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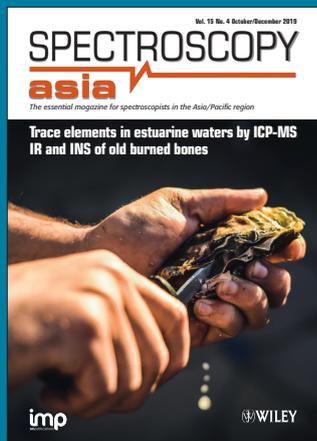
SPECTROSCOPY

asia

The essential magazine for spectroscopists in the Asia/Pacific region

**Trace elements in estuarine waters by ICP-MS
IR and INS of old burned bones**





Shellfish, such as oysters, are susceptible to storing and concentrating pollutants. The article starting on page 12 looks at the direct analysis of trace elements using triple-quadrupole inductively coupled plasma-mass spectrometry.

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WILEY

imp
implications

Earth, fire and water

Don't worry, this is not an issue on ancient science, but these three of the "elements" do reflect the content of the issue. We have articles on old burned bones buried in the earth (that's two in one) and detecting trace elements in sea water.

In our first article, Stewart Parker and colleagues describe what "Old burned bones tell us about past cultures". Burned bones are often found in archaeological sites as a result of fire or funerary practices and are often the only preserved human remains. Using inelastic neutron scattering, infrared and micro-Raman spectroscopies, the authors can reach definitive conclusions as to the temperature at which the bone was burned. This enables archaeologists and anthropologists to learn more about how ancient civilisations used fire for funerary, burial or cooking purposes.

Our second (Sponsored) article is about "Direct analysis of trace elements

in estuarine waters using triple-quadrupole inductively coupled plasma-mass spectrometry" by Lionel Dutruch, Jörg Schäfer, Mélima Abdou and Daniel Kutscher. Estuaries are where much of our marine pollution is to be found, being the gateway between the land and the ocean. They are also where much of our seafood comes. So, knowledge of elemental contamination in estuaries is vital to protect us from the ingestion of polluted seafood.

Do you have the budget for a new instrument? Well, stop... wait! Tony Davies and Marian Draaisma have very useful guidelines so that you can use "Spectroscopic data supporting investment decisions". Tony and Marian run through questions you should be asking yourself (and your team), information you should gather and how you should go about the selection process for a new spectrometer. If you're not buying just now, this is definitely a column you will want to keep for future reference.

The Quality Matters Column highlights how the reference material industry has grown. Peter Jenks asks for your help in rerunning a 2001 survey with aim of establishing how users' use of and experience with reference materials has changed. Whilst most questions are optional, such surveys are of the greatest value if most questions are answered. We hope you can spend a few minutes to complete the survey at <https://www.spectroscopyasia.com/reference-material-survey-2019>.

The Sampling Column starts a second stage of its life. Having, mostly, covered the theory of sampling up to now, the second part will focus on sampling in practice as well as other topics that Kim Esbensen sees as valuable. The "new" Sampling Column starts with "Revisiting the Replication Experiment" by Paul Bédard and Kim.

If you're thinking of conferences to attend next year, the Diary has plenty to whet your appetite.

We're on your wavelength

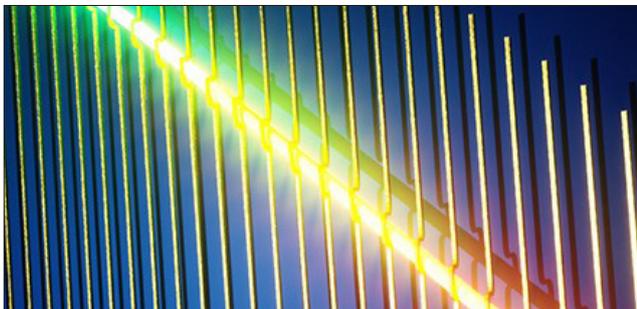
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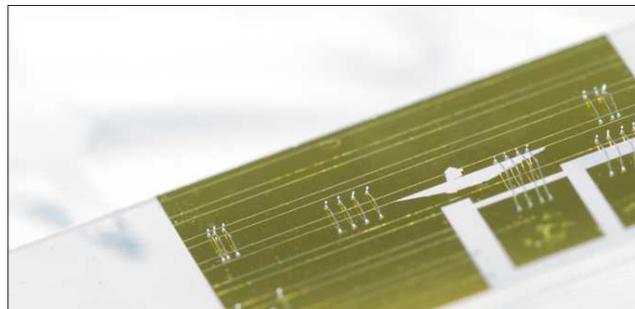


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The miniaturisation of spectrometers continues both in size reduction and in the range of technological approaches



Artist's impression of single-nanowire spectrometer. Credit: Ella Maru Studio



Chip spectrometer, ~2cm in length. (Photograph: ETH Zurich/Pascal A. Halder)

The smallest spectrometer ever designed

The device, made from a single nanowire, is the smallest spectrometer ever designed. It could be used in potential applications such as assessing the freshness of foods, the quality of drugs or even identifying counterfeit objects, all from a smartphone camera. A team from the University of Cambridge, working with colleagues from the UK, China and Finland, used a nanowire whose material composition is varied along its length, enabling it to be responsive to different wavelengths across the visible spectrum. Using techniques similar to those used for the manufacture of computer chips, they then created a series of light-responsive sections on this nanowire.

"We engineered a nanowire that allows us to get rid of the dispersive elements, like a prism, producing a far simpler, ultra-miniaturised system than conventional spectrometers can allow", said first author Zongyin Yang from the Cambridge Graphene Centre. "The individual responses we get from the nanowire sections can then be directly fed into a computer algorithm to reconstruct the incident light spectrum."

"Our approach could allow unprecedented miniaturisation of spectroscopic devices, to an extent that could see them incorporated directly into smartphones, bringing powerful analytical technologies from the lab to the palm of our hands," said Dr Tawfique Hasan, who led the study.

One of the most promising potential uses of the nanowire could be in biol-

ogy. Since the device is so tiny, it can be used for direct spectral imaging of single cells without the need for a microscope. The researchers hope that the platform they have created could lead to an entirely new generation of ultra-compact spectrometers working from the ultraviolet to the infrared range. Such technologies could be used for a wide range of consumer, research and industrial applications, including in lab-on-a-chip systems, biological implants and smart wearable devices. Details are reported in *Science* (doi: [10.1126/science.aax8814](https://doi.org/10.1126/science.aax8814)).

The Cambridge team has filed a patent on the technology, and hopes to see real-life applications within the next five years.

Chip-based infrared spectrometer

Researchers at ETH Zurich have developed a chip-based, infrared spectrometer. David Pohl and Marc Reig Escalé, in the group headed by Rachel Grange, Professor of Optical Nanomaterials in the Department of Physics, collaborated with other colleagues to develop a chip about 2cm² in size. With it, they can analyse infrared light in the same way as they would with a conventional spectrometer. In the device, the incident light is analysed with special waveguides of an optical refractive index that can be adjusted externally via an electric field. This enables the generation of an interferogram.

