large table detailing many more applications than we have room to discuss here, see Reference 8.

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Errors vs uncertainty vs measurement uncertainty

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Experience from the realm of scientific conferences around the world reveals that many authors and presenters are unconsciously often shifting between the term *error* and the term *uncertainty* without a clear vision of what is the subtle difference between them. The same applies to a large swath of the scientific literature, in which these two concepts are often used synonymously—a scientific flaw of the first order. There is always a difference, regardless of how small, signifying a non-zero difference between the true, unknown content a_L of a lot L and the *estimated* (analysed) content of a sample hereof, a_S. Is this difference an error or an uncertainty? Many practitioners will call it an error. This estimate of the lot concentration per force has to be made based on the minute (hopefully representative) analytical aliquot. As is well-known within the sampling community it is *all* about how to be able to secure a documentable representative analytical sample "from-lot-to-aliquot" upon which to carry out valid analysis.

Introduction and background

Tradition in many scientific fields has established the word "error" as common practice, though for some this term implies a mistake, an error that *could* or *should* have been prevented. This possible accountability does not sit well with some statisticians, which prefer the word "uncertainty" instead, because this implies no culpability. This has given rise to the need for a clear distinction between "error" and "uncertainty", which is made all the more pressing since these two terms are often used *inter alia* in less strict usages; and it certainly does not help that what is known in

certain European statistical circles as errors, are denoted uncertainty in certain American communities. Of course, everybody claims to be right ….

See Reference 1 for an almost random example of the complete confusion that is out there. So "uncertainty … is measured by the amount of error" – the confusion is complete!

Because of this state of affairs, the Council of the International Pierre Gy Sampling Association's Advisory Group has decided to clean up at least our own act—thus this "Sampling Column".

The TOS vs statistics

In the Theory of Sampling (TOS) realm it has been overwhelmingly demonstrated that there are *both* sampling errors and sampling uncertainties. The effects of some sampling errors *can* be preventatively minimised, or even eliminated (ISE), while some sampling uncertainty for a given sampling protocol is inevitable. The job is to minimise this remainder (CSE). Gy[2] stated: "With the exception of homogeneous materials, which only exist in theory, the sampling of particulate materials is always an *aleatory*[a] operation." There is always an uncertainty, regardless of how small it is, between the true, unknown content a_L of the lot L and the true, unknown content a_S of the sample S.

Thus, because the word "uncertainty" is not well suited in the real-world context of heterogeneous materials and lots, the term "error" was decided upon for the TOS,[2] making it clear that this does not necessarily imply culpability—but it *may*. The sampling errors which are subject to the possibility of elimination shall and must be so (ISE)! If not, someone or somebody is in effect

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[a]Aleatory: depending on the throw of a dice or on chance; random.

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Fact box: Too many conflicting definitions

"*Uncertainty refers to epistemic situations involving imperfect or unknown information. It applies to predictions of future events, to physical measurements that are already made, or to the unknown. Uncertainty arises in partially observable or stochastic environments, as well as due to ignorance, indolence, or both*". Wikipedia, https://en.wikipedia.org/wiki/Uncertainty

"*Uncertainty of a measured value is an interval around that value such that any repetition of the measurement will produce a new result that lies within this interval*". B. Accuracy vs. Precision, and Error vs. Uncertainty. https://www.bellevuecollege.edu/physics/resources/measure-sigfigsintro/b-acc-prec-unc/

Authors comment: a definition only involving measurement uncertainty

Defining Error and Uncertainty

"*Some of the terms in this module are used by different authors in different ways. As a result, the use of some terms here might conflict with other published uses. The definitions used in this module are intended to match the usage in documents such as the NIST Reference on Constants, Units and Uncertainty.*

For example, the term error, as used here, means the difference between a measured value and the true value for a measurement. Since the exact or 'true' measured value of quantity can often not be determined, the error in a measurement can rarely be determined. Instead, it is more consistent with the NIST methods to quantify the uncertainty of a measurement.

Uncertainty as used here means the range of possible values within which the true value of the measurement lies. This definition changes the usage of some other commonly used terms. For example, the term accuracy is often used to mean the difference between a measured result and the actual or true value. Since the true value of a measurement is usually not known, the accuracy of a measurement is usually not known either."

Peter Bohacek and Greg Schmidt, *What is Measurement and Uncertainty?*. https://serc.carleton.edu/sp/library/uncertainty/what.html [accessed 22 August 2022]

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responsible for committing an error resulting in a sampling bias (see below), which unavoidably results in unnecessary inflation of MU_{total}—thus increasing the total sampling uncertainty. This most definitely constitutes an error for which someone *is* responsible (it could be because of faulty or inferior equipment, because of an inferior standard or *ditto* procedural description—or because of an incompetent sampler or a *ditto* supervisor). The essence of the stand described here has been delineated forcefully by Pitard[3] (p. 33) who graciously informs the reader that this stand originates with Pierre Gy,[2] see also Reference 4.

Gy's choice was especially justified for an Increment Delimitation Error (IDE), Increment Extraction Error (IEE), Increment Weighting Error (IWE) and Increment Preparation Error (IPE). Because the magnitude of these errors is dictated by the ignorance, unwillingness or negligence of operators, managers and manufacturers to make these errors negligible by following the rules of *Sampling Correctness* stipulated in the TOS. For these errors, the word *uncertainty* would be totally inappropriate. Therefore, in any

project, if management is due diligent, the word error should not exist and only uncertainties remain; the problem is we are living in a world very far from perfect where the TOS is not yet mandatory knowledge for everyone in the business of creating an important analytical database. A few examples may clarify the validity of our approach in the Theory of Sampling realm.

Case #1: A necessary sample mass was poorly optimised

A sampling protocol at a mine was implemented to have a residual uncertainty no more than ±10% relative for the gold content estimate generated by industry standard 30-g fire assay. A thorough investigation of the necessary sample mass to assay to reach a 10% relative uncertainty revealed that the necessary sample mass to assay using cyanide bottle roll or gravity concentration was at least 3000 g.

Therefore, the presently used protocol was using assay samples two orders of magnitude too small. The resulting so-called huge *uncertainty* was definitely a flagrant **error**, however, due to the fact that the people in charge of the project failed to optimise their sampling protocol because of their total ignorance of the TOS.

In this case the use of the word *uncertainty* would be totally inappropriate and highly misleading; it is very clear that a huge mistake had been made.

Case #2: A non-probabilistic, therefore, incorrect sampling device was used

The content of a copper concentrate shipment was sampled at the receiving port. The copper concentrate was unloaded onto a conveyor belt. Every five minutes an operator using a scoop would collect an increment at the discharge of the belt. The composite sample was

sent to the laboratory for assaying copper.

