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SPECTROSCOPY asia

The essential magazine for spectroscopists in the Asia/Pacific region

MS pregnancy test for whales

Hyperspectral mid-infrared ellipsometry

Identification of dental materials in food

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The simultaneous measurement of 11 steroid hormones in whale blubber using LC-MS/MS has enabled a more accurate pregnancy test. See the News article on page 6.

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CONTENTS

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Ian Michael

Pittcon 2020 Awards

A number of awards are presented at each annual Pittsburgh Conference honouring scientists who have made outstanding contributions to analytical chemistry and applied spectroscopy. We report here those involved in spectroscopy.

Pittsburgh Spectroscopy Award

This award is given annually to a researcher who demonstrates outstanding achievements in spectroscopy. Dr Ji-Xin Cheng of Boston University was chosen for his outstanding contribution in the advanced understanding and applications in the field of bioanalytical spectroscopy, as well as his leadership in the scientific community and mentorship of young scientists. Dr Cheng is a Professor at Weldon School of Biomedical Engineering and Department of Chemistry.
www.bu.edu/eng/profile/ji-xin-cheng/

Pittsburgh Analytical Chemistry Award

Milan Mrksich is the Henry Wade Rogers Professor at Northwestern University, with appointments in Chemistry, Biomedical Engineering and Cell & Developmental Biology. His research programme develops and applies surface chemistries in analytical chemistry and chemical biology, and has invented the SAMDI mass spectrometry label-free technology for performing high throughput experiments.
www.mccormick.northwestern.edu/research-faculty/directory/profiles/mrksich-milan.html

Pittsburgh Conference Achievement Award

This award is presented annually to a researcher that made a significant and independent impact in an area of analytical chemistry, within the first ten years of their academic career. Livia Schiavinato Eberlin was chosen based on her inventive research, strong collaboration and development of diagnostic tools for cancer detection. Professor Eberlin is an Assistant Professor in the Department of Chemistry at The University of Texas at Austin.
eberlin.cm.utexas.edu/livia/

SEAC—Charles N. Reilley Award

Shelley Minteer is a Professor of Chemistry at the University of Utah. She received her PhD at the University of Iowa working with Professor Johna Leddy. She began her career at Saint Louis University before moving to the University of Utah. Her interests are focused on bioelectrochemistry and electroanalytical chemistry.
chem.utah.edu/directory/minteer/

Advances in Measurement Science Lectureship Award

Jennifer Brodbelt is the Rowland Pettit Centennial Professor of Chemistry and Chairperson at the University of Texas at Austin. Her research focuses on the development of photodissociation mass spectrometry for characterisation of biological molecules. She serves as an Associate Editor for the *Journal of the American Society for Mass Spectrometry*.
cns.utexas.edu/directory/item/12-chemistry/81-brodbelt-jennifer-s?Itemid=349

Mass spectrometry provides a new way to probe single molecules

The new approach, called individual ion mass spectrometry (I2MS) can determine the exact mass of a huge range of intact proteins. It analyses the mass of each and every molecule on an individual basis. Details were published in *Nature Methods* (http://doi.org/dq4x). The researchers show that their method, which uses the commercially available Orbitrap mass spectrometer system, can be used on super complex mixtures of intact proteins, and even whole virus particles carrying diverse cargo within them. This power and versatility will enable a new wave of molecular precision to be brought to diverse problems in vaccinology, virology, neurodegenerative plaques and disease biology in general.

"Quickly characterising the masses of viruses and their infectious cargo over time may help scientists understand mutations that are occurring", said Neil L. Kelleher, who led the research. "Whether directly characterising different strains of viruses or profiling different vaccine formulations, our new technology now can be deployed directly on these protein-containing samples to pursue the most urgent challenges of the day", he said.

The technology will help scientists further understand the composition of the exterior of a virus (called the capsid) and the infectious cargo held within the capsid, Kelleher said. Because the researchers can analyse a handful of single virus particles at a time, they can extract information about precise variations in each particle.

"Many research groups are studying the use of viral capsids filled with cargo as a means to deliver life-saving drugs to patients", said Jared O. Kafader, the study's first author. "Our technology provides a practical way to determine if the cargo contains the correct drug or to find out what is actually within each virus particle."

A related study by the Kelleher group, published recently in the *Journal of Proteome Research* (http://doi.org/dq4z), extends the use of I2MS to the fragmentation of intact species. By fragmenting intact proteins, important information can be obtained about where modifications or mutations on the protein can be identified, the researchers said. These modifications have implications for understanding how proteins change or mutate in cancer patients.

Taking hyphenated techniques to the next level

Analytical chemists from the University of Amsterdam and Vrije Universiteit Amsterdam have, together with the Netherlands Forensic Institute and the companies Genentech, Shell and DSM, started the PARADISE project to develop robust, automated methodologies that allow multidimensional characterisation of the most challenging man-made and natural products. The project has been awarded funding of almost a million euros from the Science PPP Fund of the Netherlands Organisation for Scientific Research NWO.

A multitude of analytical techniques is often required to assess the composition and to characterise the various properties of the components of the analysed materials. A major problem is that in many cases those techniques have to be applied separately, as a result, often the most-relevant properties, and in particular the relations between these, cannot be assessed. As an example, many drug-delivery systems used in medical applications consist of biopolymeric particles that release an active pharmaceutical ingredient. Analytical methods are applied to study, for instance, the composition of these particles, their stability, their drug content and the release characteristics of these drugs. However, most of the time it is impossible to study several of these properties in conjunction. The methods allow for instance analysis of particle content or particle size, but not both since the former analysis requires the destruction of the particles.

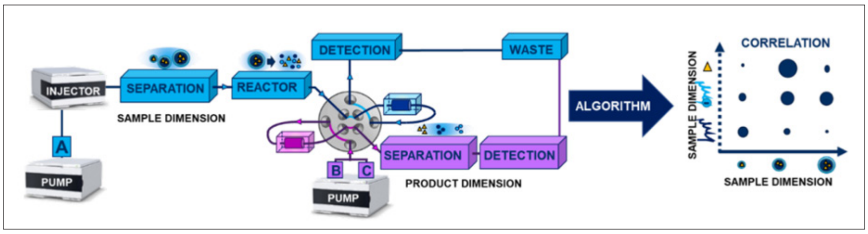
The PARADISE project provides a novel approach for the analysis of multiple properties of complex samples through the sequential application of complementing analytical techniques. The challenge here is to retain property relations at the molecular scale through intelligent combination (or modulation) of the subsequent analysis steps.

Successful modulation allows the coupling of two orthogonal methods, yielding information on two dimensions of the sample and the correlation between these. In the PARADISE project, new modulation tools will be developed for the coupling of previously incompatible techniques. Furthermore, methods will be developed to characterise sample properties that cannot currently be measured as well as methods to obtain correlations between different sample dimensions from single experiments.

In addition to developing new technologies to enable meaningful comprehensive orthogonal analysis, PARADISE will also develop tools for (largely) automated method data analysis and optimisation also by using Artificial Intelligence tools.

NIR spectroscopy can detect acrylamide

Acrylamide is a natural neurotoxin often found in starchy foods. It is formed as a chemical reaction when the food is cooked at high temperatures. Examples of foods with higher levels of acrylamide include fries/chips, potato chips/crisps, breads, cereals and coffee. According to the National Cancer Institute, studies in rodent models have shown that exposure to acrylamide can increase the risk



Example of a PARADISE setup featuring an innovative reaction-modulation interface to combine two separation techniques, which characterise two orthogonal sample dimensions. Algorithms will be developed to translate data obtained from the fragment analysis into information on the original sample dimension of interest. A feasibility study showed that reactions can be made sufficiently fast to meet the demands of other components of the system, such as the first separation step. Image: HIMS.

of several types of cancer. Furthermore, because the body converts acrylamide into glycidamide, there is also an association between acrylamide intake and mutations in and damage to DNA.

In light of these risk factors, in 2018, the European Union implemented a directive that urges food operators to closely monitor the presence of acrylamide in foodstuffs. Unfortunately, doing so is easier said than done. That's because existing techniques for detecting acrylamide are time-consuming, expensive and overly complex. To help food operators comply with the EU directive, the EU-funded AFREELAMIDE project developed a detection device that uses visible/near infrared (vis/NIR) spectroscopy. The new device allows users to conduct real-time monitoring of individual food pieces on site and at a cost that is five times cheaper than traditional inspection means.

"This is the first time that spectroscopy systems have been used to detect acrylamide levels in regular, everyday foods", explains Efrén García, Founder and CEO of Centaurea, a Spanish tech company and lead partner in the AFREELAMIDE project. "With spectroscopy, we can monitor 100 % of a producer's food products for the presence of acrylamide, saving the company money and ensuring the health and well-being of consumers."

During the course of the project, the AFREELAMIDE system was tested in several real industrial settings, including global agri-food company Siro Group. "These tests demonstrated that by using a near infrared system to measure acrylamide, companies can ensure that their entire output is completely free of this potentially dangerous neurotoxin", explains García.

Based on a market study conducted by project researchers, AFREELAMIDE has the potential to be both scalable and profitable. Sold at a unit price of EUR 20,000, García notes that his company anticipates sales of more than EUR 8 million by 2025. In preparation for its marketisation, Centaurea is currently working to obtain financing through the EIC Accelerator. This funding will be used to develop a large database of all the foods affected by acrylamide.

Mass spectrometry: a better pregnancy test for whales


A humpback whale. Credit: Sally Mizroch/NOAA

You may be surprised to learn that it is not easy to do pregnancy tests on whales. For decades, the only way scientists could count pregnant females was by sight and best guesses based on visual characteristics. For the last several years, researchers have relied on hormone tests of blubber collected via darts, but the results were often inconclusive, and researchers couldn't confidently say if the animal was pregnant or just ovulating. Research from the National Institute of Standards and Technology (NIST) and Australia's Griffith University points to a weakness of that testing and provides a new method for hormone testing that offers better results.

"Previous tests only looked at progesterone in blubber samples", said Ashley Boggs, a research biologist at NIST who helped to develop the new tests. "We found that androgens, and especially androstenedione in combination with progesterone, are much more likely to be a reliable marker of pregnancy."

"Up to this point, most measurements had been conducted using immunoassays, which require a 'one at a time' approach to hormone measurements", Boggs said. "We know that hormones act together in suites to cause large physiological changes. If you focus on only one hormone at a time, you might miss the major hormone of interest or simply run out of money or samples."

The research team studied female humpbacks along the east coast of Australia during two stages of migration. Fifty-two individuals were randomly sampled before reaching the calving grounds in June/July (austral winter) and again after departing the calving grounds in September/October (austral spring). Before reaching the calving grounds, only one had a high concentration of progesterone, the hormone that has previously been used as an indicator of pregnancy in this species. This number seemed too low to be reliable. The evidence indicated that the animals' hormone profiles change in late pregnancy, since multiple calves were seen later.

Finding chemical indicators for pregnancy in marine mammals is very different from finding them in humans, where a protein hormone, human chorionic gonadotropin (hCG), is detected right after the implantation of the embryo. Other protein hormones are difficult to use as a standard indicator for pregnancy because their levels vary from one species to the other. Previous whale reproductive research had relied on the measurement of the hormone progesterone using an ELISA test. ELISA has been the "go to" assay for years, but NIST scientists have determined that not all species rely on the same hormones during pregnancy, and in addition, some species' hormone levels exhibit significant shifts during different phases of the gestation process.

NIST researchers realised that mass spectrometry would potentially allow the measurement of many hormones simultaneously and could help biologists to understand which hormones can be found at different stages of pregnancy. The new analysis from NIST and Griffith University scientists allowed the gathering of a simultaneous measurement of 11 steroid hormones in each sample of whale blubber using LC-MS/MS.

"By casting a wider net and looking for more hormones, we were able to get a better set of biomarkers," said Boggs. The same technique will also likely be applicable to other species including North Atlantic right whales, which are dying at alarming rates and considered to be a species in peril. This work was reported in *Scientific Reports* (http://doi.org/dq42).

Perturbation-free studies of single molecules

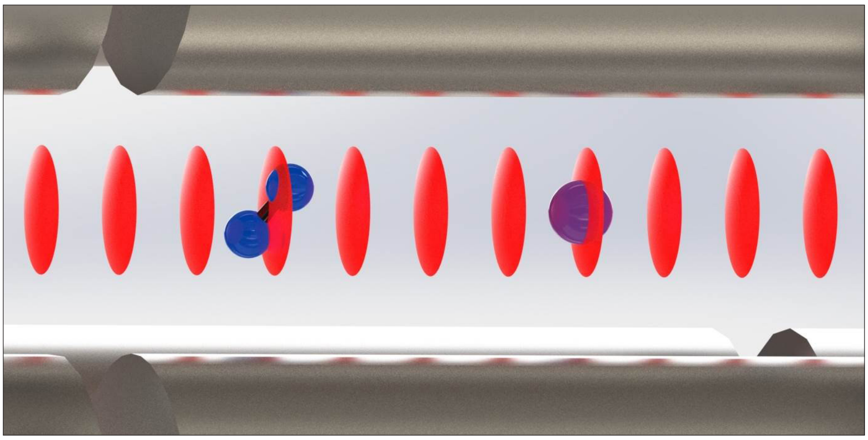
Researchers of the University of Basel have developed a new method with which individual isolated molecules can be studied precisely—without destroying the molecule or even influencing its quantum state. This highly sensitive technique for probing molecules is widely applicable and paves the way for a range of new applications in the fields of quantum science, spectroscopy and chemistry.

Inspired by quantum methods developed for the manipulation of atoms, the research group of Professor Stefan Willitsch at the Department of Chemistry of the University of Basel has developed a new technique which enables spectroscopic measurements on the level of a single molecule, here as an example a single, charged nitrogen molecule. The new technique does not disturb the molecule or even perturb its quantum state. In their experiments, the molecule is trapped in a radiofrequency trap and cooled down to near absolute zero. To enable cooling, an auxiliary atom (here a single, charged calcium atom) is simultaneously trapped and localised next to the molecule. This spatial proximity is also essential for the subsequent spectroscopic study of the molecule.

Subsequently, a force is generated on the molecule by focusing two laser beams on the particles to form a so-called optical lattice. The strength of this optical force increases with the proximity of the irradiated wavelength to a spectroscopic excitation in the molecule resulting in a vibration of the molecule within the trap instead of its excitation. The strength of the vibration is thus related to the proximity to a spectroscopic transition and is transmitted to the neighbouring calcium atom from which it is detected with high sensitivity. In this way, the same information on the molecule can be retrieved as in a conventional spectroscopic experiment.