Depending on how the waveguide is configured, researchers can examine different regions of the electromagnetic spectrum. "In theory, our spectrometer

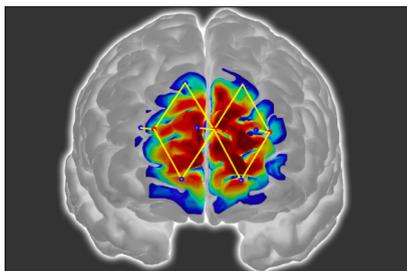
lets you measure not only infrared light, but also visible light, provided the waveguide is properly configured", Escalé says. In contrast to other integrated spectrometers that can cover only a narrow range of the light spectrum, the device developed by Grange's group has a major advantage in that it can easily analyse a broad section of the spectrum.

For their spectrometer, the ETH researchers employed a material that is also used as a modulator in the telecommunications industry. This material has many positive properties, but as a waveguide, it confines the light to the inside. This is less than ideal, as a measurement is possible only if some of the guided light can get out. For this reason, the scientists attached delicate metal structures to the waveguides that scatter the light to the outside of the device. "It required a lot of work in the clean room until we could structure the material the way we wanted", Grange explains.

The work is at an early stage, and the spectrometer currently uses an external camera, which would need to be integrated in any final device. Details are reported in *Nature Photonics* (doi: [10.1038/s41566-019-0529-9](https://doi.org/10.1038/s41566-019-0529-9)).

fNIR spectroscopy can detect pain levels

Researchers from MIT and elsewhere have developed a system that measures a patient's pain level by analysing brain activity from a functional near infrared spectroscopy (fNIRS) device. The system could help doctors diagnose and treat pain in unconscious and non-communicative patients, which could reduce the



Researchers from MIT and elsewhere have developed a system that detects pain in patients with an fNIRS device, which could help doctors diagnose and treat pain in unconscious and non-communicative patients. Credit: Researchers and MIT

risk of chronic pain that can occur after surgery.

Pain management is a surprisingly challenging, complex balancing act. Overtreating pain, for example, runs the risk of addicting patients to pain medication. Undertreating pain, on the other hand, may lead to long-term chronic pain and other complications. Today, doctors generally gauge pain levels according to their patients' own reports of how they are feeling. But what about patients who cannot communicate how they are feeling effectively, or at all, such as children, elderly patients with dementia or those undergoing surgery?

The researchers use only a few fNIRS sensors on a patient's forehead to measure activity in the prefrontal cortex, which plays a major role in pain processing. Using the measured brain signals, the researchers developed personalised machine-learning models to detect patterns of oxygenated haemoglobin levels associated with pain responses, which predicted whether a patient is experiencing pain with around 87% accuracy.

"The way we measure pain hasn't changed over the years," says Daniel Lopez-Martinez, a PhD student in the Harvard-MIT Program in Health Sciences and Technology. "If we don't have metrics for how much pain someone experiences, treating pain and running clinical trials becomes challenging. The motivation is to quantify pain in an objective manner that doesn't require the cooperation of the patient, such as when a patient is unconscious during surgery."

Traditionally, surgery patients receive anaesthesia and medication based on their age, weight, previous diseases and other factors. If they do not move and their heart rate remains stable, they are considered fine. But the brain may still be processing pain signals while they are unconscious, which can lead to increased post-operative pain and long-term chronic pain. The researchers' system could provide surgeons with real-time information about an unconscious patient's pain levels, so they can adjust anaesthesia and medication dosages accordingly to stop those pain signals.

NIR spectroscopy shows that heart disease in our ancestors

A new study of the mummified arteries of people who lived thousands of years ago using near infrared (NIR) spectroscopy has revealed that their arteries were more clogged than originally thought, according to a proof-of-concept study published in the *American Heart Journal* (doi: [10.1016/j.ahj.2019.06.018](https://doi.org/10.1016/j.ahj.2019.06.018)).

"I wanted to see if heart disease is a modern-day problem. It appears to have been a problem for a very long time", said Mohammad Madjid, the study's lead author and an assistant professor of cardiovascular medicine at the McGovern Medical School at UTHealth, Texas, USA.

In the past when researchers have analysed the hearts and arteries of mummies, they used computed tomography that creates images of blood vessels, organs and bones. However, these scans detect only accumulated calcium in the arteries, not the build-up of cholesterol. Madjid said his team is the first to examine mummified arterial remains from different parts of the world with a technique that can detect cholesterol: NIR spectroscopy.

Madjid's samples included mummified arterial tissue from three men and two women ranging in age from 18 to 55–60. Three died presumably of pneumonia and one of renal failure. The cause of death for the fifth person is unknown. Four lived in South America and one in the Middle East. They lived from the late Chinchorro era, 2000 BC, to Cabuza, 350 to 1000 AD.

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Cholesterol build-up is a hallmark of atherosclerosis from the very early stages, while calcium accumulation is a sign of late stages of the disease. Therefore, relying only on calcium shown in CT scans underestimates the true prevalence of the disease, Madjid said. Madjid noted that factors such as exposure to smoke from fire pits, viral infec-

tions, bacterial infections and bad genes might have contributed to the plaque build-up in people living centuries ago. The build-up was also present in people at a relatively young age, he said.

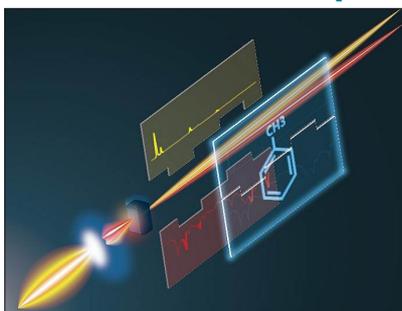
The study offers new insight into the earlier pathological stages of atherosclerosis, showing a prevalence of cholesterol-rich plaques even in ancient times,

the authors reported. Madjid plans to examine additional mummified remains to see how widespread the arterial problems were. The authors concluded, "Non-invasive near infrared spectroscopy is a promising technique for studying ancient mummies of various cultures to gain insight into the origins of atherosclerosis."

New technique measuring infrared and Raman spectra simultaneously

Researchers at the University of Tokyo have combined infrared and Raman spectroscopy into a new technique they call complementary vibrational spectroscopy. The complementary information available from Raman and infrared spectroscopies has been appreciated for some time, but in the technique they are combined and measured simultaneously.

Advances in ultrashort pulsed laser technology have made complementary vibrational spectroscopy possible. Inside the complementary vibrational spectrometer, a titanium-sapphire laser illuminates the sample with pulses of near infrared (NIR) light with a width of 10 fs. Before reaching the sample, the light is focused onto a crystal of gallium selenide. The crystal generates mid-infrared light pulses. The NIR and mid-infrared light pulses are then focused onto the



sample, and the absorbed and scattered light waves are detected by photodetectors and converted simultaneously into Raman and infrared spectra. So far, researchers have tested their new technique on samples of pure chemicals commonly found in labs. They hope that the technique can be used to understand how molecules change shape in real time.