A QA/QC programme collecting interleaved increments to assess the uncertainty affecting each copper assay was implemented. Because the scoop used by the operator was not an equi-probabilistic sampling device and consisted of an operator-dependent *grab sampling* practice, the use of the word *uncertainty* would be totally inappropriate and highly misleading. It is very clear that a huge mistake had been made using an increment sampling device transgressing the most elementary rules of sampling correctness. The word *error* would be the only appropriate word to use because of the ignorance of the management team involved.

Case #3: A well-optimised sampling protocol generated an uncertainly well within well-defined Data Quality Objectives

A manager asked the laboratory to assay the calcium content of a cement clinker within a well-defined Data Quality Objective, set at ±3 % relative. The sampling and subsampling devices were fully in compliance with the TOS to prevent the occurrence of a sampling bias due to increment delimitation errors (IDE) and increment extraction errors (IEE).

The QA/QC programme revealed that the residual uncertainty affecting the assaying of calcium was only ±2 % relative.

In this case it is correct and totally appropriate to use the word *uncertainty* because the management team was competent at performing a qualified sampling job.

A statistical aside

As an interesting aside, some (other) statisticians in fact have no problem using the term "error". The eminent statistician-educator David S. Salsburg has produced a wonderful popularising book: *Errors, Blunders and Lies – How to tell the Difference*,[5]

in which there is no problem using the term error and no hesitating in finger-pointing regarding how and how *measurement or observation uncertainties arise*. Such will always manifest themselves when observations or measurements are repeated is the message. But one is obliged to *do something* about this situation, and this is where statistics arrives as a knight on a white horse—enter classical statistics. It is worth noting that this type of error is *solely* related to measurement/observation, the only uncertainty-generating source in this realm. In the present TOS context, this type of error is identical to the analytical error, TAE. However, there is here no recognition or acknowledgement of the situation in which a much more complex process is needed before one can perform the act of "observation/measurement", i.e. analysis. This process is, of course, the complete lot-to-aliquot sampling process, which has the, for many, unknown characteristic that the process itself will influence the outcome of analysis; the sampling process itself will incur error effects if not in compliance with the TOS. Thus, another sampling procedure, another selection of equipment, another sampler (if not TOS-competent) at work, will give rise to a principally different analytical result. This is a "measurement error" of a fundamentally different nature that what is conceived of in statistics.

Thus, the TOS community is justified in establishing the error vs uncertainty context promulgated above: repeated sampling-plus-analysis is taking place within the full TSE + TAE framework and its attending consequences, and *someone* (a *legal person*) or something (standard, guide, norm-giving document etc. which are also *legal persons*) is responsible for dealing with the effects due to heterogeneity in a rational fashion. Sampling errors give rise to varying uncertainties in the analytical data base,

many of which can be dealt with very effectively, however (reduced, CSE), but some of which are fatal: professional samplers are always obliged to rid of all those pesky ISE!

The TOS vs MU

Esbensen and Wagner treated the complicated relationship between the TOS and the concept of Measurement Uncertainty (MU) in all pertinent details: see their concise summary:[6]

"A critical assessment of GUM and the EURACHEM guide shows that not all influential uncertainty sources are considered with respect to their full MU impacts. In particular, effects caused by ISEs are insufficiently defined and integrated. While GUM exclusively focuses on estimating the analytical MU, the EURACHEM guide indicates and incorporates some error sources related to sampling (mainly only the Fundamental Sampling Error), but detailed analysis reveal several deficiencies compared to TOS' full sampling-error framework. While the EURACHEM guide acknowledges the existence of the CSEs, it stays with the assumption that all other sampling error sources have been eliminated by other parties—which gives no practical help to the sampler/analyst relying on MU alone."

"By excluding both the concept of, and the risk incurred by, the inconstant sampling bias, the sampler/analyst may well not even beware of the risk that the effective MU estimate will be principally different each time it is re-estimated. The user is left without the crucial understanding that ISE effects unavoidably result in uncontrolled and unquantifiable, inflated MU_{total} estimates, i.e. the sampling variance, the sampling uncertainty, is increased because of incorrect sampling **errors**. *Only the TOS offers complete theoretical and practical understanding of all features related to heterogeneity*

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and full practical insight into the intricacies of the sampling process when confronting the gamut of heterogeneity manifestations. Closing this gap between TOS and MU necessitates a certain minimum TOS competence, and practical confidence, that all sampling processes can indeed be correct (bias-free sampling), opening up for representative, or fit-for-purpose representative sampling, which is the only way to an acceptable level of uncertainty. This minimum competency is outlined for example in the standard, DS 3077."[7]

"To derive a valid estimate of the complete uncertainty for any measurement procedure (sampling-and-analysis), all ISEs and CSEs, as well as the TAE ($MU_{analysis}$) must be considered in their proper place. This opens the way to a unified sampling-and-analysis responsibility. A detailed analysis of MU points out that TOS can simply be inducted as an essential first part in the complete measurement-process framework, taking charge and responsibility of all sampling issues at all scales along the entire lot-to-aliquot process. What is called for is a constructive integration between TOS and MU, allowing reconciliation of these two frameworks that all too long have been considered only antagonistically."

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Conclusion

The use of the term *uncertainty* should apply only in cases where the *legal person* responsible for sampling (management) has successfully trained its staff appropriately (scientists, technicians, front line samplers) and has taken a solemn commitment to apply the principles and recommendations offered by the TOS. Anything less will be an irresponsible, fragrant *error*. All sampling must be fully accountable.

The evergreen confusion arising from lack of distinction, or from synonymous usage of the terms error vs uncertainty, is unfortunately often also broadened by a prominent lack of proper understanding of the meaning of error vs uncertainty vs MU.

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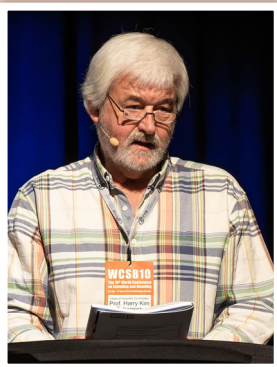
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Photo: Bo Svensmark

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QUALITY MATTERS

Four Generations of Quality: science fiction or science fact?

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Introduction

Over the last eighteen months in this series of articles[1–9] we have explored many of the Quality aspects associated with the application of spectroscopy in regulated environments, and how these have evolved in the Four Generations discussed.

Now, in this last article in the series, having brought us up to date in these areas of interest, let us "Cross the Rubicon" and hypothesise on just where events will take us into the 5th Generation.

This might be simply described as personal Crystal Ball gazing, no pun intended, however, before we discuss the 5th Generation of the Quality aspects discussed in this series, let us consider the above title.