This method, which is a new type of force spectroscopy, introduces several new concepts. First, it relies on single molecules instead of large ensembles. Second, it represents a completely non-invasive technique as detection is accomplished indirectly (via a neighbouring atom) and without a direct excitation of spectroscopic transitions. Therefore, the quantum state of the molecule is left intact, so that the measurement can be repeated continuously. As a result, the method is much more sensitive than established spectroscopic methods that rely on the direct excitation and destruction of a large number of molecules.

There is a range of prospective applications of the new method, Professor Willitsch explains: "Our type of force spectroscopy allows extremely precise measurements on molecules which are not possible with conventional spectroscopic techniques. With the new method, one can study molecular properties and chemical reactions in very sensitively and under precisely defined conditions on the single-molecule level. It also paves the way for investigations of very fundamental questions such as 'Are physical constants really constant or do they vary with time?' A more practical application could be the development of an ultraprecise clock based on a single molecule—or the application of molecules as building blocks for quantum computers." The work was reported in *Science* (http://doi.org/dq43).



A charged nitrogen molecule is interrogated by a charged calcium atom in an optical lattice. (Image: University of Basel, Department of Chemistry)

Bruker partners with ANPC to combat the COVID-19 threat

Bruker is partnering with the Australian National Phenome Centre (ANPC) at Murdoch University to support the work of their researchers into the COVID-19 pandemic threat. The ANPC team, led by Professor Jeremy Nicholson, and working with the South Metropolitan Health Service COVID-19 Response Team and the broader Western Australian (WA) healthcare community, has launched a

continued on page 12

Hyperspectral mid-infrared ellipsometric measurements in the twinkling of an eye

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Introduction

Fast, contact-less and destruction-free hyperspectral infrared (IR) techniques that enable large-area mapping within short measurement times are highly relevant for research and industry in environmental, biomedical, material and space applications. Laser-based methods provide high optical throughput as well as high spectral, spatial and temporal resolution, and are thus of particular interest for analytical, process, laboratory and field applications.

In this article, we focus on a recently introduced, rapid, laser-based hyperspectral method for thin-film analysis in the mid-IR fingerprint range. Featuring a polarimetric single-shot design, both phase and amplitude information related to refractive and absorption indices are simultaneously recorded. A tuneable, pulsed quantum cascade laser (QCL) enables spectrally highly resolved ($<0.5\,\text{cm}^{-1}$) ellipsometric mapping at lateral resolutions of $\leq 120\,\mu\text{m}$. High QCL tuning speeds provide access to time-dependent measurements of single spots in the µs/ms range, and fast hyperspectral mapping of large sample areas ($50 \times 50\,\text{mm}^2$). The single-shot approach ensures robustness with respect to pulse-to-pulse intensity variations and changes in environmental conditions like humidity. Hyperspectral and time-dependent phase and amplitude images facilitate the chemical identification of a specific material via its vibrational fingerprint, but also interpretation with respect to film thickness, molecular structure, composition and homogeneity of these parameters.

Laser-based IR imaging

Mid-infrared (MIR) imaging beyond classical Fourier-transform infrared (FT-IR) techniques recently showed promising developments for material science,[1–8] medical diagnostics and the study of biological samples.[9–11] IR spectroscopic applications are significantly broadened and strengthened by modern technical advances such as upconversion-based hyperspectral imaging,[4,9] hyperspectral nanospectroscopy,[1–3,5–7,10] frequency-comb techniques,[12] visible-diffraction limited optical photothermal IR multispectral imaging,[11] and QCL-based single-shot polarisation dependent hyperspectral concepts.[7,8,13]

IR imaging as a linear optical technique can reveal vibrational and structural sample information. Being contact-less, label-free and non-destructive, IR imaging can also be performed under varying environmental conditions. The latest advances in detection systems, optical set-ups and the use of brilliant MIR light sources open the door for new IR spectroscopic possibilities with respect to lateral and time resolution, structure analyses, as well as imaging capabilities in general. Particularly the involvement of QCL sources allows one to overcome the limits of classical FT-IR spectroscopy both in far-field and near-field IR-related techniques. Typical applications are time-resolved[13] and hyperspectral IR ellipsometry[8] (Sentech Instruments), photothermal AFM-IR[2] (e.g., from Bruker/Anasys), visible-diffraction limited spectroscopy[11] [e.g., optical photothermal infrared (O-PTIR) spectroscopy from Photothermal Spectroscopy Corp.], and scattering near-field IR spectroscopy[1,5] (e.g., from Neaspec and Bruker).

In this article, we focus on MIR laser-based polarimetric imaging (i.e., hyperspectral ellipsometry) as a new method to simultaneously reveal vibrational, structural and thickness information.[8,13]

Method of hyperspectral single-shot ellipsometry

The developed hyperspectral IR laser polarimeter incorporates a four-channel division-of-amplitude concept[14] that enables simultaneous single-shot amplitude and phase measurements at the laser repetition rate. Time-dependent measurements of individual spots (pixels) are possible with sub-decisecond resolution spectrally and sub-millisecond resolution down to $10\,\mu\text{s}$ at a single wavelength. Fast hyperspectral amplitude and phase imaging is achieved by lateral sample mapping. Compared to classical IR ellipsometric imaging using FT-IR, measurement times are considerably reduced with this new laser-based ellipsometric technique.

The polarimeter (Sentech Instruments) consists of a tilt- and height-adjustable sample mapping stage (50 mm × 50 mm) and an autocollimation unit for defined sample alignment. A pulsed external-cavity QCL (MIRcat 2100, Daylight Solutions) is employed as a brilliant, broadband-tuneable radiation source. A custom-built beamsplitter optics divides and directs the laser beam into four parallel detection channels, each equipped with a polarising unit (KRS-5 wire-grid polariser, Specac) and a photovoltaic InAsSb detector (P13894-211, Hamamatsu). A custom-built gated-integrator electronics synchronises the polarisation-state analysers of the four channels, thus enabling single-shot ellipsometric measurements.

By selecting specific polariser settings and additional optical elements like retarders, it is possible to tailor the four detection channels. This flexible approach allows one to measure various polarimetric parameters, for example, ellipsometric amplitudes ($\tan\Psi$), phases (Δ) or specific Mueller-Matrix elements related to the sample's polarimetric properties (e.g., circular/linear dichroism and birefringence).[15,7]

In this contribution, we focus on single-shot measurements of the ellipsometric angles Ψ and Δ. Using a different polariser setting α in each of the four detection channels, the single-shot ellipsometer measures Ψ and Δ from the intensities I_α according to:

$$\cos 2\Psi = \frac{I_{90°} - I_{0°}}{I_{90°} + I_{0°}},$$

$$\sin 2\Psi \cos\Delta = \frac{I_{45°} - I_{135°}}{I_{45°} + I_{135°}} \qquad (1)$$

Further technical details regarding the device and measurement protocol can be found in References 8 and 13.

The ellipsometric angles Ψ and Δ are functions of the incidence angle φ_0, the wavelength λ, the optical constants of substrate (N_S), ambient medium (N_0) and layers (N_j), as well as of the individual layer thicknesses d_j,

$$\tan\Psi \cdot e^{i\Delta} = \frac{r_p}{r_s} = F\left(\varphi_0, \lambda, N_S, N_0, N_j, d_j\right) \qquad (2)$$

$j=0, 1, 2,\ldots$ number of layers, with r_p and r_s being the p- and s-polarised complex reflection coefficients. The optical constants $N=n+ik$ (N: complex refractive index; n: refractive index; k: absorption index) are related to the complex dielectric function $\varepsilon=\varepsilon_1+i\varepsilon_2$ via $N=\sqrt{\varepsilon}$.

Measuring both Ψ and Δ yields complementary information regarding the sample's optical and structural properties, thus providing a means to in-depth sample analysis. Various routes are possible for evaluating the structure-related baselines and material-specific vibrational bands of hyperspectral IR ellipsometric data, including direct spectral interpretation, optical modelling and multivariate analysis.

Hyperspectral IR ellipsometry

Single-shot hyperspectral IR polarimetric imaging of a sample provides ellipsometric parameters dependent on wavelength and up to three spacetime coordinates. Regarding data interpretation, characteristic vibrational bands such as carboxyl, CH_x and amide bands[16] of lipids, proteins and polymers yield detailed insights into the film's properties.[13] Such data, therefore, enable unprecedentedly comprehensive, laterally resolved analysis with respect to molecular structure, chemistry, interactions, optical anisotropy, composition, morphology, film thickness and variations thereof.

In the following, we present two examples: one on the simultaneous phase and amplitude mapping of a heterogeneous surface and one on a time-dependent investigation of individual sample spots under external temperature stimuli.

Hyperspectral imaging of heterogeneous thin films

The first sample is a drop-cast thin film of myristic acid (MyA) $[CH_3(CH_2)_{12}COOH]$ on a gold substrate.[13] The homogeneous, 150 nm thick film was partially chemically modified using droplets of NaOH dissolved in ethanol, resulting in areas of pure MyA, intermediate regions and areas of complexed-like MyA. Hyperspectral measurements of the film are presented in Figure 1, showing exemplary images and spectra obtained from the multidimensional data cube.

Figure 1a shows complementary phase and amplitude contrast images at different IR wavelengths. These images readily identify areas with varying thicknesses (mainly in $\cos\Delta$) and/or a chemical structure (mainly in $\tan\Psi$). The chemical contrast image in Figure 1b shows the difference in band amplitudes of two vibrational markers at 1706 cm^{-1} (associated with C=O carboxyl vibrations of pure MyA) and at 1379 cm^{-1} (associated with complexed MyA), thus enabling the direct visual domain separation of pure and complexed MyA.

A spectral $\tan\Psi$ linescan along the red line in Figure 1b is presented in Figure 1c, highlighting the distinct vibrational characteristics of the different sample areas. Comparing the variations in spectral intensities reveals that regions of pure MyA are rather homogeneous, whereas those with complexed MyA exhibit pronounced heterogeneity in surface coverage. Pure and complexed domains are separated by an area with low coverage.

A detailed vibrational analysis of, for example, the ν(C=O) band can elucidate molecular interactions and orientations in such complex heterogeneous samples. Beyond such a spectral interpretation, a polarimetric approach can (as stated in Reference 15) "provide a number of contrast mechanisms besides traditional unpolarized radiation intensity, including linear depolarization, circular depolarization, cross-polarization, directional birefringence and dichroism." The high contrast resulting from the ellipsometric measurement could have tremendous application potential for the imaging and characterisation of complex surfaces (e.g., tissue classification for cancer identification, quality and morphology control of materials). In this regard, hyperspectral data analysis based upon neural networks, multivariate analysis and/or optical modelling could be a promising tool for gaining a detailed understanding of the structural and chemical specificities of the sample.

Together with the high stability concerning disturbing environmental

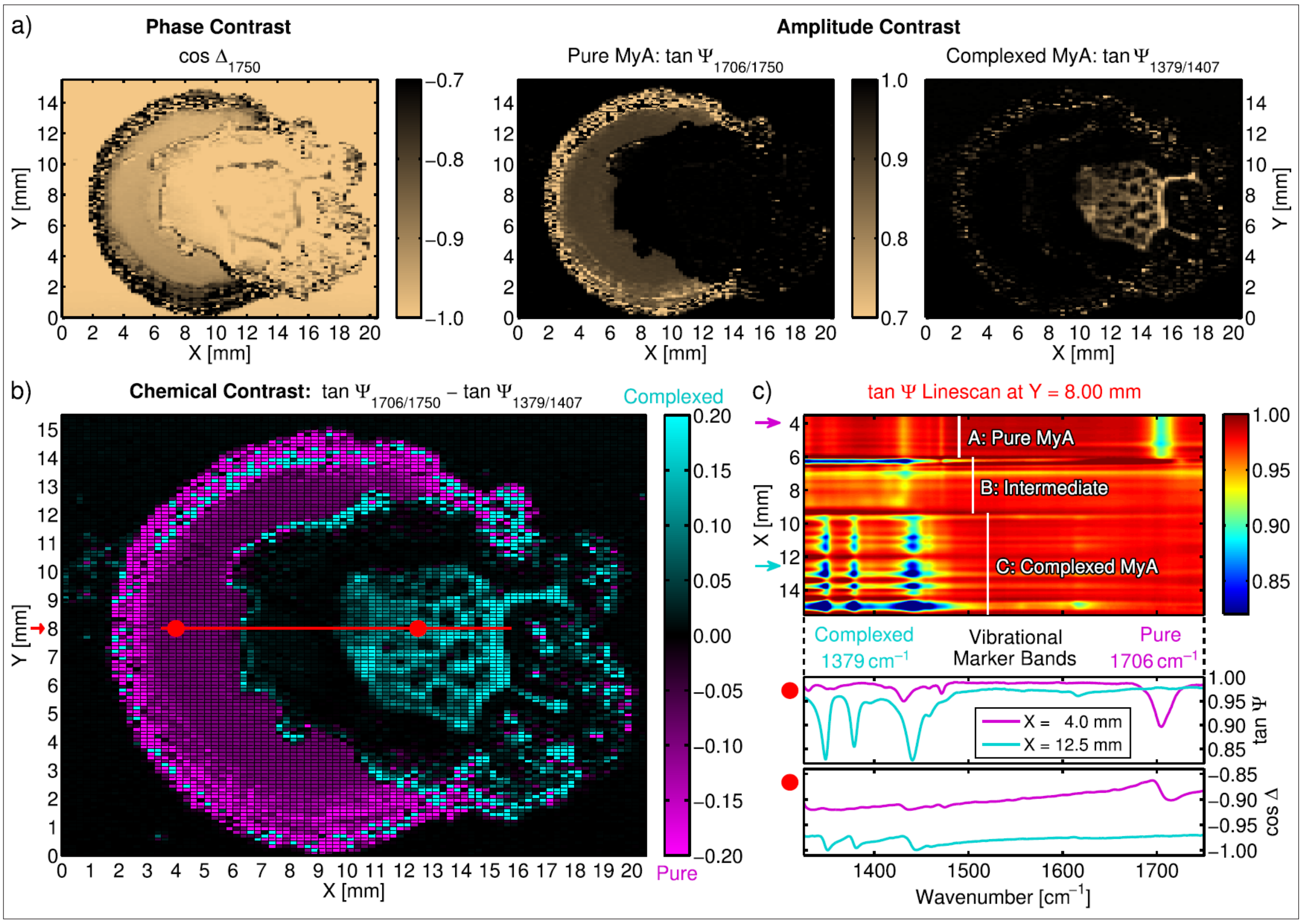

Figure 1. Hyperspectral IR ellipsometry images of a partially chemically modified, drop-cast MyA film. a) Phase and amplitude contrast at different wavelengths. b) Chemical contrast between pure and complexed MyA. c) tan Ψ linescan across the heterogeneous sample surface and exemplary ellipsometric spectra. Reprinted with permission from Reference 8 (©2019, Optical Society of America).

absorptions,[7] the capabilities of the IR laser ellipsometer make accessible multiple new IR spectroscopic imaging applications regarding the analysis of structured, anisotropic films and biological samples. Interesting systems to be investigated with the device range from functionalised surfaces and coatings, to polymer and protein materials, minerals, as well as solar cells, OLEDs and other optoelectronic devices and sensors.