Artists representation of complementary vibrational spectroscopy, which relies on improvements in ultrashort pulsed laser technology. Researchers at the University of Tokyo hope to use complementary vibrational spectroscopy to see molecules change shape in real time without invasive techniques. Image by Takuro Ideguchi, CC BY-ND-4.0

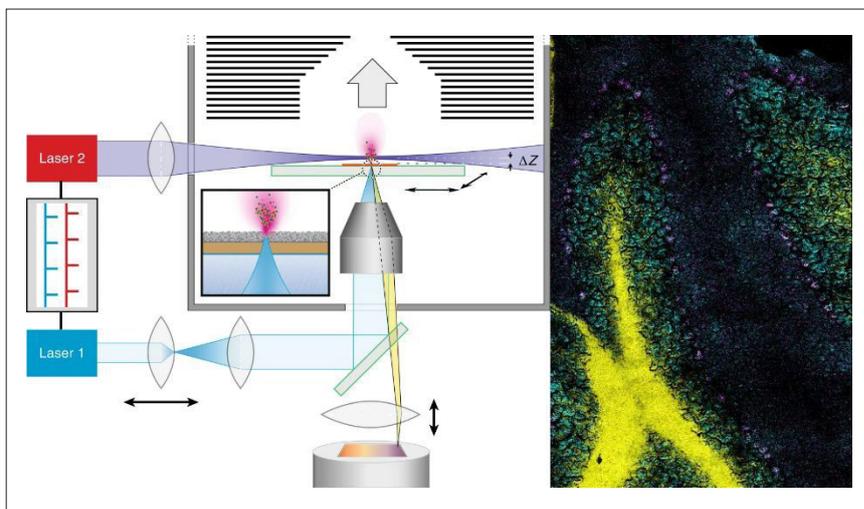
"Especially for biology, we use the term 'label-free' for molecular vibrational spectroscopy because it is non-invasive and we can identify molecules without attaching artificial fluorescent tags. We believe that complementary vibrational spectroscopy can be a unique and useful technique for molecular measurements", said Associate Professor Takuro Ideguchi from the University of Tokyo Institute for Photon Science and Technology.

The new technique is reported in *Nature Communications* (doi: [10.1038/s41467-019-12442-9](https://doi.org/10.1038/s41467-019-12442-9)).

sample, and the absorbed and scattered light waves are detected by photodetectors

Significant increase in MALDI MSI resolution

The working group headed by Professor Klaus Dreisewerd and Dr Jens Soltwisch from the Institute of Hygiene at the University of Münster have developed a method which has improved the spatial resolution of MALDI mass spectrometry by around one nanometre. The researchers have named the new technique t-MALDI-2 (with "t" standing for transmission mode). It uses two specially adapted lasers: one generates a particularly small focus on the material removed, while the other produces the necessary signal enhancement for many biomolecules by up to several magnitudes—for example, for fat-soluble vitamins such as vitamin D, cholesterol or administered medication. Information on their precise distribution in cells and tissues can, among



Left: how the t-MALDI-2-MS imaging method works. Right: an example, in which the complex structure of a mouse's cerebellum is shown by means of the superimposition of three ion signals. © Nature Research/Marcel Niehaus

other things, help to produce a better understanding of disease and inflammation processes and show new strategies for treating them.

“The decisive improvement which our method offers, in comparison with established MALDI imaging methods, is based on the combination and extension of two technical methods previously in use”, explains Dr Marcel Niehaus, one of the two lead authors of the study. “For one thing, in the transmission geometry we irradiate our samples on the reverse side. This enables us to place high-quality microscope lenses very close to the sample, thus reducing the size of the

laser dot. This is different from what is possible, for geometrical reasons, in standard methods—where the samples are irradiated from the direction of the mass analyser.” However, in the minute areas of the sample which are removed by the laser, there is only an extremely small amount of material available for the subsequent MS measurement. The second decisive step was, therefore, the use of a method (called MALDI-2) which the researchers had already introduced in 2015. The effect is that the so-called post-ionisation laser produces an increased transfer of the initially uncharged molecules to an ionic form.

In their study, published in *Nature Methods* (doi: [10.1038/s41592-019-0536-2](https://doi.org/10.1038/s41592-019-0536-2)), the researchers demonstrate the possibilities offered by their technology, taking the fine structures in the cerebellum of a mouse and using kidney cell cultures. “Our method could improve the future understanding of many processes in the body at molecular level”, says Professor Dreisewerd. “Also, established methods from optical microscopy—for example, fluorescence microscopy—could be merged with mass spectrometry imaging in a ‘multi-modal’ instrument”, he adds.

Nuclear magneto-optic method sensitivity increased 100x

The nuclear magneto-optic (NMO) phenomena, the first of which has been observed in 2006, enable emerging methods for studies of materials and molecules. NMO effects arise from the magnetic moments of nuclei which, if properly oriented in space, can cause small changes in the properties of light as it passes through the material. With its ability to look into the matter at the resolution of individual atoms, without permanently altering the sample properties, NMO approaches offer a valuable window into the properties of matter that only a few methods can give. In this respect, the NMO methods are similar to NMR spectroscopy.

The NMR Research Unit at the Faculty of Science of the University of Oulu in Finland has been active in the field of NMO since 200. Lately, the group has also been involved in the development of experimental NMO techniques. The ultimate aim of the NMO research is to provide high-sensitivity optical data with atomic-resolution about the studied material. It is crucial to improve the spectroscopic sensitivity, so that smaller samples can be measured and higher-quality information can be obtained.

The sensitivity improvement can be gained by hyperpolarisation, when the magnets of the atomic nuclei are

oriented in the desired direction to a far greater degree than possible under ambient conditions. In a paper, published in *The Journal of Physical Chemistry Letters* (doi: [10.1021/acs.jpcllett.9b02194](https://doi.org/10.1021/acs.jpcllett.9b02194)), Petr Štěpánek and Anu Kantola of the NMR Research Unit have shown how this can be achieved via the use of specially prepared hydrogen gas.

Hydrogen gas molecules can be present in two forms, *ortho*- and *para*-hydrogen, which differ by the mutual orientation of their own two nuclear magnetic moments. The high degree of

orientational order contained in the gas that contains an excess of *para*-hydrogen, can, via a catalytic reaction, be transferred to the studied molecule, leading to an increase in the observed signal.

The researchers have used this method in a new combined approach and improved the sensitivity of NMO measurements by a factor of more than one hundred. This allows measurements of substances that would otherwise not be viable and opens new possibilities for further development of this new and exciting field.