"...science fiction or science fact?" and a few everyday examples where indeed fiction has become fact. Blue text is taken from the previous column articles.

Science fiction or science fact?

This series has been based on a chronological timeline from 1940 to the present day, and within this timeframe consider these three

examples from the *Star Trek* series, first screened in 1966, and from an iconic sci-fi film from 1968, where fiction has now become fact.

Star Trek Communicator: If memory serves, characterised by a five-note audio sequence when opened, and a flip-top cover, this design of effective communication was replicated by the modern flip-phone design. Whilst this phone design was superseded by the rise of the touchscreen App-based smartphone, as technology has moved on, it has reappeared again in the latest version of the Samsung Galaxy Z Flip4.

Star Trek Tricorder: A device that records vital signs by close proximity contact? Modern smart watches will report pulse, blood pressure etc. and even Electrocardiogram (ECG) data to an associated App. Also, invaluable in reporting blood oxygen saturation levels in the COVID-19 pandemic, the Pulse Oximeter, rather appropriately to this journal, uses visible (650 nm) and near infrared (950 nm) optical measurements to calculate this vital life sign.

Talking/Handheld Computer: Introduced in the original *Star Trek* series by the haunting phrase '...working!' when questioned, and who can forget the HAL 9000 computer in Stanley Kubrick's classic 1968 film *2001—A Space Odyssey*. Now we are all fully familiar with Android tablets, or Apple iPads and the associated Alexa/Siri voice controllers, depending on your preference.

5th Generation: where next for the concepts discussed in the series chapters

Having shown that "fiction" can indeed be turned into "fact", let us now summarily review each of the articles in the series and suggest a 5th Generation for each, with a summary conclusion at the end.

Chapter #1: definition of quality

"In business, engineering, and manufacturing, quality has a pragmatic interpretation as the non-inferiority or superiority of something; it's also defined as being suitable for its intended use while satisfying customer expectations. Quality is a perceptual, conditional, and somewhat subjective attribute and may be understood differently by different people. Consumers may focus on the specification quality of a product/service, or how it compares to competitors in the marketplace. Producers might measure the conformance quality, or degree to which the product/service was produced correctly. Support personnel may measure quality in the degree that a product is reliable, maintainable, or sustainable." Wikipedia

5th Generation

As stated in this article:

"...one of the aethereal considerations of quality, that one would hope is the outcome of commitment to a policy of 'quality first', and which will be discussed in the final Chapter in the series."

Now we are here, what of the "quality first" policy, will it survive into this generation?

We would suggest that the answer is self-evident in the fact that both this Quality Matters column exists, and this series of articles has been produced; and new areas of interest continue to boil to the surface of this cauldron of Quality Matters, on a regular basis.

Chapter #2: ISO

This article concentrates on the International Organization for Standardization (ISO) organisation, its standards and their place within the Quality environment. By definition, it discusses the role of ISO in the administration and control of these standards and their evolution and harmonisation into the standards currently in existence. This article will not discuss the specific application of ISO/IEC 17025 and ISO 17034 in association with ISO/REMCO, and accreditation authorities in the implementation of these standards; this aspect will be covered in the next article in the series.

5th Generation

ISO have their "Strategy 2030", "...Making lives easier, safer and better", which can be found on their website (https://www.iso.org/strategy2030.html), and a summary of which is available from https://www.iso.org/files/live/sites/isoorg/files/about%20ISO/strategy/download/ISO%20Strategy-Snapshot.pdf, and outlines the pivotal role of this International organisation in the future.

Chapter #3: Accreditation standards and their role in quality assurance

This article reviews the two principal ISO 17000 series accreditation standards, namely ISO/IEC 17025 and ISO 17034, their impact on the associated Quality Assurance, Testing or Calibration Laboratory

and historically how these standards have evolved and continue to be implemented. In addition, this article discusses the role(s) of the ISO 9000 series and ISO 17000 series support standards within this controlled environment. It introduces the ISO Technical Committee, ISO/REMCO, which has supported this evolution/development. This Reference Material committee, now formally reorganised by ISO as TC 334, will be reviewed and discussed in the next article in the series. As we shall see, the "Don't risk it" message is a common theme through this review and is explicitly addressed in the latest revision of ISO/IEC 17025.

5th Generation

In this generation we will undoubtedly see the production and certification of standards that address the production and use of Reference Materials designed to qualify normative properties, e.g. identification, and expand the arena of the qualitative value assignment and use into the equally important, or perhaps more so, qualitative determination of value.

Chapter #4: "Measuring Up"—new ISO committee for reference materials committed to excellence in accurate measurement results

This article, the fourth in the series, details the ISO technical committee that is responsible for Reference Materials (RMs) etc. This Reference Material committee, formally ISO/REMCO, has now been reorganised by ISO as TC 334. The history of ISO/REMCO is discussed, together with the probable changes in this technical committee brought about by its conversion to TC 334.

5th Generation

In this next generation we will see the completion of the conversion of all the existing guidance documents

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1st Generation	2nd Generation	3rd Generation	4th Generation
1940	1975	2000	2021

into formal standards with all the regulatory emphasis that this process implies and deserves. With this structure in place one of the primary tasks will be to promote this quality system to other specific regulatory environments, e.g. Pharmaceutical. Why—because from a personal perspective, all the "...do you have?" quality questions being asked on the numerous Quality Questionnaires we are being asked to complete can be answered by the same universal response:

"...yes, established under the requirements of our ISO standard(s)".

Chapter #5: GxP pharmaceutical quality assurance, an alternative track?

This article, the fifth in the series, details the history and evolution of the GxP Quality Assurance environment, used exclusively in the pharmaceutical and related support industries. In the above acronym, x = L or M, relating the Good Practice to either the "Laboratory" or "Manufacturing", amongst others.

5th Generation

In the 4th Generation, it was significant, and a major step forward in all aspects of harmonisation of international

standards, that the USA FDA chose ASTM International as the vehicle to produce a series of standards for pharmaceutical manufacturing, and committee E55—"Manufacture of Pharmaceutical and Biopharmaceutical Products" was established.

Now fully established, with a comprehensive range of associated standards, it is significant that the following statement below is rated #1, in the referenced industry trends in 2022, and undoubtedly will need E55, and the industry in general to address these issues?