Time-resolved studies of thin-film phase transitions

Spectra of single spots can be monitored within about 100 ms, and single wavelengths even as rapidly as 10 µs. The IR laser polarimeter, therefore, pushes new possibilities for time-resolved measurements of non-cyclic processes via flexible measurement durations from the µs to hour range. This point is illustrated in Figure 2, which displays the temperature-resolved investigation of the thermal phase transition of a 150 nm thick MyA film around 55 °C.[13] The amplitude and phase images in Figure 2b show the ν(C=O) band progression (cf. spectra in Figure 1c). Characteristic band components due to differently interacting C=O groups can be identified. A strongly interacting component (mode 1) is found in the solid phase at 50 °C, and a weakly interacting one (mode 2) occurs at higher wavenumbers mainly in the liquid phase at 60 °C.

Figure 2c shows the time-dependent development of the two ν(C=O) band components obtained from Figure 2b during slow heating. Figure 2d displays the corresponding single-wavelength monitoring of mode 1 during rapid cooling with a time resolution of 200 µs. While both mode 1 and 2 are present below the phase transition, only mode 2 is found above it. These observed changes reveal a tight interrelationship between molecular interactions and thermo-induced phase transition.

As these presented measurements demonstrate, the new IR polarimeter covers multiple time scales ranging from µs to minutes. The results highlight the applicability and sensitivity of the technique for time-dependent analyses of non-cyclic, irreversible processes and reactions. We see powerful applications in process and quality control, but also in rheology, relaxation and related studies. Because of the ellipsometer's small spot size (0.03 mm^2), low sample volumes and amounts can also be studied.

Currently, we are working on coupling microfluidic flow cells[17] to the instrument in order to image and analyse processes

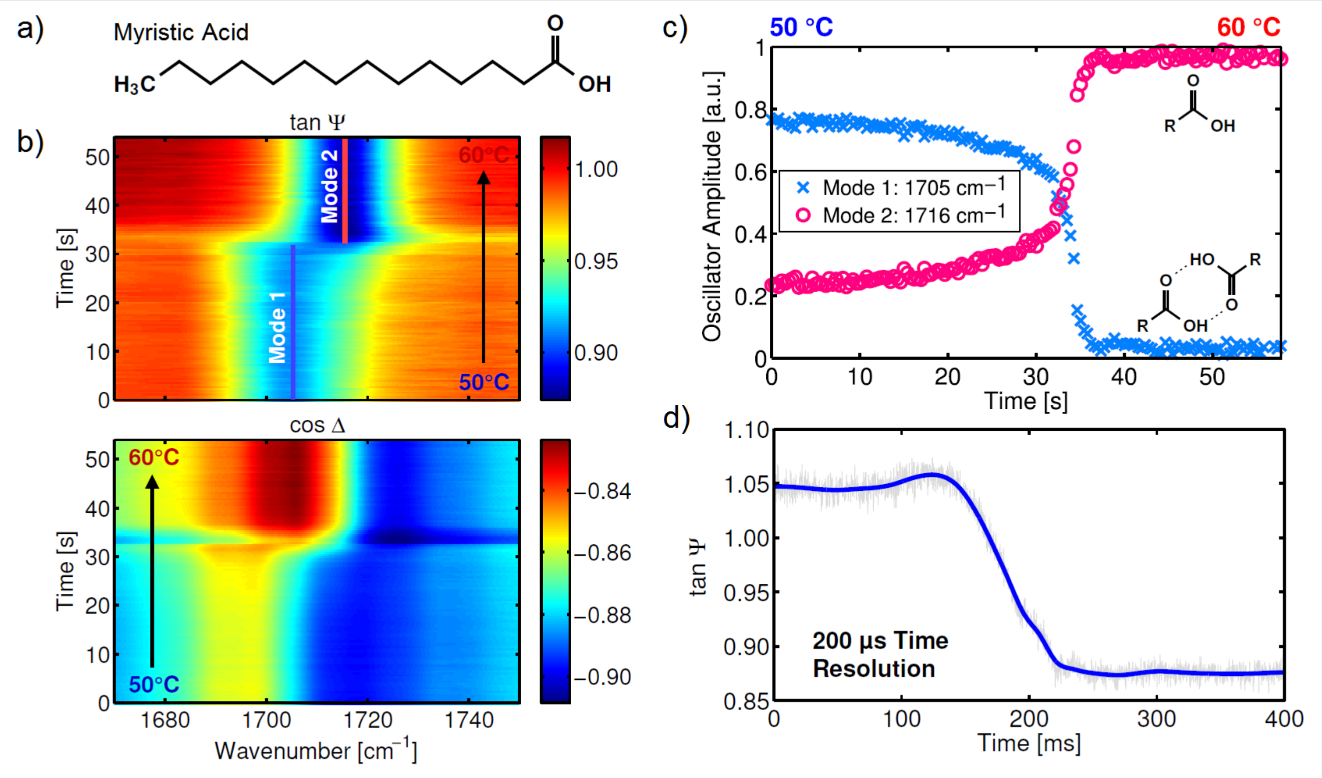

Figure 2. Time-resolved IR laser ellipsometry of the solid-to-liquid phase transition of a fatty-acid thin film upon heating/cooling. a) Chemical structure of MyA. b) ν(C=O) band amplitude and phase during slow heating. c) Oscillator amplitudes of mode 1 and 2 fitted from the spectral images. Reprinted with permission from Reference 13 (©2019, Optical Society of America). d) Sub-ms single-wavelength monitoring of mode 1 during rapid cooling.

of functional, sensor and biocompatible surfaces at solid–liquid interfaces. The set-up will then allow for sensing of molecular adsorptions, structural transitions and the study of intra- and intermolecular interactions of nL to mL samples.

A laser application laboratory will be opened at ISAS Berlin (anticipated for the end of 2020) operating and making available a hyperspectral IR laser ellipsometer.

Funding

Europäischer Fonds für regionale Entwicklung (EFRE) (1.8/13); Ministerium für Innovation, Wissenschaft und Forschung des Landes Nordrhein-Westfalen; Regierender Bürgermeister von Berlin – Senatskanzlei Wissenschaft und Forschung; Bundesministerium für Bildung und Forschung.

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continued from page 7

major research and diagnostics project to better understand and predict variation in COVID-19 severity and determine the complex genetic, environmental and lifestyle interactions that influence its pathogenicity in individuals. Later they will engage with clinical trials of novel antiviral agents and when available vaccines in order to predict responder/non-responder outcomes.

The goal is to deliver diagnostic and prognostic solutions in an accelerated time-frame. Most importantly, the risk of severity of infected patients needs to be assessed rapidly to help guide and optimise the clinical patient pathway. Researchers at the ANPC will use a range of NMR and MS instrumentation, as well as data modelling approaches, to perform broad and deep metabolic analysis of the molecular, physical and biochemical characteristics of blood plasma and urine samples to create informative translational models. These models will predict variation in the severity of the disease and help understand differential responses to therapeutic interventions.

Professor Nicholson said: "At the ANPC, we are dedicating 100 % of our resources to the COVID-19 fight for at least a year. This is the greatest emergent healthcare challenge on the planet and there is no better equipped metabolic lab in Australia, or possibly anywhere in the world, to undertake this type of investigative work in an excellent clinical and hospital framework. Linked to our genomics team, led by Professor Simon Mallal and Associate Professor Mark Watson, we're setting out to identify specific biomarkers of the disease to figure out who has it, how we can detect it and stratify patients by severity risk, and assess the real time patient responses to treatments."

Wiley acquires Bio-Rad's Informatics

John Wiley and Sons Inc. has acquired Bio-Rad Laboratories, Inc.'s Informatics products including the company's spectroscopy software and spectral databases. As one of the largest providers of spectral libraries and a long-standing partner of Wiley, Bio-Rad's Sadtler spectra databases in IR, Raman, UV-Vis and NMR allow Wiley to expand its offering in the spectroscopy market. The acquisition includes Bio-Rad's KnowItAll desktop spectroscopy data system (SDS), server SDS, web-server SDS and ChemWindow chemical structure drawing software.

Thermal analysis GC/MS agreements

NETZSCH and JEOL have entered into agreements in Europe, the USA and Japan to sell the JEOL Q1500 single quadrupole mass spectrometer as part of the NETZSCH integrated thermal analysis GC/MS system. A quasi-direct coupling solution enables software-controlled gas sampling and gas injection at short intervals.

Advion's parent to acquire Interchim group

Advion's parent company Beijing Bohui Innovation Biotechnology Co., Ltd, has filed its intention to acquire Adchim SAS, the parent company of the Interchim group companies. This transaction will result in the combination of Advion Inc. and Interchim SA, a provider of chromatography and purification instrumentation and consumables. The companies have had an OEM partnership for seven years and intend to combine for sales of instruments, consumables and customer service.

Identification of dental materials in food

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Nuts, bread crusts, toffee, apples—a hearty bite into food can be the trigger that breaks a tooth. However, the real reason may not be the food or contaminants or particles in it, but the tooth's natural age, structure and chemical make-up, or even caries. In case of complaints, how can food producers trace what has happened and begin an analysis of the cause? It starts with the materials…

There are many different types of dental products and their characteristics vary according to their intended purpose. Examples include temporary dressings, dental restorations (fillings, crowns, bridges), endodontic products (used in root canal therapy), impression materials, prosthetic materials (dentures) or dental implants. They all differ from the natural substances of a tooth.

In order to analyse the substance of materials, Fourier transform infrared (FT-IR) spectroscopy and energy-dispersive x-ray fluorescence (ED-XRF) are the methods of choice. Here, we report the analysis of a human tooth and various dental restorative materials by an FT-IR spectroscopy instrument coupled to an FT-IR microscope for molecular analysis and an ED-XRF spectrometer for elemental analysis. Due to the diversity of dental materials, which include hybrid materials containing organic and inorganic components, metal materials and others, a combined analysis approach was chosen.

Dental materials

Dental materials, such as metals, porcelain, ceramics and composite resins, are used in restorations, for example to repair carious lesions or other defects on single teeth. Fillings may be necessary in both the crown part and the root part of a tooth. A classic example is the amalgam filling, which has been used for centuries.[1]

Fillings can break off the tooth completely or only in pieces, and they may then be found as supposedly unknown foreign bodies in food. In some cases, this contamination is wrongly perceived as food contamination and consumers send it to the food manufacturer, complaining that the contamination originates from the production process. This can be clarified quickly by a combined analysis with FT-IR and ED-XRF; the complementary nature of these techniques can identify the source of contamination unambiguously.

Measurement methods and instruments

ED-XRF is a method used in material analysis and is one of the most frequently used methods for the qualitative and quantitative determination of the elemental composition of a sample, since the samples are not destroyed by the measurement and no digestions are required. It is widely used in the metal-working industry, in the analysis of glass, ceramics and building materials, as well as in the analysis of lubricants and mineral oil products.

The detection limit of ED-XRF depends on the sample matrix and the element itself. It can reach ppm levels for most elements. Here, an ED-XRF system with a Rhodium tube was used, leading to a visible Rh signal in the ED-XRF energy spectrum. These x-ray target signals are subtracted automatically from the result table, since they are not part of the sample composition. Nevertheless, Rh can be analysed by ED-XRF via the use of primary filters.

FT-IR spectroscopy can be used for the analysis of a solid, liquid or gas, simultaneously collecting data with high spectral resolution over a wide spectral range. With appropriate accessories, such as attenuated total reflection (ATR) or an

FT-IR microscope, even small samples (down to few µm) can be characterised quickly.

The analyses were conducted using an ED-XRF spectrometer (Shimadzu EDX-8000) and an AIM-9000 infrared microscope attached to an IRAffinity-1S FT-IR spectrophotometer (Shimadzu). Compared to other methods such as atomic absorption spectroscopy (AAS), inductively coupled plasma optical emission spectroscopy (ICP-OES) or inductively coupled plasma mass spectrometry (ICP-MS), both measurement techniques examine the sample destruction-free in its original state. For larger samples, it may be necessary to use a smaller piece to make it fit into the measuring instrument or accessory. If required, further tests can be performed with other analytical instruments to determine additional details of the sample.

Table 1 shows the analysis conditions of the ED-XRF and FT-IR instruments.

Human tooth—strontium and calcium phosphate make the difference

In a first step, a human tooth was analysed. Figure 1 shows a photograph of the contaminant, and Figure 2 shows a qualitative and quantitative analysis by ED-XRF. Calcium (Ca) and phosphorus (P), which are the main components of teeth, could be confirmed. Although the elemental composition is similar to that of bone, the lower content of strontium (Sr) is a characteristic feature of teeth. Like calcium, strontium is an alkaline earth metal and, therefore, can also be used to build teeth.

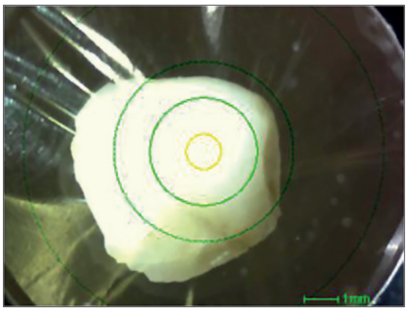
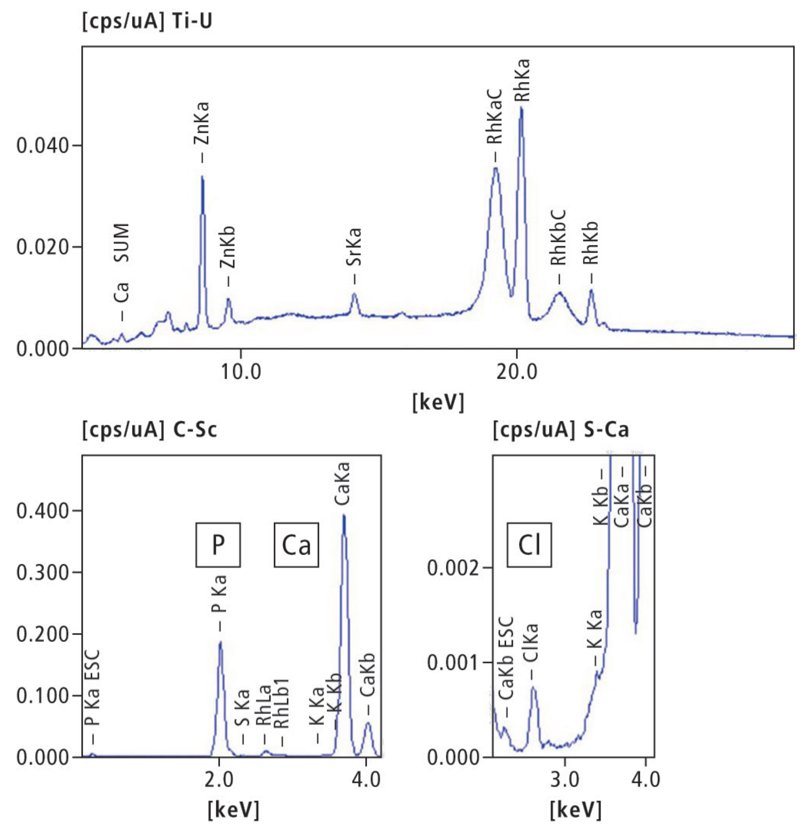


Figure 1. Photograph of contaminant (the yellow circle is the x-ray irradiation range: 1 mm ∅).