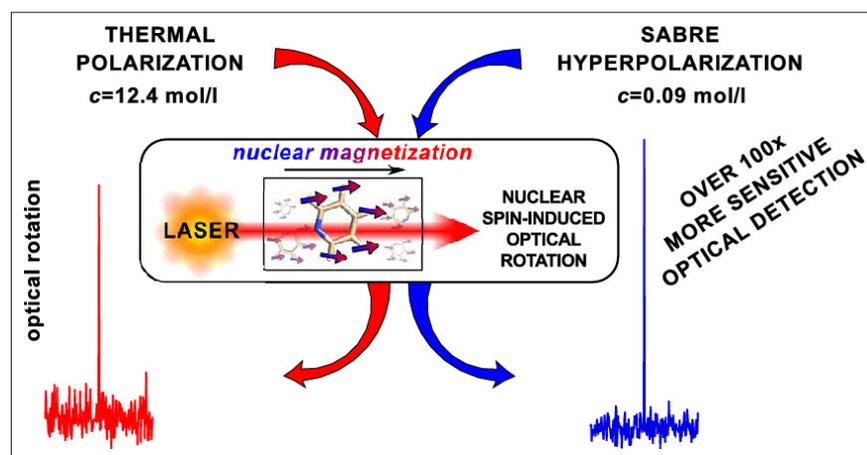


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Old burned bones tell us about past cultures

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Introduction

Bone is a composite biomaterial mainly comprised of packed collagen fibres and an inorganic matrix of crystalline hydroxyapatite [$\text{Ca}_{10}(\text{PO}_4)_6\text{OH}_x$, Hap], the hydroxyl and phosphate groups being partly substituted by carbonate. Bone mineral characteristics, such as crystallinity and carbonate/phosphate content vary as a function of age, sex, location in the skeleton, diet and pathological state of the person. With increasing burning temperature ($>200^\circ\text{C}$), the bone matrix suffers structural and dimensional variations, usually leading to higher crystallinity. These are reflected in the vibrational spectra and a combination of inelastic neutron scattering (INS), infrared and micro-Raman spectroscopies was used by the authors for the investigation of

human bones burned under controlled conditions.¹ This has enabled definitive conclusions about the temperature at which the bone was burned to be made. INS is particularly well-suited for studying hydrogenous compounds.² The neutron scattering cross-section (σ) of an atom is characteristic of that atom and independent of the chemical environment. Since the value for hydrogen ($\sigma=80$ barns) far exceeds that of all other atoms (typically $\sigma \sim 5$ barns), the modes with significant hydrogen displacement dominate the spectra.

Burned bones are often found in archaeological sites as a result of fire or funerary practices and are often the only preserved human remains. The specimens currently under study (both human and faunal) were found at different archaeological sites in Italy, from the Neolithic, the copper age, Roman period and the Middle Ages (Figure 1).

Interpretation of the diagenetic alterations in the analysed samples was based on vibrational spectroscopic results previously¹ gathered for modern human bones burned at well-defined temperatures (400–1000 °C). Hydroxyapatite’s hydroxyl libration (OH_{lib}) at $\sim 630\text{ cm}^{-1}$ in particular, was found to be a very useful diagnostic signal of the burning process, as it was found to undergo a clear shift to lower energy with increasing burning temperature. Comparison between these data and that measured for the archaeological samples has yielded valuable information on ancient practices and customs.

Roman bones

Figure 2 shows the INS spectra of human bones, from the same skeleton, discovered in tomb 36 at the *Guidonia-Montecelio* Roman archaeological site (Figure 1C). The skeleton was found

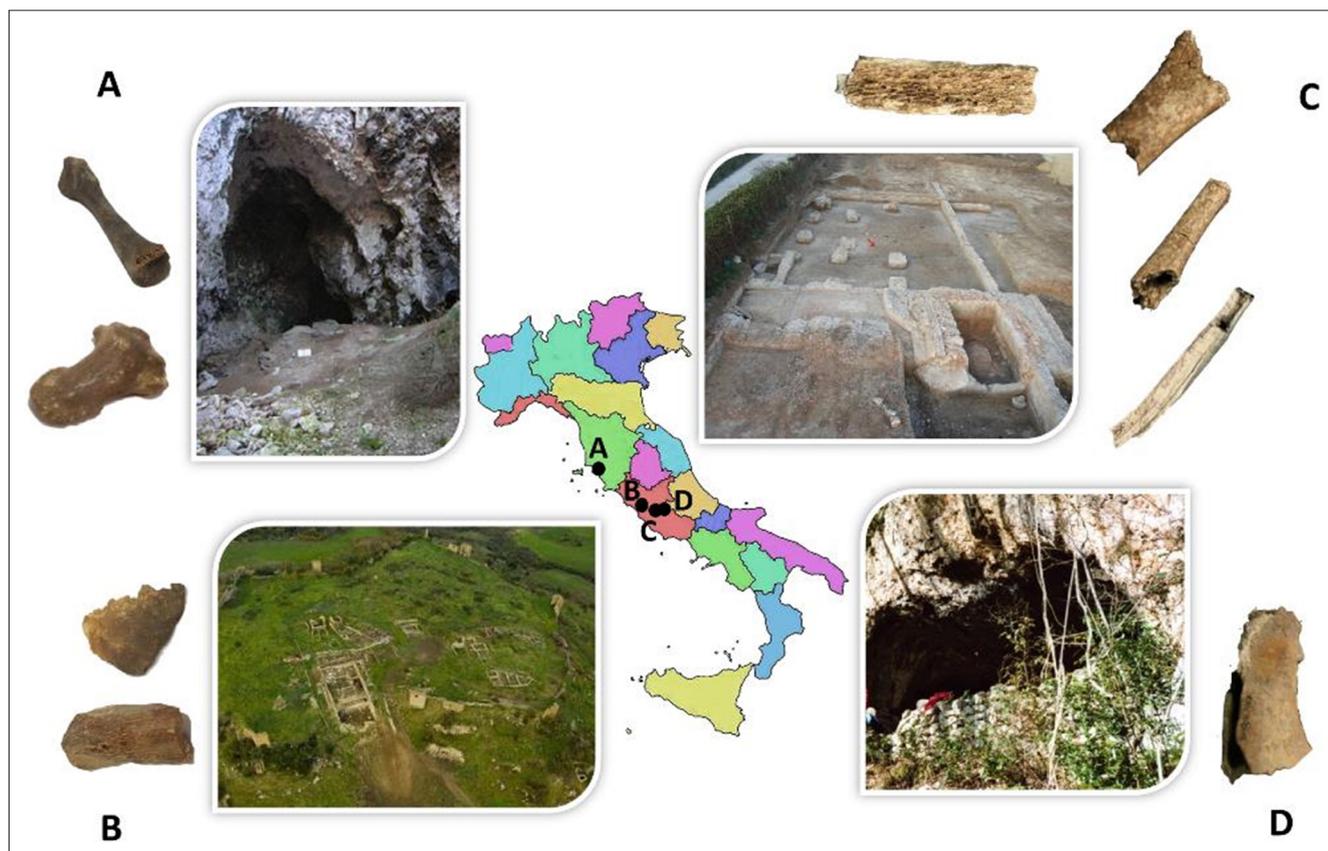


Figure 1. Archaeological sites where the samples investigated were found. A, *Scoglietto* cave—hand distal phalanx and metacarpal bone; B, *Leopoli-Cencelle*—skull and tibia fragments; C, *Guidonia-Montecelio* (tomb 36)—fibula, ulna, femur and humerus fragments; D, *Mora Cavorso* cave—sheep/goat jaw fragment.