1. Pharmacovigilance market will scale up

Medicines and vaccines often have undesirable and unexpected side effects. This treacherous tendency calls for more emphasis to be made on the drugs' quality and the aftermath of their usage. So, here comes pharmacovigilance from the Greek word Pharmakon meaning a medicinal substance and the Latin word Vigilia meaning to keep watch. It is a set of scientific activities for preventing adverse drug effects and other medicine-related safety problems. Its ultimate goal is to optimize the benefit-risk ratio of healthcare products usage by sharing accurate information with patients and health care professionals.[10]

Chapter #6: Key Quality Indicators and their impact on the associated environments

Having now established two of the principal tracks with respect to the quality systems in use, this article, the sixth in the series, focuses on some of the specific Quality "tools" in use in both the ISO and GxP environments; how these are defined, applied and used; and how these have evolved with time. In addition, the crossover, with respect to these specific concepts, is investigated by the use of the adoption/exchange in each environment, e.g. Qualification from GxP being implemented in ISO labs, and vice versa, the use of Expanded Uncertainty Budgets from ISO being used in updates to pharmaceutical standards etc. As we shall see, this process began in earnest at the end of the last millennium and continues, evolves and accelerates to this day.

The key quality "tools": Traceability, Validation Qualification and Calibration (VQC) and Uncertainty of Measurement.

5th Generation

As succinctly stated in this article, in this generation the use of these fundamental tools of Traceability, Validation Qualification and Calibration (VQC), and Uncertainty of Measurement will

still be essential, and continue to evolve.

Chapter #7: A new approach

As stated in the last article, this article marks a change in this series because from this point forward, we will concentrate on specific areas of interest in the Quality environment in which we are/will be working in the future.

As an unexpected consequence of the COVID-19 Pandemic, the enforced isolation of the last two years has allowed time for reflection in all aspects of life. In the scientific area, the postponement or reassignment to virtual events of major international conferences, e.g. Pittcon 2022 at the time of writing this article, has prompted an explosion of on-line webinars, presentations etc. on a whole host of topics, and the Quality environment is no exception. Available to a much wider audience than by physical attendance at a conference, this "information explosion" in these areas of interest is summarised below and reinforced by the author's direct involvement in these areas of interest.

Regular readers of this Quality Matters column will be familiar with the regulatory environments and standards often discussed by the authors; and in that respect this series of articles is no different. Discussed below are two new areas of interest, their associated terminology, standards and guidance on the topic.

The "new" quality "tools" of Analysis of Risk and Lifecycle Management

Whilst neither of these concepts can be described as new in the fundamental definition of the term, their use and application in our areas of interest certainly is.

5th Generation

This generation will undoubtedly see these key concepts, introduced in the previous generation become required constructs and maintain the high-level shift of emphasis discussed in the final overview.

Chapter #8: Software and Data Integrity: an essential partnership?

As stated in the last article, we will now consider the essential component that controls our modern instrument systems and the associated concept of data integrity that is fundamental to the quality of the data being generated.

Researching this topic, as an article within this Four Generations series, confirmed my statement in the introductory article:

"As someone who has been involved with the science of analytical measurement and/or spectroscopy for the majority of my adult life, this period of reflection centred not only on the last 12 months, but importantly on the changes since I started in a QA laboratory many years ago."

If I related this time frame specifically to the development and evolution of UV/visible instrumentation, once again the chronology of the software and data integrity can be effectively mapped to the timelines, and as in many technical areas impacting on our modern lives, there is the interesting conversion of analogue to digital electronics. For a more detailed insight into this electronic transition of our scientific instrumentation, no pun intended, see the detailed discussion below.

5th Generation

One of the hot topics in the current (4th) generation, Data Integrity will continue to be fundamentally required and challenged in the next generation, particularly with the move to "cloud"-based storage solutions, and we may see introduction of Blockchain approaches to address some of the issues raised.

Chapter #9: A spectroscopic trio

In the last article, we concentrated on how the "screwdriver in the spectroscopist toolkit" the UV/visible spectro(photo)meter, and its associated software has evolved during our Four Generations. In this article we'll now look at three other related spectroscopic techniques/tools in the box, namely, Fluorescence, near infrared (NIR) and Raman; and discuss the "what", "where" and "how" of these techniques are being used to improve the quality of the measurement processes associated with them.

This article, therefore, uses the Four Generations in the previously described time periods and does uncover some interesting points for discussion. This chronology effectively plots the evolution of these techniques from Research to Analytical Quality Assurance (QA), and the associated Quality requirements associated with them.

5th Generation

The use of these spectroscopic techniques will continue to evolve into new application areas in the clinical and biochemical fields, accelerated by the recent global pandemic and on-going related situations.

5th Generation: Overall summary

In all aspects of the evolution of any of the topics discussed in this series, the overall and structural aspect of the change can be succinctly described as a movement from a one-off or batch, to a continuous or at very least cyclic process; with the appropriate quality checks and balances clearly evident and available to prove and verify the desired control.

In many environments, the availability of appropriate integrity data should also provide sophisticated trend analysis to produce a proactive, rather than a reactive response to a perceived risk.

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No doubt, in a few years' time it will be interesting to review this article, and

"…see how the pot has boiled?"

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APPLICATIONS

## Optimising the workflow for microplastic analysis by FT-IR microscopy

Microplastics are becoming a major global environmental concern with regular major newsworthy studies revealing the presence of plastics and microplastics in remote geographic locations, or as contaminations in many different consumer products, especially food and beverages, and within the digestive systems of marine species.

Analysis of environmental samples containing microplastics is essential to determine their prevalence and their impact. A range of analytical techniques have been applied to the analysis of microplastics. Of the techniques adopted, infrared (IR) spectroscopy, and more specifically IR microscopy, is the primary analytical technique for the detection and identification of microplastics.

The different steps involved in getting from the raw sample to answers—including the initial sampling through to data analysis—are discussed in this article.
PerkinElmer

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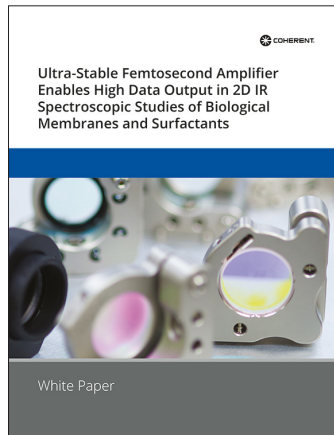
Ultra-stable femtosecond amplifier enables high data output in 2D IR spectroscopic studies

Researchers in the laboratory of Prof. Carlos Baiz (University of Texas, Austin) are investigating the dynamics of hydrophilic/hydrophobic interfaces to understand the detailed function of both biological membranes and industrial surfactants. They make extensive use of a two-dimensional infrared spectroscopy set-up based on an Astrella ultrafast laser amplifier, where the combination of output stability and operational simplicity has enabled high lab throughput as measured by publishable data.
Coherent

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Qualification of powdered gelatin agar by NIR spectroscopy

Agar is a mixture of polysaccharides widely used in food, chemical and pharmaceutical industries for its jelly-like structure. Among a variety of purposes, it can be used as a solid substrate for culture media, as a laxative, as a substituent to gelatine or as a preservative for fruits. As it is obtained from different species of red algae, it can be quality can vary from lot to lot.