Table 1. ED-XRF and FT-IR analysis conditions.

Instrument: EDX-8000/EDX-8000P	
X-ray tube target	Rh target
Tube voltage/Tube current	15 kV (C-Sc, S-Ca), 50 kV (Ti-U)/Auto
Atmosphere	Vacuum
Collimator	1 mm ∅
Integration time	90 s
Sample container (film used)	PP film, 5 µm
Instrument: IRAffinity-1S + AIM-9000	
Resolution	8 cm^{-1}
Accumulation	40 times
Apodisation function	Sqr-triangle
Detector	MCT (mercury cadmium telluride)



Element	Ca	P	Cl	Zn
Quantitative value (%)	70.99	27.23	1.21	0.21
Element	**K**	**S**	**Sr**	
Quantitative value (%)	0.18	0.16	0.019	

Figure 2. ED-XRF qualitative and quantitative analysis results of human tooth.

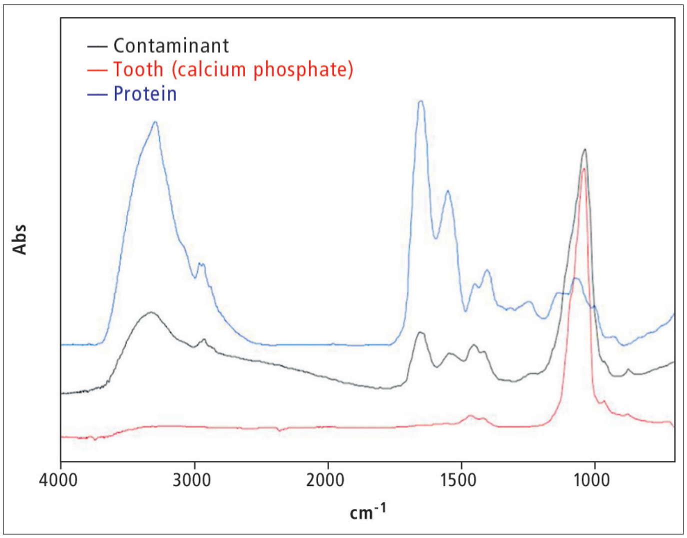
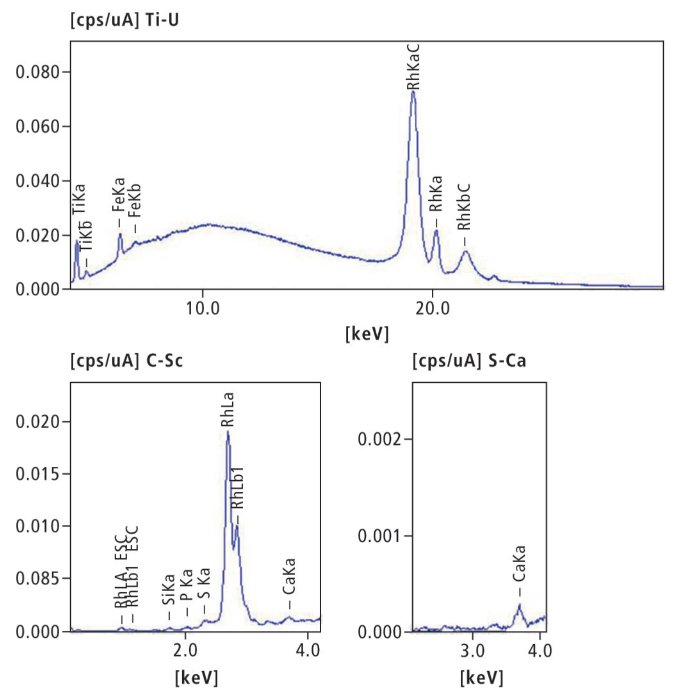


Figure 3. FT-IR absorbance spectrum of human tooth, contaminant and protein.



Element	**Ti**	**Si**	**S**	**Ca**
Quantitative value (%)	0.032	0.023	0.017	0.013
Element	**P**	**Fe**	**CH_2O**	
Quantitative value (%)	0.011	0.006	99.90	

Figure 5. EDX qualitative and quantitative analysis results.

Figure 3 shows the results of a qualitative analysis by FT-IR spectroscopy. Because the contaminant had a distorted shape and was extremely hard, a small amount of the sample was scraped off and a microscopic transmission measurement was conducted while holding the sample in a diamond cell.

The main component is calcium phosphate, and peaks originating from proteins can also be confirmed. The proteins are considered to be deposits on the tooth surface. When an adhering deposit exists, the sample is cleaned with water or ethanol, dried and then measured. However, care is required so that the contaminant is not lost before it can be analysed.

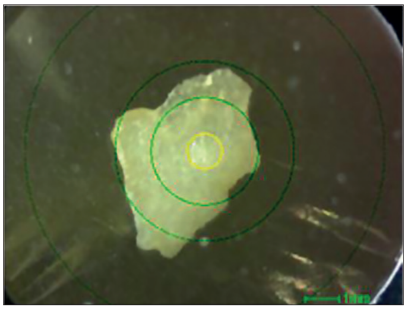


Figure 4. Photograph of contaminant (the yellow circle is the x-ray irradiation range: 1 mm ∅).

Artificial tooth (composite resin): PMMA and PMEA

In the second part of the experiment, a piece of an artificial tooth was analysed. A photograph (Figure 4) shows the contaminant, and Figure 5 shows the results of a qualitative and quantitative analysis by ED-XRF. Because the main component is organic material, a quantitative calculation was carried out assuming the balance is CH_2O. This is necessary since ED-XRF systems cannot analyse organic material, but can take the matrix effect of the organic composition into account for the elemental concentration calculation. A simplified description of the organic matrix, here CH_2O, leads to good quantitative results.

Figure 6 shows the results of a qualitative analysis by FT-IR spectroscopy. A small amount of the sample was scraped off, and a microscopic transmission measurement was conducted while holding the sample in a diamond cell. An FT-IR library search of the spectra was performed, and hits were obtained for polymethyl methacrylate (PMMA) and polyethyl methacrylate (PEMA). Based on these results, the contaminant is considered to be a piece of artificial tooth (composite resin).

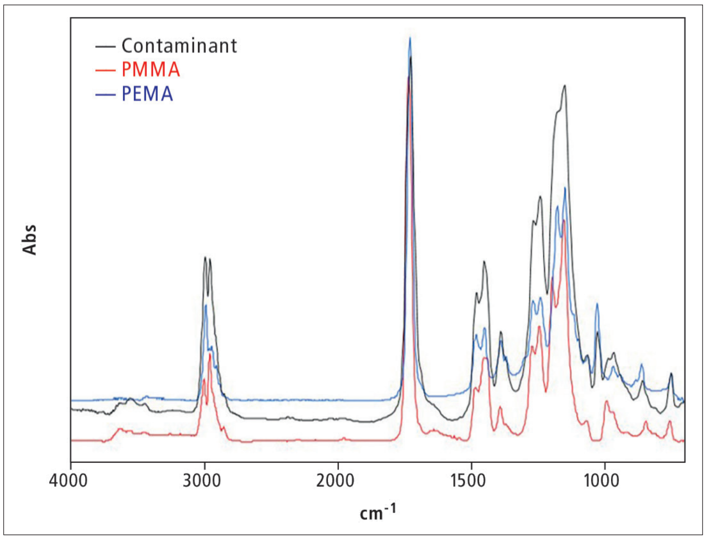
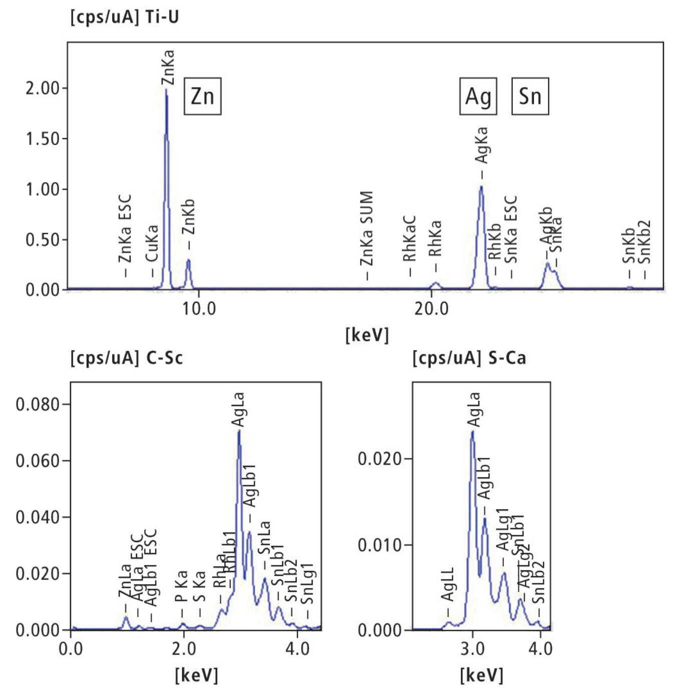


Figure 6. FT-IR absorbance spectrum of contaminant, PMMA and PEMA.



Element	Ag	Sn	Zn	P
Quantitative value (%)	61.81	22.08	15.59	0.38
Element	**S**	**Cu**		
Quantitative value (%)	0.088	0.054		

Figure 8. EDX qualitative and quantitative analysis results.

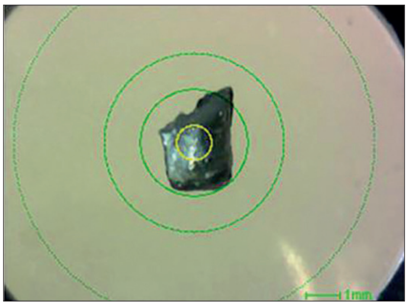


Figure 7. Photograph of contaminant (the yellow circle is the x-ray irradiation range: 1 mm ∅).

Artificial tooth (metal alloy) made of Ag, Sn and Zn

An artificial tooth piece was analysed in the last part of the study. Figure 7 shows a photograph of the contaminant, and Figure 8 shows the results of the qualitative and quantitative analysis by ED-XRF. The main components are silver, tin and zinc. No significant peaks were detected in the FT-IR analysis. Based on these results, the contaminant is an artificial metal tooth filling alloy. Figure 7 shows a piece of a silver crown.

Conclusion

Contaminants in the form of a human tooth and various dental restorative materials were analysed by ED-XRF and FT-IR microspectroscopy. Due to the diversity of dental materials, which include hybrid materials containing organic and inorganic components, metal materials and others, a combined analysis technique using ED-XRF and FT-IR spectroscopy is very effective. Together, both techniques clarify the molecular and elemental composition of the food containment. Stainless steel, small stones, glass or rubber pieces can be analysed and differentiated quickly.

These techniques support food producers in detecting the cause of food contamination when they receive pieces found in food which are wrongly supposed to originate from the production process, enabling possible customer complaints to be clarified quickly. Since the methods are non-destructive, further analytical steps can be taken afterwards such as AAS, ICP-OES, ICP-MS and others. They provide more in-depth results, if required even down to concentrations in sub-ppm levels.

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Robust single method determination of major and trace elements in foodstuffs using the Thermo Scientific iCAP PRO X Duo inductively coupled plasma optical emission spectrometer

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Goal

This application demonstrates the ability of the Thermo Scientific™ iCAP™ PRO Series inductively coupled plasma optical emission spectroscopy (ICP-OES) instrument to determine trace elements and major components in foodstuffs to comply with regulations.

Introduction

Food protection from potentially hazardous contaminants has become a major topic of public interest. As well as the standard regulatory testing, it is necessary to account for contaminants, which may enter the food chain via many means, such as industrial pollution or environmental contamination, i.e., polluted rainfall on crops. Once toxic elements are in the food chain, they can pose significant health risks. With this in mind and the increasing number of micronutrients requiring determination, it is critical that the method of testing is a rigorous and reliable one. The organisation charged with the development of the Codex standards (worldwide food and consumer regulations) is the Codex Alimentarius Commission, which is an intergovernmental body jointly sponsored by the Food and Agriculture Organization and the World Health Organization.

Instrumentation

The Thermo Scientific iCAP PRO Series ICP-OES instrument employs a high-resolution echelle spectrometer with a charge injection device (CID) detector. Advancements in CID technology allow this detector to feature lower noise and better separation of spectral orders than any of its predecessors. With the new optical design, the full spectrum from 167 nm to 852 nm can be measured with a single exposure. A Thermo Scientific™ iCAP™ PRO X Duo ICP-OES instrument was chosen for this analysis as this enables maximum sensitivity using axial view whilst maintaining excellent matrix tolerance in radial viewing mode. The instrument parameters used are listed in Table 1.

Table 1. Instrument parameters.

Parameters	Setting
Pump tubing	Sample: Tygon® orange/white Drain: Tygon® white/white
Spray chamber	Glass cyclonic
Nebuliser	Glass concentric
Centre tube	2.0 mm
Pump speed	45 rpm
Nebuliser gas flow	0.6 L min^{-1}
Auxiliary gas flow	0.5 L min^{-1}
Coolant gas flow	12.5 L min^{-1}
RF power	1150 W
Exposure time	Axial: 15 s
	Radial: 15 s

Table 2. Element, wavelength and plasma view used.

Element	Wavelength (nm)	Plasma view
Ca	317.933	Radial
Cu	324.754	Axial
Fe	259.940	Axial
Mg	285.213	Radial
Mn	257.610	Axial
Ni	231.604	Axial
P	178.284	Radial
Pb	220.353	Axial
Zn	206.200	Axial

Method development and analysis

Initially, more than one wavelength was selected for each element (using both axial and radial view). The sub-arrays for each wavelength were then examined and the most appropriate wavelength for the application was chosen (Table 2) based on the presence of interferences, calibration curve quality, QCs and CRMs and the required linearity for the element. The sub-array plots for each element can be easily manipulated by the analyst, allowing the optimum peak integration and background correction points to be selected. The Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software was used for data acquisition and provides easy options for post-analysis data manipulation. Qtegra ISDS Software has integrated Quality Control (QC) checks that allows the user to define automatically controlled actions to ensure data integrity, as well as a Flags and Limits function which flags samples according to user specifications.