intact, incorporated in a clay matrix and placed supine with the arms along the pelvis. Surprisingly, different types of bone from the same skeleton displayed very different INS profiles, consistent with diverse heating conditions from below 400 °C up to 500 °C or 800–900 °C. This can be explained by ancient funerary practices that involved burning the corpse inside the grave. Correlating the heating conditions with the type of bone, it can be seen that the temperature progressively increases for the ulna, femur, humerus and fibula which indicates that the body may have been folded in a foetal position. In addition, for some of these samples a band at $ca\ 900\text{ cm}^{-1}$, ascribed to $\nu(\text{HCO}_3^-)$, was detected (mainly for the fibula), showing contamination from the soil which has a limestone composition in the *Guidonia-Montecelio* region (since the samples were found unwrapped inside the earth tomb). The major factors prone to influ-

ence the degree of combustion of the skeletal remains, and, therefore, the effect on bone composition and crystallinity, are the temperature, the heating time and the environmental conditions (e.g. presence or absence of oxygen). The spectroscopic data depicted in Figure 2 show that the lower area of the skeleton reached higher temperatures than the upper part. Coupled with the discovery of lamp fragments by the feet of the body, it suggests that the burning occurred *in situ* and that the lamp had an active role in this ritual funerary practice.

Copper age bones

The infrared and INS spectra of human hand distal phalanx (PFF12) and metacarpal bone (PFF27) from the copper age, found at the *Scoglietto* cave, a 40 m deep cave located in the *Parco Naturale della Maremma* near Grosseto (Tuscany, Italy) (Figure 1A) are shown

in Figure 3. The FTIR-ATR results show that these samples were subject to heating at temperatures no higher than 450–500 °C. They display the typical infrared features of bone still containing traces of protein (collagen I), mainly evidenced by the amide I band (Figure 3A). The INS data goes further in identifying some lipid components through the corresponding $\delta(\text{CH}_2)$ and $\nu(\text{CH})$ signals at $ca\ 1450\text{ cm}^{-1}$ and 3000 cm^{-1} , respectively (Figure 3B). Upon comparison with the data obtained for reference samples heated at defined temperatures, both spectroscopic signatures correspond to a burning temperature around 500 °C, virtually homogeneous for the three ancient skeletal remains. This temperature is compatible with an incomplete cremation in home fires.

Raman micro-spectroscopy measurements performed on these ancient samples provided clear evidence of the presence of gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$),

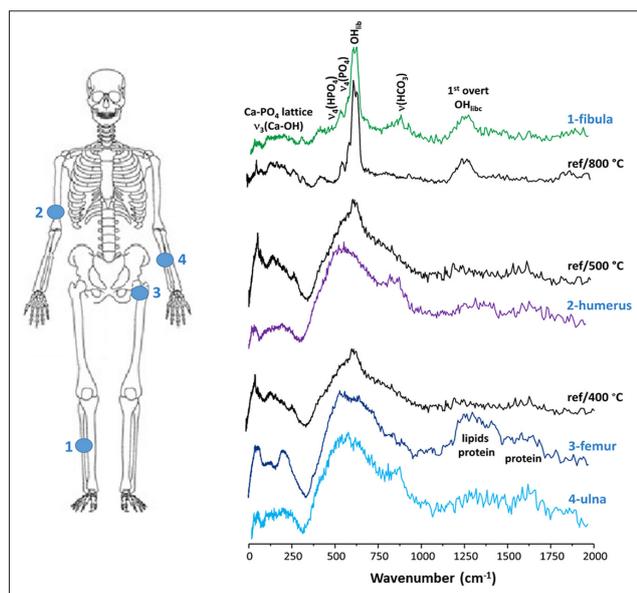


Figure 2. INS spectra of different human bones from the Roman period (humerus, ulna, femur and fibula from the same skeleton, *Guidonia-Montecelio*, Italy, tomb 36). Comparison with human modern bone samples burned at 400°C, 500°C and 800°C.

through the distinguishing band from the sulphate symmetric stretching [$\nu_1(\text{SO}_4)$] at 1008 cm^{-1} and the $\nu_2(\text{SO}_4)$, $\nu_4(\text{SO}_4)$ and $\nu_3(\text{SO}_4)$ signals at $415/493$, 670 and 1136 cm^{-1} , respectively. This constituent is indicative of contamination from the soil surrounding the skeletal remains, as this archaeological area is characterised by a cavernous limestone geology with a high gypsum content.

Medieval bones

Figure 4 shows the FTIR-ATR and INS experimental data measured for human skeletal remains found at the *Leopoli-Cencelle* medieval archaeological site (Figure 1B)—fragments from tibia (US1241) and skull (US1043)—as well as a reference sample from modern bones. *Cencelle* was considered to be a farm because of the presence of estates that specialised in supplying timber to the alum-based industries. The infrared results clearly demonstrate that the medieval samples were subject to heating, although this may have occurred at a later time. Comparison with the infrared spectrum of an intact (modern) human bone showed no evidence of lipids [absence of the $\delta(\text{CH}_2)$ and $\nu(\text{CH})$ signals at $1340\text{--}1460\text{ cm}^{-1}$ and

$2900\text{--}3000\text{ cm}^{-1}$, respectively] and hardly any traces of protein [which would be evidenced by the amide I/II and $\nu(\text{CH}_2)_{\text{collagen}}$ bands at 1570 , 1665 and 2970 cm^{-1}] (Figure 4A). Additionally, the presence of traces of fluorapatite [calcium fluorophosphate, $\text{Ca}_5(\text{PO}_4)_3\text{F}$, generally known as francolite] was revealed in these samples, through the characteristic $\nu_3(\text{PO}_4^{3-})$ feature appearing as a shoulder at 1090 cm^{-1} on the very strong signal of the $\nu_1(\text{PO}_4^{3-})$ band at 960 cm^{-1} .