This application note presents how and if NIR spectroscopy can be used to identify optimal and sub-optimal lots of powdered gelatine agar.
ABB Measurement & Analytics

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APPLICATIONS

Complete soil and sediment analysis with WDXRF

Geological samples are formed by constant deposition of inorganic and organic materials over millions of years. As the layers of deposits accumulate, the weight from the additional layers creates pressure and increased temperature creating rocks or other formation. Over time, these formations are eroded and carried away by rain, wind and snow and deposited in stream and lake beds. Analysis of these materials is of vital importance because it gives not only indications of the minerals bodies present, but also the environmental contaminates.

Analytical analysis of soil and sediment samples encompasses a vast variety of material and matrix types with many elements to be analysed having concentrations ranging from low parts per million (ppm) levels to major weight percentages of the composition. These attributes make accurate quantitative analysis ideal for wavelength dispersive X-ray fluorescence (WDXRF).

Thermo Fisher Scientific

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Spectroscopic Methods for Sunscreens Characterisation

Spectroscopic methods for sunscreens characterisation

Sunscreens protect the skin from the damaging effects of both UVA and UVB rays. It is important to characterise these products' photoactivity, composition and formulation. In this application note, fluorescence spectroscopy, Raman microscopy and particle size distribution analysis prove to be valuable techniques to assess their effectiveness, stability and skin absorption.

HORIBA Scientific

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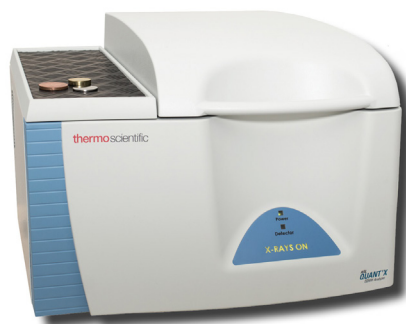
Analysis of air filters using the ARL QUANT'X EDXRF spectrometer

The United States Environmental Protection Agency's (US EPA) commitment to improve air quality across the USA has created an increasing demand for the monitoring of specific elements in ambient particulate matter collected on filters in residential, industrial and recreational areas. X-ray fluorescence is the preferred method to analyse samples related to air monitoring due to the non-destructive nature of the technique and the superior sensitivity achievable. The Thermo Scientific™ ARL™ QUANT'X Energy Dispersive X-Ray Fluorescence (EDXRF) instrumentation is ideal for determining concentrations of multiple elements on air filters. The bench top instrument can analyse the elements sodium to americium with minimal sample preparation and can accommodate filter sizes from 25 mm to 47 mm in diameter using a sample changer or up to 220 mm if loaded manually.

Thermo Fisher Scientific

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Optical Micro-spectroscopies on a Path to Identify the Source of Life

Optical micro-spectroscopies on a path to identify the source of life

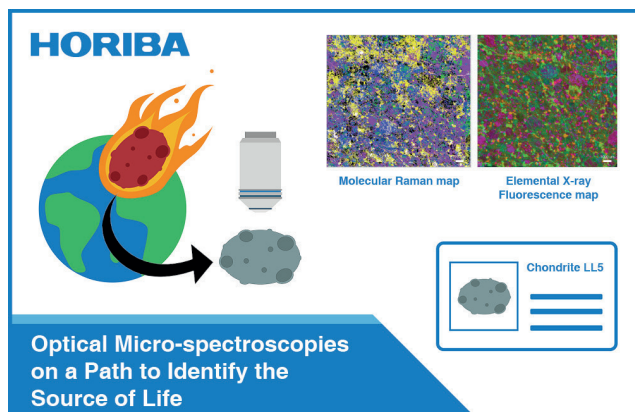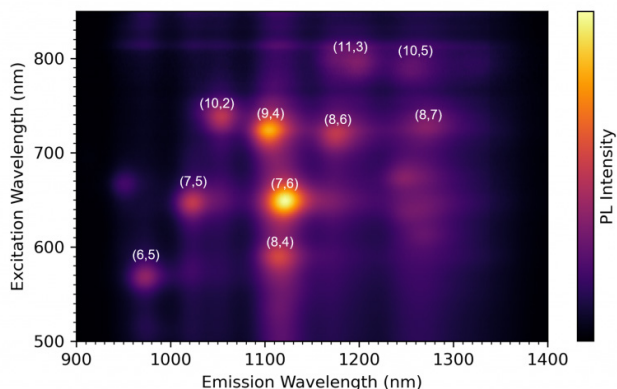Meteorites play an important role in understanding the universe we belong to. This article presents how Raman and X-Ray Fluorescence microscopies can be combined to shed some light on the universe origin, thanks to their high specificity, good spatial resolution and non-destructiveness.

*HORIBA Scientific*

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Rapid excitation emission matrix analysis of single wall carbon nanotubes

Single-wall carbon nanotubes (SWCNTs) have unique electrical, thermal, mechanical and optical properties which make them attractive for a wide variety of applications; ranging from drug delivery to battery electrodes. SWCNTs can be envisaged as a graphene layer that has been rolled up into a cylinder with different tube structures possible depending on how the graphene layer is rolled. The tube diameter and chirality (orientation of the graphene lattice with respect to the tube axis) are defined by the roll-up vector. The roll-up vector magnitude and angle are described by a pair of integers (n, m) called the chiral index.

SWCNTs are interesting because their electronic properties heavily depend on the chiral index of the tube structure. SWCNTs with different chiral indexes exhibit metallic and semiconducting properties, with the bandgap varying with chiral index. This gives SWCNTs a wealth of tunability and their properties can be tailored to specific applications. Many SWCNT synthesis methods result in a mixture of chiral structures and identifying the chiral structures present in a synthesised sample is, therefore, vital. One of the most powerful methods for SWCNT identification is photoluminescence spectroscopy. In this application note, the FLS1000 photoluminescence spectrometer equipped with an InGaAs NIR camera is used to identify the chiral indexes present in a SWCNT sample using excitation emission matrix spectroscopy.