Sample preparation

Samples were prepared using certified reference material (CRM) standards. 0.5 g aliquots of rice flour (NBS1568b) and bovine liver (NBS1577c) were digested in 9 mL TraceMetal™ grade nitric acid (Fisher Chemicals, Loughborough, UK) using a standard food method programme in a high-pressure microwave system. The final digests were made up to 50 mL with deionised water before analysis.

Standard preparation

High purity standards (1000 mg kg$_{-1}$ single element standards) were used to prepare the calibration standards for this method. All samples and standards were acid matched. Table 3 indicates the concentration of each of the standards, selected to cover the concentration range of the samples.

Results

The instrument was calibrated using a blank and at least two standards for each element. After inspection, a linear fit was applied to all elements. The calibration standards and samples were analysed in a single sequence with an acid rinse between samples. The sample data was measured by interpolation and results are shown in Table 4. Suitable dilutions were made to over-range elements to ensure they fell within the calibration range. Method detection limits (MDLs) were also established by analysing the acid matched calibration blank using a ten-replicate analysis and multiplying the standard deviation of the analysis by three. This was repeated three times and the average values for method detection limits are also summarised in Table 4.

Conclusion

The results table shows that major and trace elements were measured with equal success. Precise, accurate results for digested foodstuff samples are easily attained on the Thermo Scientific iCAP PRO X Duo ICP-OES instrument. The full wavelength coverage of the unique CID detector allowed the optimum wavelengths to be selected and the whole spectrum to be measured with one exposure, while the sensitivity of the axial view provided the low detection limits for this application.

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Table 3. Standard concentrations in mg kg^{-1}.

Element	STD 1	STD 2	STD 3	STD 4
Pb	0.01	0.05	0.2	0.5
Mn, Ni, Zn, Cu	0.1	0.5	2	5
Ca, P, Fe, Mg	1	5	20	50

Table 4. Results of measurement, CRM and MDLs in mg kg^{-1} and calculated recoveries in %.

Element	SRM1577c			SRM1568b			MDL
	CRM	Measured	Recovery (%)	CRM	Measured	Recovery (%)	µg kg^{-1}
Ca	131	124	95	118.4	107	90	1.90
Cu	275.2	272.3	99	2.35	2.13	91	0.4
Fe	197.94	205.97	104	7.42	7.78	105	1.5
Mg	580	579	100	549	478	87	1.23
Mn	10.46	10.12	97	19.2	18.1	94	0.04
Ni	0.0353	0.0325	92	—	—	—	0.52
P	—	—	—	1490	1302	87	12
Pb	0.0628	0.0623	99	<MDL	—	—	2.9
Zn	179.1	163.3	91	19.16	15.59	81	0.28

COVID-19: Lock-down and up-skill

Antony N. Davies[a,b] **and Henk-Jan van Manen**[b]
[a]SERC, Sustainable Environment Research Centre, Faculty of Computing, Engineering and Science, University of South Wales, UK
[b]Expert Capability Group – Measurement and Analytical Science, Nouryon Chemicals BV, Deventer, the Netherlands

With a significant proportion of our regular readership probably under home lockdown, we were wondering if we could help you at this difficult time by pointing out some useful online resources. So, when we finally come out of this pandemic, you could do so better skilled and more up-to-date than when we went in to it. Indeed, it is extremely rare in our modern lives that we get the chance to sit back and reflect on what we can be doing better, more efficiently and more collaboratively. Take the opportunity to download and test out some of the free versions of innovative data processing and reporting software that is available. There are also numerous very informative video tutorials available around new instruments and accessories, so go explore. This column will point you at a few resources you might like to use to start off. If you find some great resource yourself or feel we have overlooked something you yourself have put online, please let us know (and/or use the online commenting facility) and we will look to publicise the resource.

As many of us are home… let us start there

At https://www.spectroscopyasia.com, Ian and the team have brought together articles, news, product information and events together by technique area.

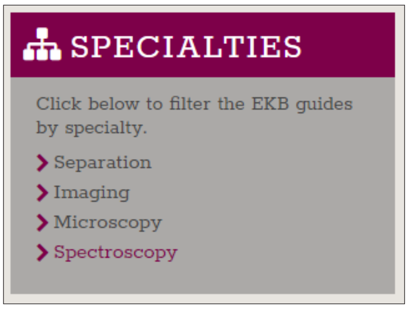
While it can never be definitive, you might find some interesting leads to follow by looking at activities in techniques you currently don't have in your own laboratory. The content is a useful mix of high-level introductory content down to detailed discussions of specific research results in targeted problem areas—so something for most tastes.

With the demise of other sites integrating information across multiple vendors, this is a good starting point. The majority of instrument and software vendor websites unfortunately are now focussing more and more on promoting their specific products rather than helping potential new customers in understanding the various techniques in general.

It is worth looking for general interest areas within the offerings of the scientific publishers. One example from Wiley is the Essential Knowledge Briefings—a resource I must admit I had never heard of before, and these are quite nice relatively short summaries of different techniques with examples of their application which can be downloaded not only as PDFs but also in Kindle and Apple device formats for different eReaders (https://www.essentialknowledgebriefings.com). One little warning: if you are searching for articles you might like to read, be aware that the pre-defined filters under the box "Specialties" are a little arbitrary (Figure 1). Raman Microscopy is under the categories Imaging, Microscopy and Spectroscopy, which is fine, but the Dynamic Secondary Ion Mass Spectrometry briefing I wanted to read was only in Microscopy! What is very nice to see is that the reference lists for further reading with each briefing are not restricted to Wiley publications.

Instrument vendor resources

One of the instrument vendor websites which includes non-product specific training videos from basics to more advanced applications is hosted by Thermo Fisher Scientific (https://www.thermofisher.com/uk/en/home/industrial/spectroscopy-elemental-isotope-analysis/spectroscopy-elemental-isotope-analysis-learning-center.html). It also does not require you register to access the content.



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Click below to filter the EKB guides by specialty.

- Separation
- Imaging
- Microscopy
- Spectroscopy

Figure 1. Preconfigured "Specialties" filters on the Wiley Knowledge Briefings site—make sure you use with care.

Their Spectroscopy, Elemental & Isotope Analysis Learning Center includes their Academy sections of introductory videos and webinars going right back to the first principles and explanation of the terms used without overloading the viewer with sales pitches for their products. Obviously as you move to more detailed webinars, such as the webinar on the Near Infrared Analysis of Biofuels for example, you will encounter more product-focused content. However, this is well mixed in with a good discussion of the benefits of the technique as a whole in this application area against other analytical approaches and as such is quite educational. The lessons you will learn are then equally applicable to a range of product solutions from other vendors.

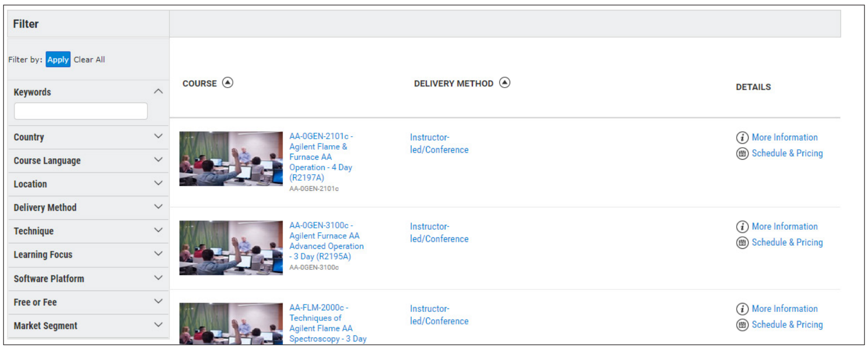
For educators, there are also resources available in the Chemistry in the Classroom Resource Center with several pre-planned lessons and lab experiments. The individual links there lead to content ranging from simple single experiments to full multiple-experiment course plans or quite involved sample preparation, extraction and measurement practicals.

Agilent also hosts a vast amount of content within the Agilent University (https://inter.viewcentral.com/events/cust/catalog2.aspx?cid=agilent&pid=1&lid=1). Unfortunately, Agilent still require a lengthy personal registration process be completed to access any of this content and it appears that some of the content hides behind a pay-wall. If you persist and register yourself, then there is some very interesting educational content, although it does take some finding. Don't forget to tick on the "remember me" box or you will be forced to fill in all your details for every single content access.

Agilent's approach is different to Thermo's in the way the content is built with almost a complete dump of all their resources: generic information presentations as well as very targeted highly specific instrument operation training in one location. Before you give up in despair, you need to use the filters on the left-hand side of the screen, which are very useful in firstly filtering on "free" content and then drilling down to those subject areas you are actually interest in, rather than scrolling through pages and pages of links, see Figure 2.

Once you have mastered their search strategy, there is some excellent content, such as a long and detailed webinar on troubleshooting GC separation: very helpful for the large number of us with GC-MS systems delivering huge numbers of analyses. There is also content on the main Agilent website (https://www.agilent.com/en/training-events/eseminars). This is a different domain and your stored login information from the "University" won't be recognized, so you will have to repeat the process here. The content is made available in different ways, so for some content it is not possible to run the training in full-screen mode… for others the installation of, for example, the Cisco WebEx browser extension plug-in will be required. Try also https://www.agilent.com/en/training-events/eseminars as a route in, where you will get a list of future live webinars you can sign up for as well as access to the large library of recordings.

Bruker as you would expect also makes a lot of information available that you can benefit from looking at during your enforced absence from normal working practices (for example, https://www.bruker.com/se/service/education-training/webinars/optical-spectroscopy.html). Here I was rather confused by the "register to order" the webinar link for the recording I wanted to view. I then received an email confirming that I had requested the webinar. The reason appears to be the age of the webinar I was interested in viewing. Bruker makes use of YouTube to host their more recent webinars and if you follow the links they provide, this content is (obviously) available without the need to register. There are also some webinars hosted directly on Bruker.com. One drawback I found was that accessing their site, similarly to Agilent, initially presents content in a multitude of languages not making use of my browser's language settings. Automatically filtering in this way would be useful to control the amount of content initially presented to the user.



COURSE	DELIVERY METHOD	DETAILS
AA-0GEN-2101c - Agilent Flame & Furnace AA Operation - 4 Day (R2197A) AA-0GEN-2101c	Instructor-led/Conference	More Information Schedule & Pricing
AA-0GEN-3100c - Agilent Furnace AA Advanced Operation - 3 Day (R2195A) AA-0GEN-3100c	Instructor-led/Conference	More Information Schedule & Pricing
AA-FLM-2000c - Techniques of Agilent Flame AA Spectroscopy - 3 Day	Instructor-led/Conference	More Information Schedule & Pricing

Figure 2. Agilent University webinars: use the Filter function on the left side to simplify identifying interesting content.

Now please don't just stick to the big multi-national vendors, there is plenty of very good educational content made available by the more specialist instrument manufacturers and software vendors; these smaller vendors have a tendency to keep their content more up to date.

Download and try-out

So now you are overwhelmed with on-line training, let's move to an area where you can take what you learn and potentially improve the working environment for you and your colleagues when the world returns to normal. There are several vendors who allow you access to fully functioning copies of their software on a trial basis. This is a very generous offer, which I think you probably now have time to make the most of. I am sure that many of you have had the experience of being given a fantastic demonstration of a software product by an experienced sales person or software engineer, only later finding out that the operations you were shown only really worked on the test data provided by the vendor or the specific operations demonstrated in a predefined order they were shown.

Mestrelab Research, for example, will allow you to install a fully functioning version of their Mnova data processing suite (https://mestrelab.com/software/mnova/) for you to test as you wish. There are some very helpful videos supporting the functionality you will be trying out. Finally, are you bored of up-skilling yourself on your own? Have you been forced by COVID-19 to realise that your collaborative environments are well short of what they need to be? Are you still having to use that old USB stick to get data off your spectrometer… oh dear! You could look at another Mestrelabs fully functioning trial, of their cloud-based electronic laboratory notebook solution Mbook. This has a free 45-day license and the capability of adding your co-workers into your test environment for a more realistic exploration. Once you have completed the registration, you will have a short wait for the environment to be set up after which you will be ready to enroll your colleagues for a truly collaborative trial—maybe role-playing different positions within your organisation.

Now there are many other vendors with trial or cut-down versions of their software available for free for you to install and test now, but this column certainly doesn't have the space to list them all. So, as mentioned above, if you find something you would like to recommend to others or have content that fits the criteria we are trying to use in this column then please get in touch.

Learn a new skill

Design of Experiments

Those who know me well will never forgive me if I didn't use this opportunity to suggest that you should also use the available time to look to getting some training in skills that will make you—whether academic or industrial spectroscopist—better at delivering your daily workload when the world gets back to normal. One area I have been preaching about has been the wider use of Experimental Design or Design of Experiments (DoE). You might be working on anything from driving efficiency in a multi-million euro chemical plant or finding the best settings for a new analytical method—adopting the DoE approach will enable you to deliver more robust solutions faster than if you attempt to carry out the work the old-fashioned way optimising one factor at a time. There are several strong vendors in this field, but I have been looking at Design Expert by StatEase, as our University has recently deployed it along with the chemometrics package Unscrambler from Camo AS. With our PhD students in lockdown, I have been suggesting they try out the freely available DoE training available from StatEase. As with other vendors they offer extensive paid training courses, but what caught my eye was some freely available training in what they call the Stat-Ease Academy (eLearning) (https://www.statease.com/training/academy/). Having signed up, the training starts off with a short (if embarrassing) test on your level of basic statistics knowledge. Going on at a nice slow pace you can take the PreDOE: Basic Statistics for Experimenters course which might be a little basic for some of you. However, it does provide some essential explanation of terms that you will use later in the courses.

If you want to try out the concepts as you go along and don't have access to the DesignExpert package, you can download a fully functional version of the package on a one-month temporary license to play with.

Project management

I would also like to take the opportunity to push for better Project Management skills, especially for those who do not primarily see themselves as project managers in the classical sense (i.e. spectroscopists!). For those working in research, development or any environment that involves change, having a basic understand of good project management techniques can certainly make your life simpler and more efficient. An additional benefit of learning to structure your projects well is the removal of any sort of blame culture when issues arise around delivery. Documenting the aims of a project at the beginning and having them signed off by your management against planned costs and timelines with milestones, binds all levels of stakeholders into a transparent team with clear roles and responsibilities and targets on delivery.