Regarding the INS results, a remarkable similarity was observed between the profiles of the modern and ancient samples (Figure 4B). The presence of small amounts of lipids and proteins is clearly seen, evidencing a higher sensitivity of the technique relative to infrared spectroscopy for this kind of H-containing constituents. The vibrational profile of the skull fragment (US1043) was found to be comparable to the reference bone burned at 600°C , while that of the tibia (US10114) was identical to the 400°C

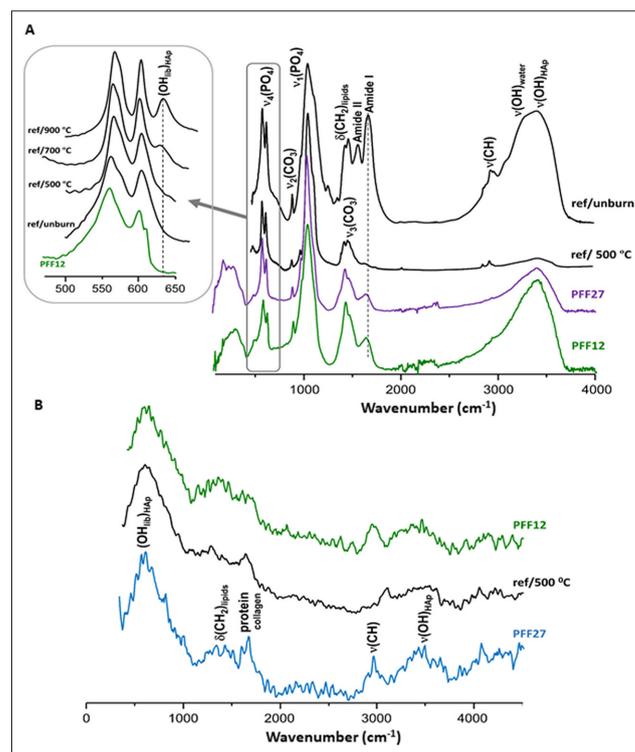


Figure 3. Vibrational spectra of human bones from the copper age (*Scoglietto* cave, Italy). Comparison with modern human bone samples burned at 500°C . A, ATR-FTIR; B, INS. (The inset in Figure 3A depicts the temperature-dependence of the spectral profile in the $500\text{--}650\text{ cm}^{-1}$ interval).

reference. It should be emphasised that these two archaeological samples could only be discriminated through neutron techniques, as the small chemical differences between them were not clearly revealed by infrared spectroscopy. While the FTIR-ATR fingerprint of the medieval bones provided only an average temperature range to which the bones were exposed, INS yielded a precise burning temperature for each analysed sample, being able to differentiate between 400°C (for the tibia) and 600°C (for the skull). These archaeological bones were probably subject to a char process with a concomitant crystallinity increase and almost total loss of the organic constitu-

Approximate dates for periods mentioned

Medieval period: 476–1450 AD
 Roman Empire: 27–476 AD
 Copper (Bronze) Age: 3500–2300 BC
 Neolithic Era: 4500–3000 BC

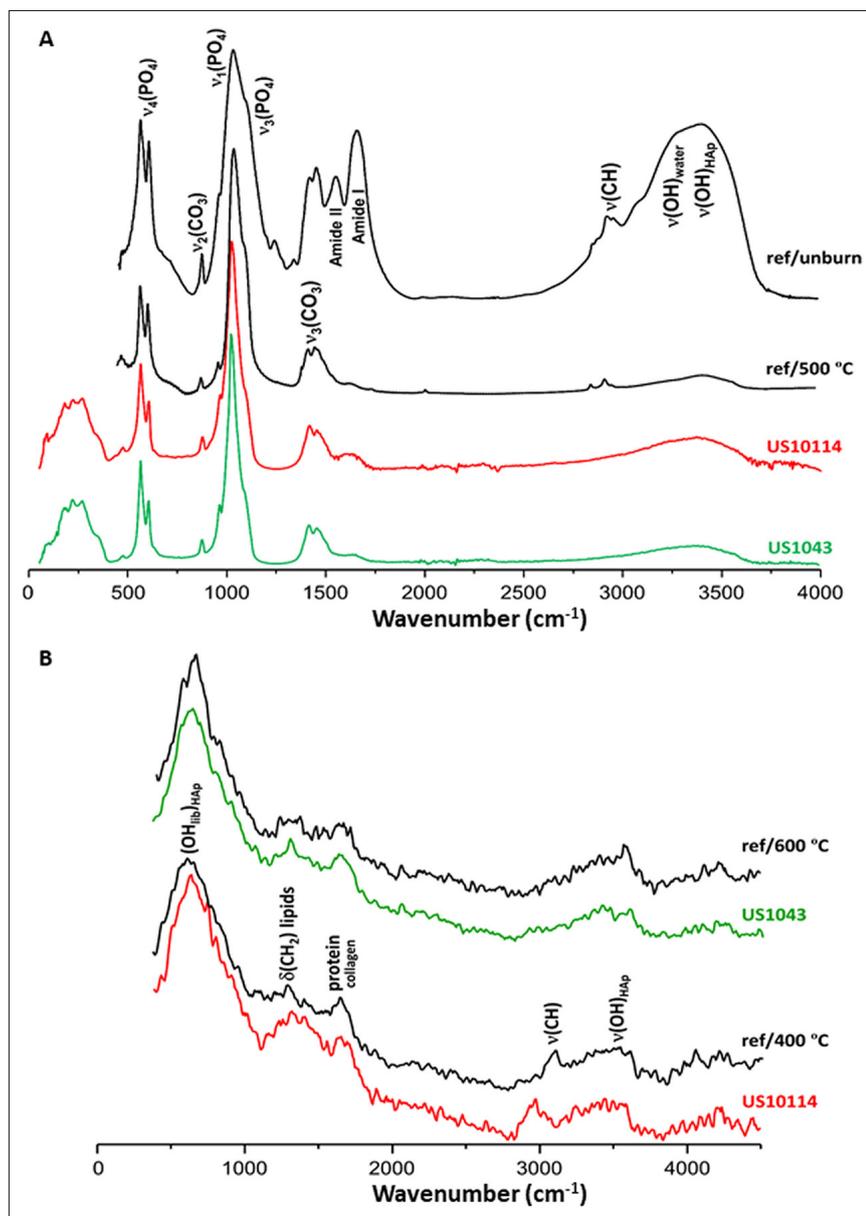


Figure 4. Vibrational spectra of medieval human bones (*Leopoli-Cencelle*, Italy). Comparison with human modern bone samples burned at 400, 500 and 600°C. A, FTIR-ATR; B, INS.

ents: the lipids having been completely destroyed above 400°C, while some collagen is still present within the bone matrix. Additionally, the detection of francolite revealed the occurrence of fluoride in the bone matrix, probably due to environmental contamination from either the neighbouring soil or water.

Neolithic bones

In order to assess the usefulness of this methodology and to extend its capabilities beyond human skeletal remains,

Neolithic faunal bones from the *Mora Cavorso* archaeological site (Figure 1D) were also investigated. By analysis of the most informative vibrational bands (from phosphate, carbonate and bone's organic components), the archaeological remains appear to have been exposed to temperatures below 500°C, which is compatible with the temperatures reached in hearths used in Neolithic settlements for cooking. However, care should be taken when interpreting these data, since it was found that human and

faunal bones display different reactions to heat, especially at higher temperatures.

Conclusions

This study (see Notice below) constitutes the first application of neutron spectroscopy to archaeological skeletal remains and will allow archaeologists/anthropologists to gather relevant information on ancient civilisations regarding the locations, and funerary, burial or cooking practices. Future research will enlarge the number of samples and archaeological sites in order to gather data on a wider range of contexts—geographical, historical and anthropological. Further spectroscopic biomarkers will then be identified, enabling a precise statistical analysis of the results linking particular spectral signatures to specific burning scenarios and environmental settings.