Edinburgh Instruments

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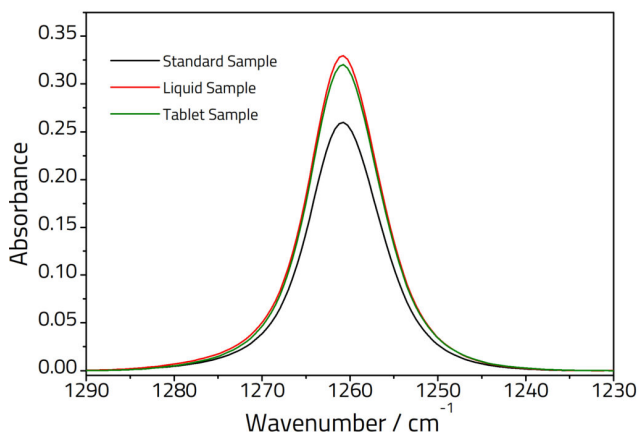
Quantifying Simeticone according to European Pharmacopoeia Standard 10.0

Learn how the Specac Pearl can be used to quantify the percentage content of Simeticone in your tablets using a toluene-acid extraction. Determination of the active pharmaceutical incipient (API) in a finished pharmaceutical product is critical to ensure the patient receives a safe dosage. FT-IR spectroscopy is a rapid, low cost technique that can give quantitative measurements in seconds to the QC analyst.

Simeticone is used for the treatment of flatulence, trapped wind and colic in babies. The European Pharmacopoeia Standard 10.0 (01/2017:1470) sets out a method for the use of an FT-IR transmission measurement to determine the % content of a sample, using a toluene extraction method. This note demonstrates that the Pearl is fully compliant with this standard and more broadly a great choice for the quantitative analysis of pharmaceutical products.

Specac

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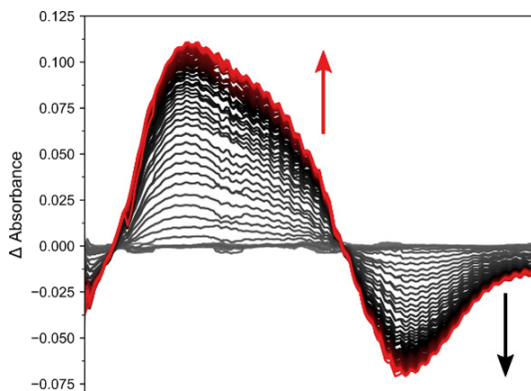
APPLICATIONS

Rapid reaction monitoring of UV cured adhesives

Fast spectroscopic monitoring is a proven tool in R&D and production environments, where it is used to gain insight into chemical reactions. This application note showcases how the Specac High Temperature Golden Gate can be combined with ultrafast spectroscopic methods to monitor curing reactions, where it can provide invaluable insight on the performance of coatings and adhesives.

Infrared spectroscopy is in many ways an ideal measurement technique for this application as it can perform non-invasive *in situ* measurements that provide direct information about the structure and bonding of the sample. However, many high-performance adhesives have fast curing rates and the speed of off-the-shelf spectroscopic techniques becomes the limiting factor in the analysis.

Specac

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Product Focus on Handheld/Mobile Instruments

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Spectrus JS
Spectrus JS lets you process and interpret your NMR data in your web browser so you can work whenever and wherever you want. Beginning fall 2022, you can also process LC/UV/MS, GC/MS data and predict complete 1D and 2D NMR spectra with industry-leading accuracy.

MORE INFORMATION »

Spectrus Processor
Process, interpret and report your NMR spectra alongside all your other analytical data. Spectrus Processor is a single vendor-neutral software application designed to handle all your spectrometric and spectroscopic data.

MORE INFORMATION »

NMR Workbook Suite
NMR Workbook Suite is the ultimate software for NMR data interpretation, structure characterisation and knowledge management. It features a full suite of advanced processing, analysis and databasing functionalities for 1D and 2D NMR data from all major vendor formats.

MORE INFORMATION »

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https://www.aptechnologies.co.uk

RedBullet 900–1700 nm Microspectrometers
RedBullet ultracompact Czerny–Turner microspectrometers replace collimating and focusing mirrors with a single concave mirror.
- $51 \times 36 \times 21\,mm^3$
- 128-pixel InGaAs sensor
- 900–1700 nm, ≤15 nm FWHM
- 8051 processor—short integration, fast readout, high accuracy trigger, low power
- USB and I/O pins
- GUI and SDK

MORE INFORMATION »

SilverBullet 180–1100 nm Microspectrometers
SilverBullet ultracompact C-T microspectrometers replace collimating and focusing mirrors with one concave mirror.
- $40 \times 36 \times 25\,mm^3$
- 1024-pixel CMOS sensor
- 180–1100 nm, 25/50/100 µm slit, 3–15 nm FWHM
- 8051 processor—short integration, fast readout, high accuracy trigger, low power
- USB, I/O pins
- GUI and SDK

MORE INFORMATION »

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Ocean ST Microspectrometer
Ocean ST is a powerful microspectrometer with excellent UV response, high-speed spectral acquisition, and impressive signal to noise ratio performance for applications from

PRODUCT FOCUS

DNA absorbance to colour measurement. Ocean ST is ideal for integration into setups where a small footprint is desired.

<p style="text-align:center">MORE INFORMATION »</p>

Ocean SR2 Spectrometer

The Ocean SR2 spectrometer has a combination of speed (integration times to 10 µs) and SNR (380 : 1) that provides application versatility without the trade-offs of comparable spectrometers. Ocean SR2 is highly configurable, has excellent thermal stability and provides low unit-to-unit variation.

<p style="text-align:center">MORE INFORMATION »</p>

Ocean HDX Spectrometer

Ocean HDX uses a novel optical bench design and high-performance components to provide high throughput, low stray light and excellent thermal stability for integrated, industrial and research applications. Ocean HDX is available in options for UV-Vis, Vis-NIR and extended wavelength range coverage.

<p style="text-align:center">MORE INFORMATION »</p>

Quantum Design UK and Ireland Ltd

Tel: 01372 378822
info@qd-uki.co.uk
https://www.qd-uki.co.uk

Specim IQ Hyperspectral Imaging Camera

Specim IQ is a portable hyperspectral camera, where data capturing, data processing and result visualisation are integrated into a one ready-to-use package. The camera is able to screen the imaging target and show the results on the camera display in just seconds. The weight of the camera, 1.3 kg containing a chargeable battery and a memory card for data storing, allows true portability for imaging in locations, where it has not been possible before.

Features

- VNIR 400–1000 nm (CMOS)
- 5 Mpx
- SD card max 32 GB
- Specim Dataset with ENVI compatible data files
- 5200 mAh Li-Ion (Type 26650)
- Approximately 100 measurements with one SD card and battery
- 4.3" touch screen + 13 physical buttons

<p style="text-align:center">MORE INFORMATION »</p>

NEW PRODUCTS

ATOMIC

New version of the SPECTROMAXx arc/spark OES analyser

SPECTRO Analytical Instruments has introduced the SPECTROMAXx LMX10 arc/spark OES analyser for elemental analysis in metal producing and fabricating plants, and iron and non-ferrous foundries. The LMX10 is the newest version of the SPECTROMAXx series. Advanced diagnostics and easy maintenance prevent downtime. In addition, the analyser features SPECTRO's proprietary iCAL 2.0 calibration logic, which requires only five minutes and a single sample per day—and automatically compensates for most changes in environmental temperature or pressure.