We successfully trained over 60 scientists, engineers and support staff in different levels of project management over a three-year period resulting in significant improvements in the planning, execution, documentation, quality and timely closure of their project work. We chose to standardise on the PRINCE2 Project Management qualifications (PRojects IN Controlled Environments https://www.axelos.com/best-practice-solutions/prince2) as PRINCE2 is very flexible and scalable, allowing the methodology to be tailored to meet our differing internal project demands. At the start, we focussed on classroom delivery style, as many of the concepts were new to the teams so the ability to question and discuss in the formal sessions and outside the structured delivery was important. However, more recently, and perhaps more relevant to this column, was the move to online delivery for the initial Foundation level qualification allowing colleagues to study at their own speeds.

Now if you want to obtain the official PRINCE2 certification, as we wanted for all our staff as part of their continuing professional development, you will need to get your boss to agree this is a useful use of your time stuck at home and pay for the course and examination fee. There are several online course providers so I won't go out on a limb to recommend any specific vendor and they will vary depending on where you are located. Regardless of the provider you select, the examinations are all centralised to ensure quality and conformity.

Conclusion

There is far too much content to list everything here, but please think a little out of the box when looking for educational material to make good the time you must spend at home in the coming months. Self-discipline will help in ensuring that when you are finally allowed back into a normal working rhythm you are still useful to your colleagues. Being self-critical or being honest about the strengths and weaknesses of one technique or another is quite rare in our field. One final recommendation could be the Metrohm webinars: https://www.metrohm.com/en-gb/support-and-service/webinar-center/ where (once they came back online) it was refreshing to see the speakers in the NIR spectroscopy webinar I watched actually going into details of when the technique they were "selling" was weak and should not be used. Nice work! Talking of NIR spectroscopy, seven of the series of ICNIRS conference proceedings are freely available at https://www.impopen.com/nir-proceedings ranging from 1995 to 2017.

Questions your company should be asking when procuring sampling equipment

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Perhaps you recognise the situation?

Your company has recently realised that several sampling stations along the "lot-to-aliquot" flow-path are, in fact, not delivering what was originally promised by the manufacturer of the sampling equipment. What evidence of representativity did the manufacturer or the company selling you equipment and solutions demonstrate? Or, perhaps your company has realised that your product is not always in accordance with the specifications of which you are rightfully proud; perhaps this was discovered after many more claims than expected? Or perhaps your company has conducted an intensive audit, where a consulting expert very quickly, all too quickly for comfort, zoomed in on "obviously non-representative samplers" installed at one or a number of locations? As a consequence, your company has made critical decisions partly based on what has hitherto been viewed as dependable analytical results. It has now turned out that this was based on invalid analytical aliquots, ultimately stemming from non-representative primary samples from the target lot. Worse, such deficiencies are far from always known or transparent to management. This column outlines what are reasonable demands to manufacturers or sellers of sampling equipment, systems or solutions.

Who/what is responsible?

When this matter comes up at your company's next board meeting, it is inevitable that one or more diligent board members will pose critical questions:

- "WHY did this company so glibly accept assurances of *representativity* without demanding proof?"
- "WHO is responsible for sampling in this company?"
- "WHY did this company not know enough to challenge the manufacturer to demonstrate sampling representativity after installation but before accepting delivery?"
- "WHY did this company not have enough in-house expertise to demand such on-line performance on our own material?"

(A competent board will have many more pertinent questions, but let that be for now…)

Readers should know by now that there are quite specific rules, principles and design principles which must be in compliance with the world's only existing, science-backed framework for sampling, the Theory of Sampling (TOS), in order for a sampling process to be representative. These prerequisites are codified in the world's only standard for representative sampling, DS3077.[1]

If you did not hear this statement from your sampling equipment manufacturer or the pertinent sales department, something is very wrong! It is only fair of you/your company to demand that the TOS is explicable, and is indeed explained readily and voluntarily by any competent sampling equipment and sampling system solutions sales representative.

Even though, at first sight, companies may appear to work with different materials and lots, and under apparently very different operating circumstances, they in fact all potentially face exactly the same issues as outlined above. Sampling—representative sampling along the complete lot-to-analysis pathway—is often a hidden elephant in the room; not only in the board room but on the shop floor as well, and everywhere in between. Lack of proper attention to this critical aspect in very many sectors of materials processing and manufacturing can very easily lead to unrecognised faulty decisions by management.

Sampling has for decades been considered a complex subject, and a subject that is difficult to do anything about. This is a completely wrong understanding, however…. Answers to all these pertinent questions, and many more, can be found easily enough in the recently published literature, for example in the entry-level book *Introduction to the Theory and Practice of Sampling*.[2] This overview of practical TOS principles will allow any company that requests representative sampling equipment to understand the fundamental information that is needed—and to able to state the critical questions that are needed to evaluate the various offerings from suppliers.

Minimum background

The following is a general procedure for establishing a representative sampling protocol.

TOS-compliant procedure for representative sampling[1,2]

A Define the analyte(s) of interest

B Delineate and prepare the lot for sampling (geometry, total mass, accessibility)

C Select equipment matching appropriate sampling accuracy demands

D Set the sampling precision required, for example 20%$_{\text{(rel)}}$, or other levels, depending on specific case demands

E Estimate primary sampling variability for the selected quality parameter(s), using a Replication Experiment (RE) or Variographic Characterisation (VC)

F On this basis, for process sampling, decide on sampling intervals (minutes for time basis sampling, or kg for mass basis sampling)

G Ascertain the material nominal top particle size in order to determine an appropriate increment mass

H Determine the number increments to be aggregated into composite samples; establish the number of composite samples required to reach the desired precision level (RE, VC)

I Determine optimal sub-sampling needed in order to deliver a representative analytical aliquot mass

J Variations to this protocol may occur, reflecting different material heterogeneities

All steps in this protocol development plan must follow the principles laid out by the TOS (Figure 1).[1,2]

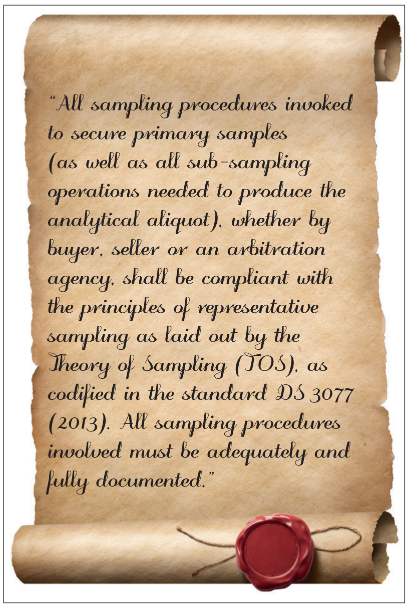
The following is non-negotiable minimum basis for representative sampling competence.

- **Critical success factor 1:** Corporation/company/departmental/individual competence w.r.t the principles of the TOS at a minimum level commensurate with References 1 and 2.
- Critical success factor 2: Corporation/company/departmental/individual *ability* and *willingness* to eliminate or modify sufficiently, wrongly designed or already existing ISE manifestations.

Guide to design and implementation of a sampling plan

The following tasks shall be carried out for each sampling stage *individually* (Figure 2).

1) **ISE reduction/elimination (IDE, IEE, IPE, IWE).** Elimination of all Incorrect Sampling Error (ISE) elements identified at each sampling stage. Before this has been achieved, there is no rational reason to proceed with task 2 below. N.B. Elimination of ISE is in practice only rarely carried out to completion. It is often acceptable to reach a "fit-for-purpose representativity" status which can be assessed by a Replication Experiment (RE) or a Variographic Characterisation (VC) of the performance of installed equipment on the specific material involved, which gives an estimate of the Total Sampling Error (TSE). Summarily this first task on any sampling agenda involves identification and complete or sufficient elimination of one, or more, of the four ISE, i.e. IDE, IEE, IPE, IWE.
2) **CSE reduction.** After partial, or complete, elimination of ISE (task 1), further sampling procedure optimisation is most often obtained by reducing GSE i.e. by *increasing the number of increments* aggregated to composite samples, or by *mixing*, where and when possible. If either, or both, of these procedures do not result in sufficient reduction of TSE (as evidenced by repeated RE or VC), the final resort is *crushing* of the lot or sample material, always followed by thorough *mixing*.
3) Whenever an ISE or CSE modification has been performed, *repeat* TSE estimation by re-doing the RE or VC verification procedure. Compare the TSE estimates, before and after, modification; the reduction in TSE is a



"All sampling procedures invoked to secure primary samples (as well as all sub-sampling operations needed to produce the analytical aliquot), whether by buyer, seller or an arbitration agency, shall be compliant with the principles of representative sampling as laid out by the Theory of Sampling (TOS), as codified in the standard DS 3077 (2013). All sampling procedures involved must be adequately and fully documented."

Figure 1. Theory of Sampling credo.

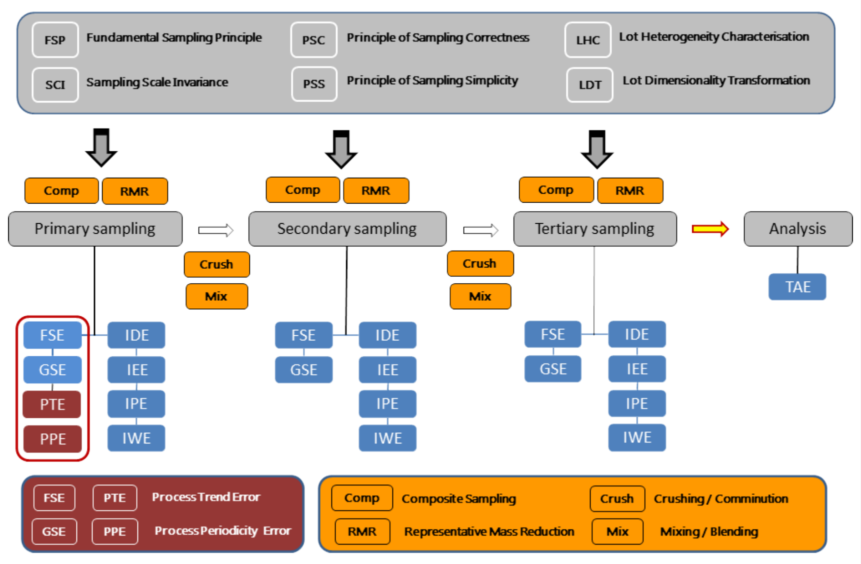


Figure 2. Framework overview of the Theory of Sampling (TOS). Sampling stages, six Governing Principles (GP), six (eight) Sampling Errors (ISE/CSE), four Sampling Unit Operations (SUO). Copyright KHE Consulting.

reflection of the degree of success of reaching the level of "fit-for-purpose" % threshold.[1,2]

4) All sampling procedures are multi-stage. Tasks 1–3 above shall be performed on each stage *individually* (primary, secondary and tertiary sampling stage), making practical implementation of TOS principles much less complicated than often perceived.

5) The primary objective for all sampling plans (for a specific target material of a given heterogeneity, with a given sampling system) is to obtain a representative primary sample (fit-for-purpose status). For materials characterised by large grain-size contrasts, extracting an acceptable primary sample$_{\text{composite}}$ will necessitate a relatively high number of increments distributed so as to cover the entire lot, in direct correlation with the lot heterogeneity—or will necessitate *crushing* of the whole lot material before primary sampling, where such an operation is reasonable. Choosing among these alternatives requires a basic TOS competence and experience, or the sampling plan design/setup should be carried out in collaboration with a sampling expert. Forging ahead without certainty of representativity is a futile exercise, doomed to fail due to introduction of a sampling bias of unknown, and unmanageable magnitude. A sampling bias can only be eliminated by a correctly designed, implemented, operated and maintained sampling process.

6) Once a representative primary sampling process has been obtained, subsequent sub-sampling is an easier and easier task, stage by stage. Once the primary sampling process *accuracy* has been achieved, adhering to similar characteristics for each subsequent sub-sampling stage is immensely easy, as this now only involves sampling precision. Sampling precision can be controlled *in analogo* of traditional statistics—a higher number of increments aggregated as a composite sub-sample will always help to reduce TSE to any practical, fit-for-purpose level desired.

7) The order of execution of these tasks is as follows, for each sampling stage:
Step 0: TSE estimation of existing procedure (RE/VC)
Step 1: ISE elimination/reduction (IDE, IEE, IPE, IWE)
Step 2: TSE estimation of modified procedure (RE/VC)
Step 3: CSE reduction (1. GSE, 2. FSE)
Step 4: TSE estimation of modified procedure (RE/VC)

8) Total Sampling Error (TSE) reduction as mitigated by appropriate sampling procedures. In Figure 3, the relative TSE contributions are proportional to the indicated red/green areas (ISE + CSE).

It is obvious that getting rid of the ISE contribution to the TSE is the absolutely most important task in any development of a representative sampling protocol.

Conclusion

The overwhelming contribution to the TSE is due to incorrect sampling equipment/sampling procedure elements, which have *not* been counteracted w.r.t. ISE (IDE, IEE, IPE; IWE) (red in Figure 3), i.e. in which the sampling bias has *not* been eliminated. Depending upon the empirical heterogeneity of the material being sampled, the ISE-component typically makes up from 50 % to 80 % of the TSE. For this reason, it makes little sense, or no sense at all, to deal with sampling precision (due to CSE only) (green in Figure 3) *without* first having demonstrated successful sampling bias elimination (or significant reduction to comply with a fit-for-purpose representative sampling threshold). This can either take place by improved equipment design (by only using equipment in which sampling bias errors have been fully eliminated), or by optimising sampling procedures by informed, TOS-compliant diligence; or call in a sampling expert.

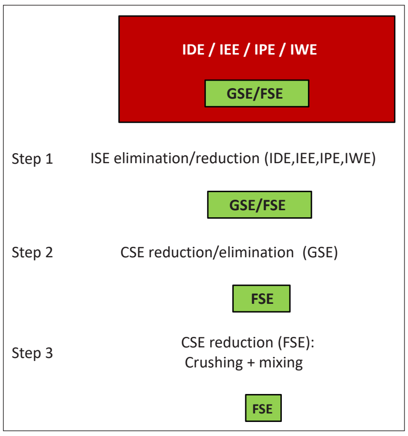


Figure 3. Counteracting TSE by successive ISE and CSE reductions. © KHE Consulting

Fair questions to ask your supplier or sampling equipment provider

1) Please *define* representativity, Incorrect and Correct Sampling Errors (ISE, CSE) and sampling bias.
2) Please *demonstrate* how ISE have been eliminated for the equipment offered.
3) Which recognised *sampling standards* have been used by the equipment supplier?
4) Please provide *evidence* that the offered sampling equipment will operate on this company's materials, under the relevant installation circumstances, with *guaranteed* representativity.
5) Please assure that the equipment supplier is familiar with the facilities of i) Replication Experiment (stationary lots) and ii) Variographic Characterisation (process sampling).

This list of five sampling Quality Control questions will reveal with certainty whether your company is interacting with a TOS-competent sales department, or *not*…. Please ask for answers in writing, and make sure it is understood that *unsubstantiated* claims e.g. "representative sample equipment" will not be acceptable.