Notice

This article is adapted from: G. Festa, C. Andreani, M. Baldoni, V. Cipollari, C. Martínez-Labarga, F. Martini, O. Rickards, M.F. Rolfo, L. Sarti, R. Senesi, F.R. Stasolla, S.F. Parker, A.R. Vassalo, A.P. Mamede, L.A.E. Batista de Carvalho and M.P.M. Marques, "First analysis of ancient burned skeletal human remains probed by neutron and optical vibrational spectroscopy.", *Science Advances* 5, eaaw1292 (2019) (<https://doi.org/10.1126/sciadv.aaw1292>) published under a CC-BY-NC licence.

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Direct analysis of trace elements in estuarine waters using triple-quadrupole inductively coupled plasma-mass spectrometry

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Introduction

An estuary is an ecosystem that is characterised by both marine (e.g. tides or erosion through waves) and riverine processes (e.g. freshwater and sediment inputs). At the interface between fresh- and sea-water domains, estuarine waters are often referred to as brackish waters with salinities between 1 and up to 35 (corresponding to total dissolved solids between 0.1% and $\leq 3.5\%$). Coastal zones including estuaries are historically populated by humans, hosting major

cities and industrial activities. Estuaries are the focal points of aquatic contaminant transport from the continent to the sea, facing contamination from industrial, agricultural and urban sources, especially over the past decades. Important examples of estuaries are Puget Sound in the north-western US, Rio de la Plata in South America and the Thames Estuary in the UK. In this study, the focus is on the Gironde Estuary in south-west France, a major fluvial-estuarine system. The main objective of

this study is to track and control historical and ongoing multi-metal contamination of the Gironde Estuary waters. Clear evidence for contamination (mainly Cd) has been observed in seafood (oysters) from the estuary mouth and the nearby Marennes-Oléron Bay, Europe's major oyster production area.¹

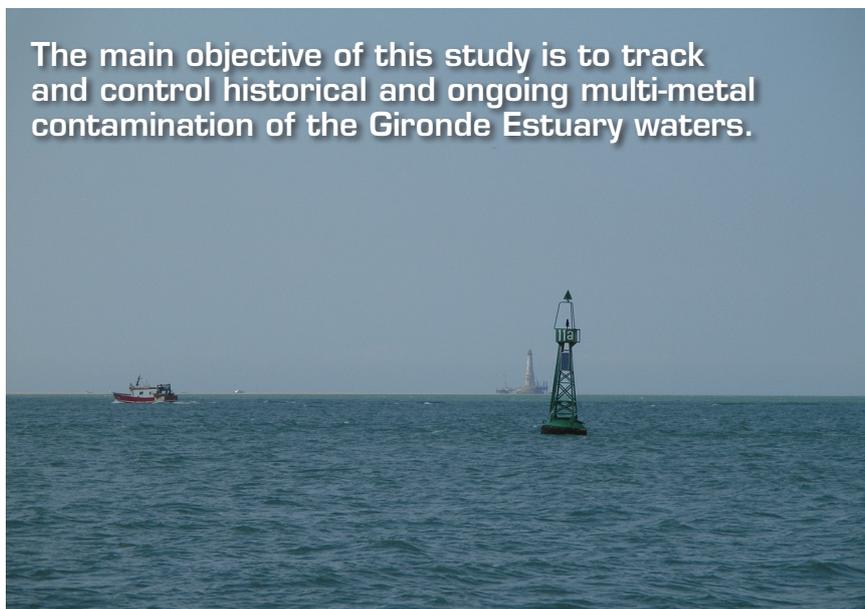
Method

Sample preparation

Estuarine water samples were collected in the high salinity range of the Gironde Estuary, i.e. relatively close to the estuary mouth, ~100 km downstream from Bordeaux, France. Sampling was performed onboard the research vessel *Thalia* (Ifremer) over a cycle of 30 hours, implying that the water masses sampled have variable salinity due to the strong ebb–flood cycle in this meso-/macrotidal estuary. Salinities in the samples ranged from 30.2 to 31.8. To avoid potential matrix effects caused by different salinity, all samples were adjusted to a salinity of 30. The star in Figure 1 shows the sampling site in the Gironde Estuary mouth, which is part of a larger sampling campaign along the entire estuarine salinity gradient (data not shown).

However, the analysis of samples containing high salt loads imposes special challenges when using induc-

The main objective of this study is to track and control historical and ongoing multi-metal contamination of the Gironde Estuary waters.



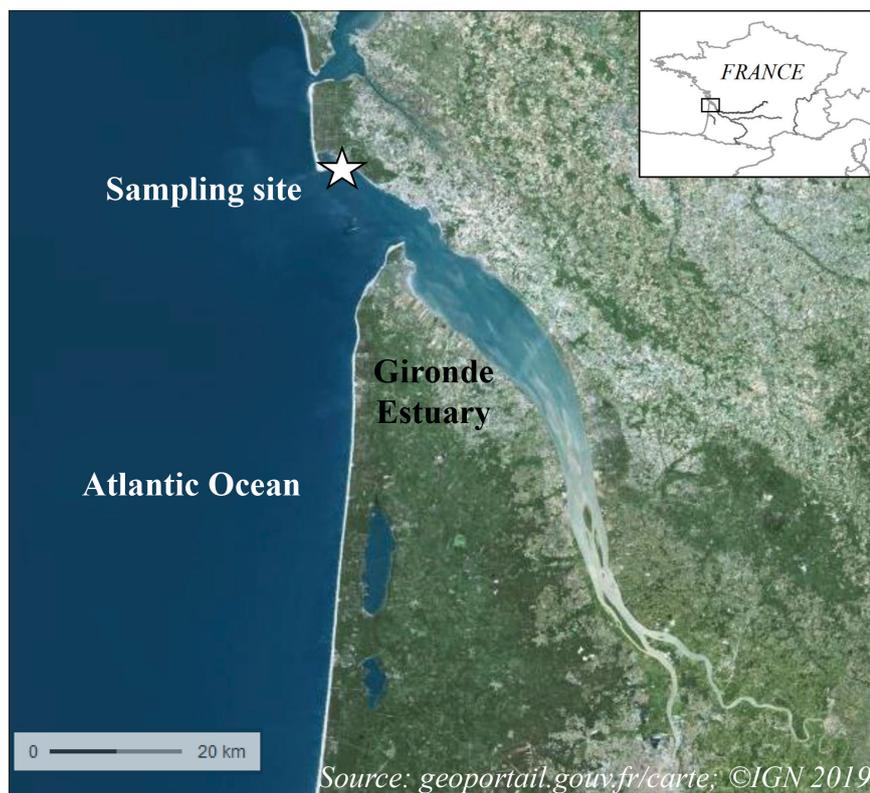


Figure 1. Sampling location in the Gironde Estuary.

tively coupled plasma-mass spectrometry (ICP-MS). Salts may crystallise during the process of nebulisation or deposit on surfaces of the interface region, leading to blockage of nebulisers and cone orifices. This may lead to severe reduction of signals and cause drift problems. With respect to spectral interferences, most commonly chlorine-based polyatomics such as those interfering on vanadium, chromium and arsenic, need to be removed.