The LMX10 has new advancements, including completely new factory calibrations; extended calibration ranges and element selections; optimised source excitation parameters; and improved limits of detection (LODs). It also introduces a new feature that allows for a total shutdown of argon flow during standby for periods configurable by the operator. The LMX10 provides routine, trouble-free analysis of 10 matrices, 65 methods and 59 elements via convenient controls for operation; easy access for use and maintenance; and enhanced software features such as quick-check programs, virtual type standards and spectrum scans.

The new SPECTROMAXx LMX10 is available in two models. The basic SPECTROMAXx features a single air optic, with state-of-the-art CMOS sensors used in the flagship SPECTROLAB S. It extends the relevant and applicable elemental wavelength range from 233 nm to 670 nm. In addition, a new option analyses relevant wavelengths to 766 nm, covering potassium (K). The SPECTROMAXx Advanced utilises the same high-resolution CMOS technology, with the air optic covering the same wavelengths as the basic model. But an added UV optic handles a lower spectral range, from 120 nm to 235 nm. This includes a new capability to analyse oxygen (O) in copper (Cu). In addition, a new option analyses relevant wavelengths to 766 nm.

SPECTRO Analytical Instruments

▸ https://link.spectroscopyeurope.com/1762-P2-2022

IMAGING

MALDI-MS imaging system

Shimadzu has introduced the iMScope QT, a MALDI-MS imaging system with a built-in optical microscope. The mass spectrometry imaging system combines the LCMS-9030 Q-TOF mass spectrometer with a built-in optical microscope, the new iMLayer AERO for matrix coating, Imaging MS Solution software for data acquisition and

NEW PRODUCTS

IMAGEREVEAL software for data analysis. The iMScope QT is suitable for medical and pharmaceutical applications as well as for agricultural and food sciences. It visualises distributions of target substances in microscopic regions by using the optical images from the integrated microscope as well as information about the localisations of *m/z* values from mass spectrometric analysis. For example, the system can be applied in cancer treatment to verify proper drug distribution.

The iMLayer AERO is an instrument for preparing MALDI imaging analysis. It allows automated sample coating with the MALDI matrix by sublimation, which provides a very consistent coating, leading to results with high reproducibility.

The iMScope QT offers high-precision guidance of the sample target and an extremely stable UV laser combined with a quadrupole time-of-flight mass spectrometer (Q-TOF). This combination achieves a spatial resolution of 5 μm. Compared to the previous model (iMScopeTrio), the iMScope QT delivers three times the mass resolution and five times the image acquisition speed. For example, the acquisition time of a tissue section (14 mm × 7 mm, 20 μm resolution) can be reduced from 4 hours to about 50 minutes.

The MALDI imaging module with integrated optical microscope can be easily coupled to or removed from the mass spectrometer. Swapped with an ESI or APCI source, the instrument can be used as a Q-TOF LC-MS system, known as the LCMS-9030. Users who already use an LCMS-9030 system can purchase the MALDI imaging unit separately.

Shimadzu

▸ https://link.spectroscopyeurope.com/104-P2-2022

Renishaw launches the inLux SEM Raman interface

Renishaw have launched the inLux SEM Raman interface. Adding this to a SEM enables *in situ* Raman spectroscopy to complement SEM information. The inLux interface is compatible with SEMs from all major manufacturers and can be easily added to new and existing SEMs on site.

The design of the inLux interface enables Raman measurements to be performed while the sample is under the SEM beam. This allows simultaneous Raman, photoluminescence (PL) or cathodoluminescence (CL) measurements with SEM imaging, making experimental workflows easier and faster. The inLux interface is used without any need to move the sample, even when performing mapping measurements. This enables simple and accurate correlation between the Raman data and SEM images. When not in use, the inLux probe can be withdrawn from the SEM chamber enabling completely independent use of the SEM.

NEW PRODUCTS

The inLux interface offers a comprehensive range of Raman capabilities. Spectra can be collected from single points, multiple points or 2D and 3D confocal Raman images generated. The inLux interface comes fully equipped for all this work as standard, enabling analysis of areas >0.5 mm in each axis. Fully encoded position control, down to 100 nm, assures precise sample movement.

The inLux interface can be equipped with up to three fibre optic modules, two different Raman excitation wavelengths from 405 nm to 785 nm and an optional CL probe. These modules offer flexibility when dealing with fluorescent or challenging samples. The inLux interface is used in conjunction with Renishaw's research grade Raman spectrometers and software. This provides comprehensive processing and analysis capabilities whilst being intuitively simple to use.

Renishaw

▸ **https://link.spectroscopyeurope.com/655-P1-2022**
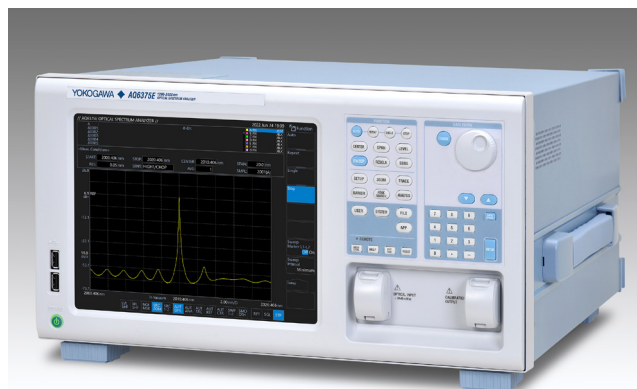
INFRARED

Optical spectrum analysers covering wide wavelength range

Yokogawa has launched two new optical spectrum analysers (OSAs). The Yokogawa AQ6375E and AQ6376E are grating-based OSAs covering SWIR (short-wavelength infrared) over 2 μm and MWIR (mid-wavelength infrared) over 3 μm. Four versions are offered covering different wavelength ranges.

Applications include environmental measurement and gas sensing, where the AQ637xE can be used in the measurement of gas absorption spectra and the characterisation of light sources used in laser absorption spectroscopy. Other major uses for the new OSAs are medical care and biotechnology and industrial laser applications. Typical applications will include the development and measurement of lasers; the characterisation of broadband light sources such as supercontinuum light sources; optical passive devices; and optical fibres.