References

1. DS3077, *Representative sampling—Horizontal Standard*. Danish Standards (2013). https://www.ds.dk
2. K.H. Esbensen, *Introduction to the Theory and Practice of Sampling*. IMP Open, Chichester, UK (2020). ISBN: 978-1-906715-29-8, https://www.impopen.com/sampling

Introduction to the Theory and Practice of Sampling

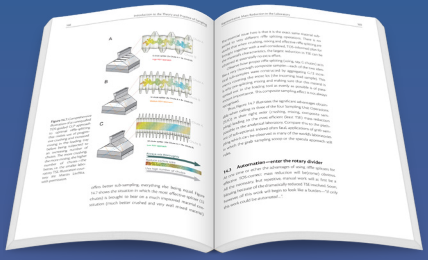
Kim H. Esbensen

NEW BOOK

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.

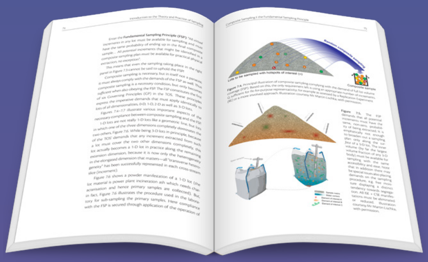
This book presents the Theory and Practice of Sampling (TOS) starting from level zero in a novel didactic framework without excessive mathematics and statistics. The book covers sampling from stationary lots, from moving, dynamic lots (process sampling) and has a vital focus on sampling in the analytical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

PRODUCTS AT PITTCON 2020

Up to a couple of days before I was due to fly to Chicago for Pittcon 2020, I was still unsure whether I would go or not. In the end, with no Government advice not to travel and the USA seeming to be no more affected by Covid-19 than the UK, I decided to go. For many others, the decision had been taken out of their hands by company or Government restrictions, or by their personal circumstances.

Did this affect Pittcon? Of course it did. Some speakers weren't able to give their presentations and some companies weren't able to take up their allocated space in the exhibition. I think I will remember the "Absent due to Travel Restrictions" cards on those stands for a long time. Further, many of the larger instrumentation companies had chosen not to attend Pittcon 2020, but to concentrate on Analytica a month later. In the end, of course, Analytica has been postponed. Nevertheless, the exhibition was still popular, although less busy than normal. Generally, the companies I spoke to were pleased with the people they were talking to: a similar number as in previous years and those were of "high quality".

The organisers did an excellent job in the circumstances. I think it is worth remembering that nearly all of those responsible for putting on Pittcon are unpaid volunteers. There is a small staff, but Pittcon would never happen without those volunteers from the US scientific community. They are all due our thanks.

IMAGING

LabRAM Soleil

HORIBA Scientific introduced the LabRAM Soleil, a multimodal Raman microscope designed for UV-vis-NIR imaging. SmartSampling™ technology makes hyperspectral mapping faster with an image-compression algorithm, and the QScan™ patented confocal imaging system enables scanning of the excitation laser over the sample surface, which provides high confocality of the image multilayer sample, the ability to map the sample without moving it and more agility with point-and-shoot operation, directly on the video image. LabRAM Soleil™ offers benefits such as true self-operation, remote maintenance and instant objective recognition. The LabRAM Soleil can support up to six motorised lasers (four built-in and two external) and up to six spectroscopic modes (including Raman, ULF, photoluminescence and up-conversion luminescence). The LabRAM Soleil™ Raman microscope can be coupled to OmegaScope™, HORIBA's atomic force microscope system, available in different controlled environments, and make spectroscopic measurements at the nanoscale.

HORIBA Scientific

‣ http://link.spectroscopyasia.com/32-023

Breeze Runtime

Breeze Runtime software simplifies the implementation of hyperspectral image analysis into systems and processes. It enables real-time classification of materials based on spectral analysis on pixel and object level. Quantification can also be performed on pixel and object levels, and objects can be identified and their distribution, shape and position analysed.

Prediktera

‣ http://link.spectroscopyasia.com/32-025

INFRARED

ThreatID

RedWave Technology (previously Czitek) were showing their new ThreatID portable diamond ATR, FT-IR analyser for identifying chemical threats. It allows users to dig into the data to a greater extent than is usual with pass/fail systems. For instance, spectra can be zoomed and functional groups highlighted. Data can be exported easily through WiFi, Bluetooth or e-mail.

RedWave Technology

‣ http://link.spectroscopyasia.com/32-033

Model SL-15

ICL's SL-15 is a sealed liquid cell that employs a dynamic O-ring sealing system with fluoropolymer seals that allow the production of precisely calibrated cells without the use of mercury and lead amalgam seals. A wide range of IR transmitting windows, pathlengths, fittings and a choice of Viton or Kal Rez O-rings is available.

International Crystal Laboratories

- http://link.spectroscopyasia.com/32-026

LUMINESCENCE

LanthaLux

This company is a spin-out from the University of Texas at Austin, where research into the discovery of Metal-Organic Frameworks (MOFs) has been undertaken. The LanthaLux reader is a hand-held device using fluorescence detection of LanthaHD test strips with customisable MOF areas consisting of different lanthanides in varying ratios, which create sensors for a broad array of chemicals. Initial applications are for water in organic solvents and isotopic purity. Initial experiments comparing D_2O purity by the LanthaLux/HD method with 500 MHz NMR showed comparable results over a wide range of purities.

Lantha Sensors

- http://link.spectroscopyasia.com/32-037

MAGNETIC RESONANCE

100e

The 100e benchtop NMR spectrometer is based on a 2.35T permanent magnet (corresponding to 100 MHz ^{1}H NMR). It has been developed for routine screening of ^{1}H NMR spectra. It has improved chemical shift dispersion and higher sensitivity, able to run samples at sub-mM concentrations. It is controlled via a large, manoeuvrable touchscreen computer. Options include kinetics package capabilities for reaction monitoring, signal suppression, an experiment designer to write custom pulse programs, an API for developing your own applications, remote control of the instrument from computer, smartphone, tablet etc., non-deuterium lock options, secure login and user profiles with custom project settings and IQ/OQ/PQ protocols.

Nanalysis

- http://link.spectroscopyasia.com/32-031

Formμla

Formμla is a battery-operated, portable time-domain NMR device. It has been designed to provide pass/fail answers to the analysis of unknowns against examples of "good" samples it has been trained on. All analytical decisions are made on-board the device, but analysis results can be uploaded to cloud servers via WiFi or cellular connections. This is of particular value when the instrument is being used to track outbreaks of disease or threats of counterfeit products, for example. Single-use sample tubes with RFID identification are required for analysis.

WaveGuide

- http://link.spectroscopyasia.com/32-032

MASS SPECTROMETRY

JMS-TQ4000GC improvements

Jeol's GC triple quad mass spectrometer system has improvements to the collision cell which result in increases in sensitivity of up to 12×.

Jeol

- http://link.spectroscopyasia.com/32-034

12th Edition of Registry of Mass Spectral Data

The latest edition of the Wiley Registry of Mass Spectral Data has improved spectral search performance, as well the inclusion of chemical classifications, calculated Kovats retention indices and splash IDs. An additional 41,450 spectra have been added, making a total of 817,290.

Wiley

‣ http://link.spectroscopyasia.com/32-027

MALDImini-1

Shimadzu has released the MALDImini™-1, a compact MALDI digital ion trap mass spectrometer, fitting in a space the size of an A3 piece of paper. It provides fast high-sensitivity measurements and detailed structural and qualitative analyses over a wide mass range, even with sub-µL sample volumes. The Digital Ion Trap (DIT) technology, along with the design of the laser optics, sample stage and vacuum exhaust system, reduces the space needed for the device. The small size and corresponding lower power requirements allow it to be plugged into a conventional AC power supply.

Sample and matrix are simply spotted on the sample plate, dried and loaded into the instrument. Analysis can begin in as little as five minutes. This fast, simple setup enables users to conduct and repeat even in-depth structural analyses in a short time span. The system's DIT uses rectangular wave RF to allow ion trapping up to 70,000 Da. This capability, when combined with a MALDI source, enables high-sensitivity MS^n analysis in a broad range of applications. In addition to checking the mass of smaller biomolecules, such as peptides and glycans, the digital RF waveform allows analysis of higher molecular weight species up to 70,000 Da. Furthermore, the MS/MS and MS^3 functionality of the DIT allows researchers to carry out comprehensive structural analyses, such as direct glycopeptide analysis, post translational modification analysis, and branched glycan structural analysis.

Shimadzu

‣ http://link.spectroscopyasia.com/32-035

GC/ID

Cerno Bioscience were demonstrating their MassWorks Rx GC/ID version 1.1, a fully automated data processing software to improve GC/MS qualitative analysis. The new version includes an enhanced approach to identifying and deconvolving mixtures and the background of co-eluting peaks to minimise the misidentification of compounds in complex samples.

Cerno Bioscience

‣ http://link.spectroscopyasia.com/32-029

NEAR INFRARED

DNIRA

The DNIRA is a distributable near infrared analyser based on the Texas Instruments DLP NIR chip. The DLP chip and a drift correction technology Neolitics have developed enables the instrument to maintain calibrations without the need for environmental housings; hot-swapping of cloned spare instruments is also possible. The analyser only requires an Ethernet connection (wired or wireless), connection to a standard power output and to a fibre-coupled process interface, enabling installation within 30 minutes. Wavelength range is 1300–2400 nm.

Neolitics

‣ http://link.spectroscopyasia.com/32-024

SAMPLE PREPARATION

Centri

Markes International has extended its Centri® automated sample extraction and enrichment platform to include additional functionality and the ability to increase or decrease the level of automation. The modular Centri platform can now be configured in a more user-defined way than before and makes trap-enabled preconcentration, extraction and enrichment available to a wider range of laboratories. The new capabilities include smart microchip technology for headspace, SPME and SPME-Arrow sampling accessories. The chip automatically loads parameters such as syringe/fibre type, provides details on minimum and maximum recommended conditioning temperatures and records start of usage when installed, providing improved reliability, traceability and ease-of-use. Liquid handling (e.g. addition of derivatisation reagent or internal standard) enables automated, unattended sample preparation. It supports trap-enabled SPME-Arrow, expanding the portfolio of extraction and enrichment techniques available. Automated GC inlet injection, replacing this normally manual step frees up the user's time. Intelligent diagnostics for automated system self-checking, including valve operation and leak isolation along the flow path. The ability to identify where a leak is enables rapid corrective action, in some cases without the need for an engineer to be available, decreasing instrument downtime.

Markes International

▸ http://link.spectroscopyasia.com/32-030

SOFTWARE

Batch Genealogy in Luminata

Luminata is a chemistry, manufacturing and controls (CMC) development decision support tool providing a single organisational source for all project materials. The new Batch Genealogy feature enables organisations to track each batch of the manufacture of their products from internal and external manufacturing sources with analytical and compositional results. This enables potential issues in the manufacture of individual batches to be spotted quickly and easily and steps taken to avoid the release of the final product.

ACD/Labs

▸ http://link.spectroscopyasia.com/32-036

UV/VIS

New UV/vis series

Shimadzu introduced the new UV-i group of UV-Vis spectrophotometers, consisting of six models: UV-1900i, UV-2600i, UV-2700i, UV-3600i Plus, SolidSpec-3700i and SolidSpec-3700i DUV. All of the spectrophotometers in the series include an automatic pass/fail determination for improved efficiency. The systems are equipped as standard with a spectral evaluation function in the software that automatically determines whether data satisfies specified criteria. This function helps to improve the efficiency of quality control operations by eliminating the manual data analysis steps required after spectra are acquired. The UV-i series of spectrophotometers also include automatic measurement, by connecting an autosampler unit, the systems can analyse up to 360 samples automatically. Used in combination with the spectral evaluation function, the entire process, including pass/fail determination, can be fully automated.

The UV-1900i double-beam spectrophotometer provides high resolution, low stray light, an ultra-fast scan function and an easy-to-use interface. The spectrophotometer can be operated stand-alone (without PC) using the touch panel or with a PC with the included LabSolutions UV-Vis software.

UV-2600i and UV-2700i double-beam spectrophotometers are research-grade instruments. The high-absorbance measurement capability and sub-nanometre bandwidths make them suitable for analysis of band gap, optical coatings and thin films, biological samples like DNA and proteins, and routine work. They are equipped with Shimadzu's propriety Lo-Ray-Ligh® diffraction gratings for low stray light levels to obtain high-accuracy measurements of samples.

The UV-3600i Plus UV-Vis-NIR spectrophotometer has measurement capability in the UV, visible and up to 3300 nm in the NIR. The device enables characterising all types of samples including solids, powders, wafers, films and liquids.

The SolidSpec-3700i and SolidSpec-3700i DUV (Deep-UV) spectrophotometers can measure from deep-UV to visible and up to 3300 nm in the NIR, suitable for optical, semiconductor and flat panel display market needs. Other applications include haze analysis, band gap analysis, photovoltaics, coatings and optical component characterisation.

Shimadzu

▸ http://link.spectroscopyasia.com/32-039

X-RAY

1 mm Thick FAST SDD

A 1 mm thick detector is more sensitive to X-rays around 15 keV, an area where there are many elements of interest, Sn, Cd and rare earths. The new detectors will be of particular interest in µXRF, where focussed X-rays may not use the whole area of the detector. Then, the increased sensitivity of a thicker detector is of particular value. In other applications the thicker detector may actually prove cheaper than a larger area detector with the same sensitivity.

Amptek

▸ http://link.spectroscopyasia.com/32-038

NEW PRODUCTS

ATOMIC

New ICP-OES spectrometers

Analytik Jena's new ICP-OES spectrometers, the PlasmaQuant 9100 and the 9100 Elite, offer more automation, greater adaptability and shorter analysis times. The PlasmaQuant 9100 series is suitable for laboratories with difficult to analyse sample matrices and a steadily increasing number of samples. The target industries for the new solutions include the chemical industry, oil and gas industry as well as the mining and metal processing industry. The PlasmaQuant 9100 series uses Analytik Jena's patented plasma technology, which allows interference-free analyses and the lowest detection limits. Analytik Jena also offers fully automated autosamplers for a large variety of sample types and applications, including solutions for oil sampling, water analysis or for highly volatile samples. New switching valves optimise analysis times by reducing sample uptake and wash out times.

Analytik Jena

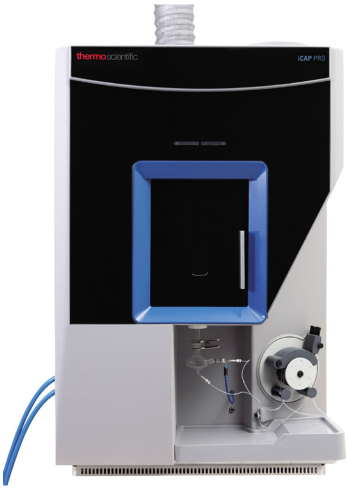
- http://link.spectroscopyasia.com/32-020

iCAP PRO series ICP-OES instrument platform

The Thermo Scientific iCAP PRO Series ICP-OES platform has been designed to provide a fast, sensitive range of trace element analysis solutions capable of capturing the complete spectrum of high-matrix samples in a single run. The iCAP PRO also starts from standby in five minutes, and gas consumption is reduced within a vertical dual-purged optical pathway interface that has lower installation requirements due to the use of a standard wall socket and low extraction flow rate. The combination of a single optical slit and a charge injection device detector deliver increased analysis speeds and readout, reducing the number of measurements per run to obtain a complete spectrum. Stable optics and polychromator reduce the requirement for recalibration of the instrument.

Thermo Fisher Scientific

- http://link.spectroscopyasia.com/32-022



IMAGING

Lightweight, airborne hyperspectral camera systems

Specim's new AFX10 and AFX17 systems are compact solutions for lightweight (2.5 kg) hyperspectral imaging that can be mounted, for example, on a drone. The AFX10 consists of a hyperspectral imaging camera, on-board computer and a high-end GNSS/IMU unit; spectral range is 400–1000 nm. The AFX17 covers the range of 900–1700 nm with a higher frame rate than the AFX10. Both systems are based on the FX product family, which has been designed specifically for industrial use. Data is acquired automatically following the waypoints on a flight plan, making the camera systems easy to operate.

Specim

- http://link.spectroscopyasia.com/32-013

INFRARED

FT-IR gas analyser

Bruker has launched the OMEGA 5 FT-IR based gas analyser for automated, high-precision and real-time monitoring of gas concentrations even in complex gas mixtures. It is designed for applications such as process surveillance in production lines, investigation of catalytic processes, determination of gas impurities and scientific research. The OMEGA 5 is a 19-inch rackmount box gas analyser equipped with a multi-reflection gas cell of 5-m optical path length. This gas cell can be heated up to 191 °C, and the pressure as well as the temperature of the target gas are automatically displayed and taken into account during the quantification with the gas analysis software OPUS GA. The optics of OMEGA 5 are sealed and purgeable, which enables the concentration of atmospheric interfering compounds like water and carbon dioxide to be minimised. The DryPath™ option enables atmospheric interfering compounds to be kept at the lowest level without the need for an external purge gas.

OMEGA 5 is equipped with a thermoelectrically cooled detector to measure concentrations down to the ppb range without the need for liquid nitrogen. It can measure with a spectral resolution of better than 1 cm^{-1}, which allows for the quantification of compounds in most gas mixtures with a high degree of overlapping infrared signals.

Bruker Optik

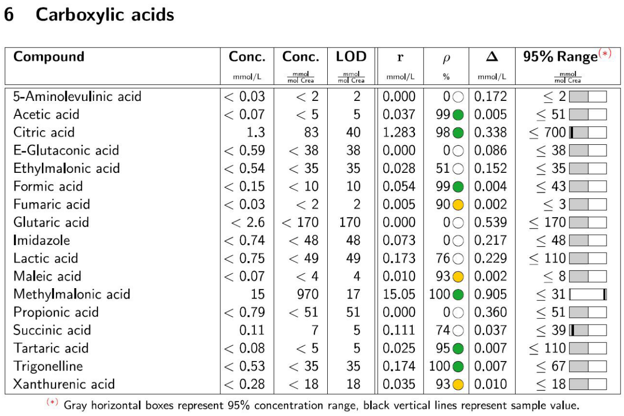
‣ http://link.spectroscopyasia.com/32-028

MAGNETIC RESONANCE

NMR module for validated metabolite quantification in urine

Bruker have announced the release of the B.I.QUANT-UR™ 1.1 module, which complies with DIN-ISO criteria for the identification and quantification of urine metabolites on Bruker's nuclear magnetic resonance (NMR) *in vitro* diagnostic research (Avance™ IVDr) platforms. The module automatically quantifies up to 150 endogenous and disease-related metabolites from urine, allowing users to obtain precise, sensitive and fully reproducible results, even below the limit of detection (LOD).

NMR produces quantification values from a single experiment, which would traditionally require multiple tests with different sample preparations using techniques such as conventional gas chromatography mass spectrometry or ion exchange chromatography mass spectrometry. B.I.QUANT-UR 1.1 combines the raw concentration of a compound with quality assessment parameters to obtain reliable quantification results, and has demonstrated potential in clinical and translational research. The new module introduces two additional parameters to provide confidence in quantification: signal correlation (ρ, %) to characterise the match between the lineshape metabolite signal and the calculated fit, with colour-coded flags added for improved visualisation; and concentration error (Δ, mmol L^{-1}), for the concentration equivalent of the difference between metabolite signal and the calculated fit.



6 Carboxylic acids

Compound	Conc.	Conc.	LOD	r	ρ	Δ	95% Range(*)
	mmol/L	$\frac{\text{mmol}}{\text{mol Crea}}$	$\frac{\text{mmol}}{\text{mol Crea}}$	mmol/L	%	mmol/L	$\frac{\text{mmol}}{\text{mol Crea}}$
5-Aminolevulinic acid	< 0.03	< 2	2	0.000	0 ○	0.172	≤ 2
Acetic acid	< 0.07	< 5	5	0.037	99 🟢	0.005	≤ 51
Citric acid	1.3	83	40	1.283	98 🟢	0.338	≤ 700
E-Glutaconic acid	< 0.59	< 38	38	0.000	0 ○	0.086	≤ 38
Ethylmalonic acid	< 0.54	< 35	35	0.028	51 ○	0.152	≤ 35
Formic acid	< 0.15	< 10	10	0.054	99 🟢	0.004	≤ 43
Fumaric acid	< 0.03	< 2	2	0.005	90 🟡	0.002	≤ 3
Glutaric acid	< 2.6	< 170	170	0.000	0 ○	0.539	≤ 170
Imidazole	< 0.74	< 48	48	0.073	0 ○	0.217	≤ 48
Lactic acid	< 0.75	< 49	49	0.173	76 ○	0.229	≤ 110
Maleic acid	< 0.07	< 4	4	0.010	93 🟡	0.002	≤ 8
Methylmalonic acid	15	970	17	15.05	100 🟢	0.905	≤ 31
Propionic acid	< 0.79	< 51	51	0.000	0 ○	0.360	≤ 51
Succinic acid	0.11	7	5	0.111	74 ○	0.037	≤ 39
Tartaric acid	< 0.08	< 5	5	0.025	95 🟢	0.007	≤ 110
Trigonelline	< 0.53	< 35	35	0.174	100 🟢	0.007	≤ 67
Xanthurenic acid	< 0.28	< 18	18	0.035	93 🟡	0.010	≤ 18

(*) Gray horizontal boxes represent 95% concentration range, black vertical lines represent sample value.

A key challenge in quantifying metabolites in urine is the complexity of the sample, which contains thousands of compounds generating multiple NMR signals. B.I.QUANT-UR includes DIN-ISO conformant wet spiking, to identify correct signals where there is significant overlap. This upgraded version of the successful B.I.QUANT-UR 1.0 module represents another iteration of a unique feature known as numerical spiking, where pure compound spectra of all 150 metabolites are automatically added to more than 20,000 urine spectra, and subjected to the quantification algorithm to determine the respective probabilistic LOD.

Bruker

‣ http://link.spectroscopyasia.com/32-015

NEW PRODUCTS

NIR

PAT and process miniature NIR spectrometer

Viavi Solutions have introduced the MicroNIR PAT-Ux, the newest addition to the MicroNIR family of miniature NIR spectrometers. Like its companion MicroNIR PAT instrument, the PAT-Ux enables real-time monitoring of drying, blending and granulation/tableting operations. The MicroNIR PAT-Ux is ATEX, NEC and IECEx certified for chemical, agriculture, food and feed applications. Derived from the popular MicroNIR PAT-U (designed for ordinary locations), the PAT-Ux is initially being deployed to monitor fluid bed drying in pharmaceutical manufacturing, flour analysis and processing, and explosives manufacturing. The instrument kit includes the MicroNIR Pro software suite, a complete and easy-to-use chemometric modelling package that enables customers to build their own solutions.

VIAVI Solutions

▸ http://link.spectroscopyasia.com/32-017

RAMAN

Handheld SORS raw material ID system

Agilent Technologies has launched the Vaya Raman raw material identity verification system, a handheld instrument that verifies raw material identity through unopened transparent and non-transparent packaging. Incoming goods can be tested quickly in the warehouse on receipt, testing through sealed containers also avoids handler exposure to high potency APIs and maintains the shelf life of sterile contents, helping to prevent unnecessary waste.

Vaya is the first true through-barrier handheld Raman instrument, incorporating Agilent's spatially offset Raman spectroscopy (SORS) technology for fast ID of incoming goods through opaque containers in a GMP-ready package. Vaya combines SORS with conventional Raman spectroscopy for maximum compatibility with a wide range of packaging—from clear glass vials and plastic bags to opaque plastics and paper sacks.

Agilent Technologies

▸ http://link.spectroscopyasia.com/32-021

Raman in bioreactor systems

Sartorius has introduced the BioPAT® Spectro, a Quality by Design (QbD) tool for use with its ambr® automated micro and mini bioreactor systems and with the BIOSTAT STR® single-use production bioreactors. The new tool offers access to Raman spectroscopy analysis in high-throughput process development, enabling faster Raman model building and scale-up into commercial manufacturing. The new BioPAT Spectro Tool, can be retrofitted into ambr 15 cell culture and ambr 250 high throughput systems, and enables quick and simple sampling for integrated, at-line analysis of a large number of analytes by Raman spectroscopy in a Design of Experiments (DoE) approach.

BioPAT Spectro consists of a multi-use flow cell integrated into the ambr analysis module, and is compatible with Raman spectrometers from Kaiser Optical Systems and Tornado Spectral Systems. The online Raman spectroscopy data, alongside data from the ambr process and selected integrated analysers, is automatically collated by the ambr software and can be easily transferred to SIMCA® Multivariate Data Analysis (MVDA) software for model building.

The BioPAT Spectro has been developed with scalability in mind and its flow cell's Raman probe interface and optical path design are identical across scales allowing automated in-line Raman spectroscopy with Sartorius' BIOSTAT STR® single-use bioreactors. An integrated qualified and ready-to-use single-use spectroscopy port in Flexsafe STR bags for BIOSTAT STR bioreactors will be launched in the near future.

Sartorius

▸ http://link.spectroscopyasia.com/32-016

TERAHERTZ

High-speed THz system for industrial quality control

Measurement speed becomes particularly relevant if the task involves rapidly moving samples, e.g. thickness gauging in extrusion lines or items on fast conveyor belts that may need to be screened with single-millimeter resolution. In contrast to conventional terahertz time-domain spectrometers, the TeraFlash smart does not use any mechanical delay unit, but includes two synchronised femtosecond lasers with an electronic delay ("electrically controlled optical sampling", ECOPS). This concept results in very high measurement speeds: the TeraFlash smart acquires 1600 complete terahertz waveforms per second.

Within a measurement time of only 625 µs, the TeraFlash smart achieves a spectral bandwidth of 3THz. The dynamic range of the pulse trace is greater than 50 dB. Within one second of averaging, this value increases to >80 dB and the bandwidth exceeds 4THz. Users can flexibly adjust the terahertz path length from 10 cm to 180 cm. The fibre-coupled transmitter and receiver antennas can be flexibly positioned and enable measurements in transmission or reflection.

Reflection measurements are particularly suitable to determine layer thicknesses, for example to investigate the wall thickness of plastic pipes or the structure of polymer coatings or single- and multi-layered paint. The 10 m long fibre cables allow a spatial separation of the control unit from the measurement head, which can even be mounted on a robot arm.

TOPTICA Photonics

▸ http://link.spectroscopyasia.com/32-018

X-RAY

Benchtop X-ray elemental analyser

Bruker have introduced the next-generation of their benchtop energy dispersive X-ray fluorescence (ED-XRF) spectrometer, S2 Puma Series 2. The instrument now has HighSense technology, which increases throughput by about a factor of 3×. Bruker's Spectra.Elements software comes with enhanced features and faster algorithms, leading to ~40 % shorter evaluation times. The S2 PUMA Series 2 can be used for elemental analysis applications from cement, steel, mining and petrochemical, to food analysis and pharma QC. A new mapping stage also extends its applications into semiconductors and coatings, where spatial resolution is required.

Bruker AXS

▸ http://link.spectroscopyasia.com/32-019

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- ☐ None

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DIARY

Notes on the Diary

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

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

Conferences

2020

24–28 May, Chiba, Japan. **Japan Geoscience Union (JpGU) Meeting 2020.** 🌐 http://www.jpgu.org/en/articles/20171208meetingplan.html.

1–12 June, Online. **ASMS 2020 Reboot.** 🌐 https://www.asms.org/conferences/asms-2020-reboot/welcome-to-asms-2020-reboot.

28 June–4 July, Gangwon, South Korea. **AOGS 17th Annual Meeting.** ✉ info@asiaoceania.org, 🌐 http://www.asiaoceania.org/society/public.asp?view=up_coming.

29–30 June, Ivine, United States. **RamanFest 2020.** 🌐 https://www.ramanfest.com/.

25–31 July, Chambersburg, United States. **International Diffuse Reflectance Conference (IDRC) 2020.** info@cnirs.org, 🌐 http://www.cnirs.org/.

17–18 August, Cairns, Queensland, Australia. **19th Australian Near Infrared Spectroscopy Group (ANISG) Conference.** ✉ secretary@anisg.com.au, 🌐 https://anisg.com.au/.

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

6–10 September, Singapore, Singapore. **SETAC 8th World Congress.** ✉ setac@setac.org, 🌐 https://singapore.setac.org/.

13–16 September, Orlando, United States. **134th AOAC International Annual Meeting & Exposition.** ✉ meetings@aoac.org, 🌐 http://www.aoac.org.

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

30 September–2 October, Amsterdam, Holland. **11th Workshop on Hyperspectral Image and Signal Processing: Evolution in Remote Sensing (WHISPERS).** 🌐 http://www.spectroexpo.com/whispers/.

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

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

15–20 December, Honolulu, Hawaii, United States. **The International Chemical Congress of Pacific Basin Societies 2020.** 🌐 https://pacifichem.org/.

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

15–21 February, Houston, United States. **2021 AAFS 73rd Annual Scientific Meeting.** 🌐 https://www.aafs.org/home-page/meetings/future-past-aafs-meetings/.

2021

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

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

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

5–9 June, Minneapolis, Minnesota, United States. **70th ASMS Conference.** 🌐 https://www.asms.org/conferences/annual-conference/future-annual-conferences.


thermo scientific