Yokogawa Test & Measurement

▸ **https://link.spectroscopyeurope.com/7007-P1-2022**

MAGNETIC RESONANCE

[15]N TROSY cryoprobe

Bruker has announced a new 8 mm ^{15}N TROSY Cryoprobe for enabling novel NMR functional structural biology investigations into larger globular proteins and protein complexes. The new technology also facilitates cell biology research into highly dynamic intrinsically disordered proteins (IDPs) or protein regions that often

play key roles in signalling pathways via phosphorylation, e.g. in cancer. The new 8 mm ^{15}N TROSY Cryoprobe provides twice the sensitivity of previous 5 mm detection and 50 % greater ^{13}C sensitivity at 800 MHz. It allows researchers to obtain information in crowded IDP spectral regions for the unambiguous assignment of IDP backbone resonances.

Bruker BioSpin

▸ https://link.spectroscopyeurope.com/2436-P1-2022

MASS SPECTROMETRY

Multi-user MALDI-TOF mass spectrometer

Shimadzu has introduced the MALDI-8030, a new MALDI-TOF mass spectrometer with dual-polarity ion source enabling analysis in both positive- and negative-ion modes. Similar to the top-of-the-line MALDI-8020, the new MALDI-8030 utilises the identical instrument design to produce a compact footprint while still achieving similar performance specifications as the larger MALDI-TOF systems. The MALDI-8030 is based on a user-centric concept. It operates at a pleasantly quiet noise level (<55 dB) and is remarkably easy to use. The intuitive, built-in MALDI Solutions software is suitable for both advanced and novice users. The multi-LED front panel keeps users accurately informed of the instrument status.

The Fast-MS design combines a solid-state laser (200 Hz) with a fast-moving sample stage, high-speed transient recorder for rapid data transfer and a load-lock system for sample target insertion without delay. These outstanding features enable faster results and improve laboratory productivity. The ion source, with patented WideBore ion optics and Near-On Axis laser focusing, provides high sensitivity and robustness as well as mass resolution. As a result, the MALDI-8030 enables low-level detection of proteins, peptides and polymers, as well as other analytes in both positive- and negative-ion mode.

The patented TrueClean function performs automatic UV laser self-cleaning of the ion optics. Elimination of desiccants and the use of an oil-free diaphragm pump reduce maintenance costs. In addition, the MALDI laser has a lifetime of 2 billion shots.

Shimadzu

▸ https://link.spectroscopyeurope.com/104-P1-2022

NEW PRODUCTS

Single quadrupole LC/MS detector

Shimadzu has introduced the LCMS-2050, a single quadrupole LC/MS detector. It is equipped with the newly developed Heated Dual Ion Source (DUIS) as standard. This hybrid source is a combination of electrospray ionisation (ESI) and chemical ionisation at atmospheric pressure (APCI). In this way, ions that ionise well either by APCI or ESI can be detected simultaneously. The result is high sensitivity to compounds in a wide range of chemical properties, including low-polar compounds. By quickly switching the polarity, it is also possible to determine both positive and negative ions during the measurement with high sensitivity. With the help of Shimadzu's MASS-IT function for displaying mass information, complex data can be collected at a glance. The function detects and superimposes the signature mass information on the LC-UV or LC-PDA chromatogram. With just one click, several components in a single UV peak become visible as well as "hidden" components without UV absorption.
Shimadzu

▶ https://link.spectroscopyeurope.com/104-P3-2022

DIARY

Conferences
2022

21–23 September 2022, Málaga, Spain. **IBER2022-XVI Iberian Joint Meeting on Atomic and Molecular Physics.** contact@iber2022.com, https://iber2022.com/

25–30 September 2022, Erice, Italy. **International School on Mass Spectrometry (IntSMS).** http://www.spettrometriadimassa.it/intsms2022

26–28 September 2022, Ulm, Germany. **18th Confocal Raman Imaging Symposium.** events@witec.de, https://www.raman-symposium.com/

2–7 October, Cincinnati, United States. **Annual Conference of Federation of Analytical Chemistry and Spectroscopy Societies, SciX 2022.** facss@facss.org, http://www.scixconference.org

3–5 October 2022, Amsterdam, Netherlands. **Rapid Methods Europe–RME 2022.** RME@bastiaanse-communication.com, https://www.rapidmethods.eu/index.php

9–12 October, Denver, United States. **2022 Geological Society of America (GSA) Meeting.** meetings@geosociety.org, http://www.geosociety.org

16–19 October, San Diego, United States. **PANIC 2022.** https://panicnmr.com/conference-schedule-sandiego-2022/

28–30 October 2022, Seoul, South Korea. **8th Asian NIR Symposium (ANS 2022).** ans2022seoul@gmail.com, https://www.ans2022.org

14–15 November 2022, Leeds, United Kingdom. **BMSS BioMacromolecular Structure Special Interest Group Meeting.** mark_mcdowall@icloud.com, https://www.bmss.org.uk/bmss-biomacromolecular-structure-sig-meeting-2022/

5–9 December, Kyoto, Japan. **XX CIGR World Congress 2022: Sustainable Agricultural Production - Water, Land, Energy and Food.** cigr2022@convention.co.jp, http://www.cigr2022.org

12–16 December, Chicago. **2022 AGU—Advancing Earth and Space Science Fall Meeting.** meetinginfo@agu.org, https://www.agu.org/Events/Meetings/Fall-Meeting-2022

2023

29 January–3 February, Ljubljana, Slovenia. **2023 European Winter Conference on Plasma Spectrochemistry.** http://www.ewcps2021.ki.si

19–22 March, Philadelphia, PA, USA. **Pittcon 2023.** https://www.pittcon.org

11–14 April 2023, Vienna, Austria. **ANAKON 2023.** office@anakon2023.at, https://www.anakon2023.at

21–27 August, Innsbruck, Austria. **NIR-2023.** https://www.spectroscopyeurope.com/events/nir-2023

17–20 September, Baverno, Italy. **SMASH 2023 - Small Molecule NMR Conference.** https://www.smashnmr.org

Courses
2022

15 October, Obergurgl, Austria. **Advanced Study Course on Optical Chemical Sensors (ASCOS).** Christian.W.Huck@uibk.ac.at, http://ascos.org/

17–21 October 2022, Gembloux, Belgium. CRA-W **Vibrational Spectroscopy and Chemometric Course.** j.fernandez@cra.wallonie.be, https://www.cra.wallonie.be/en/annual-spectroscopy-and-chemometrics-training-2

18–19 October 2022, Turin, Italy. **CHARISMA Raman School 2022** . j.friesl@yordasgroup.com, https://www.h2020charisma.eu/ramanschool2022

Exhibitions
2022

2–3 November 2022, Birmingham, United Kingdom. **Lab Innovations 2022.** Alison.Willis@easyfairs.com, https://www.lab-innovations.com/

24–26 November, Istanbul, Turkey. **Turkchem.** http://www.chemshoweurasia.com

2023

19–21 April 2023, Ho Chi Minh City, Vietnam. **Analytica Vietnam.** https://www.analyticavietnam.com/

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