Additionally, strong interferences can be observed on copper, based on the presence of sodium and magnesium in estuarine and sea waters.

In order to overcome the impact of the sample matrix, samples can be diluted using clean diluents, but especially for the analysis of elements at trace or ultra-trace levels, dilution always induces the risk of contamination or over-dilution, leading to final concentrations in the measured solution being lower than instrumental detection limits (IDLs). Another appealing alternative is the use of argon gas to dilute the sample before it enters the plasma. Although this also

leads to a significant reduction in achievable instrument sensitivity, method detection limits (taking into account all steps in sample preparation) can be less compromised as compared to liquid dilution.

Due to the extremely low concentrations in seawater commonly observed for some of the analytes (especially Pb, but also Cd), careful control over potential sources of contamination and clean laboratory conditions are key to successful analysis. The labware was acid-cleaned (soaking for three days in 10% HNO₃ Normapure®, VWR-BDH Chemicals), thoroughly rinsed with MilliQ® water (Merck), dried under a laminar flow hood in a clean lab (over-pressurised, filtered, air-conditioned atmosphere), then sealed in double plastic bags until use. All samples were filtered onboard immediately after sampling using 0.2µm membrane filters (MINISART® NML, Sartorius), acidified (1/1000, HNO₃ Suprapur®, Merck), and stored in the dark at 4°C pending analysis.

Instrument configuration

A Thermo Scientific™ iCAP™ TQ inductively coupled plasma mass spectrometer in combination with an SC-4DX Autosampler (Elemental Scientific, Omaha, NE) was used for analysis. The instrument was operated using Argon Gas Dilution (AGD) allowing direct analysis of estuarine waters without any prior dilution.

Tuning of the system was accomplished using the autotune routines provided with the Thermo Scientific™ Qtegra™ Intelligent Scientific Data

Table 1. Instrument configuration.

Parameter	Value	
Nebuliser	MicroMist Quartz nebuliser 0.4mLmin ⁻¹ , pumped at 40rpm	
Spray chamber	Quartz cyclonic spray chamber cooled at 2.7°C	
Injector	2.5mm id, quartz	
Interface	High matrix (3.5mm) insert, Ni cones	
RF power	1550W	
Nebuliser gas flow	0.73Lmin ⁻¹	
Additional gas flow (AGD)	97%	
QCell settings	SQ-KED ^a	TQ-O ₂
Gas flow	100% He, 4.2mLmin ⁻¹	100% O ₂ , 0.3mLmin ⁻¹
CR bias	-21V	-6.4V
Q3 bias	-18V	-12V
Scan settings	0.1s dwell time per analyte, 10 sweeps, 3 main runs	

^aKED: kinetic energy discrimination

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Solution™ Software. Typical operating conditions are summarised in Table 1. The selection of analytes, appropriate analysis mode (single quad vs triple quad, choice of reactive gas) was accomplished using the Reaction Finder method development assistant. For the selected elements, this resulted in only arsenic being acquired in a triple-quadrupole mode using oxygen as the reactive gas, whereas for all other analytes, the use of helium and KED was recommended. The mass selection in the first quadrupole was controlled using intelligent Mass Selection (iMS) in all cases.

General analytical conditions

For calibration and quality control, a certified reference material was used (CASS-6, Nearshore Seawater Certified Reference Material for Trace Metals and other Constituents, National Research Council Canada). Matrix-matched calibration curves were generated by the addition of increasing concentrations of the elements investigated in this study directly into aliquots of the CASS-6 certified reference material (CRM). Table 2 gives an overview on the added concentrations for the different analytes. As the

calibration was matrix matched, no internal standard was used in the analysis. Again, all solutions were adjusted to a salinity of 30.

Following eight to nine unknown samples, CASS-6 was repeatedly analysed by standard addition in order to (i) check the accuracy of the method and (ii) monitor potential sensitivity drift.

Results

The results obtained are summarised in Table 3. As can be seen, quantitative recoveries are obtained for all elements under study in the CASS-6 CRM. The CRM was analysed four times throughout the analysis and demonstrated low relative standard deviations despite the extremely low concentrations of some of the elements. For arsenic (the only element measured in both single- and triple-quadrupole modes), there is no significant difference in the results considering the uncertainty information in the certificate of the CASS-6 CRM. In this sample matrix, the predominant interference on ^{75}As are chlorine- and calcium-based species, such as $^{40}\text{Ar}^{35}\text{Cl}^+$, $^{40}\text{Ca}^{35}\text{Cl}^+$ or $^{40}\text{Ca}(\text{OH})_2\text{H}^+$. Due to their polyatomic nature, these interferences



can be efficiently removed by KED alone. However, it is worth noticing that the triple-quadrupole-based mode using oxygen offered a much higher detection sensitivity (more than double in comparison to KED) and significantly lower detection limits (more than five times lower). Other interferences, such as doubly charged ions of the Rare Earth Elements, might affect the results for elements such as arsenic or selenium, and can only be removed using triple-quadrupole technology. However, in this study they were not found to be causing any bias to the results.

The results of this study were compared to an earlier study using a different analytical technique, i.e. a submersible voltammetry system validated for measuring estuarine samples.² The comparison shows that results are very similar for arsenic, cadmium and lead. Results for copper and zinc deviated slightly from earlier results probably due to their higher affin-

Table 2. Added concentrations for calibration curves per element.

	Cu	Zn	As	Cd	Pb
Zero standard	0	0	0	0	0
Standard 1 ($\mu\text{g kg}^{-1}$)	0.5	1.3	1.0	0.02	0.01
Standard 2 ($\mu\text{g kg}^{-1}$)	1.0	2.6	2.0	0.04	0.02
Standard 3 ($\mu\text{g kg}^{-1}$)	1.5	3.9	3.0	0.06	0.03
Standard 4 ($\mu\text{g kg}^{-1}$)	2.0	5.2	4.0	0.08	0.04
Standard 5 ($\mu\text{g kg}^{-1}$)	2.5	6.5	5.0	0.10	0.05

Table 3. Results obtained for the measurement of CASS-6 CRM and 18 samples.

Element	Cu	Zn	As		Cd	Pb
Mode	SQ-KED	SQ-KED	SQ-KED	TQ-O ₂	SQ-KED	SQ-KED
Result CASS-6 CRM ($\mu\text{g kg}^{-1}$) ($N=4$)	0.57 ± 0.012	1.89 ± 0.23	1.04 ± 0.11	1.09 ± 0.08	0.027 ± 0.004	0.013 ± 0.002
Certified value ($\mu\text{g kg}^{-1}$)	0.530 ± 0.032	1.27 ± 0.18	1.04 ± 0.10		0.0217 ± 0.0018	0.0106 ± 0.0040
Concentration range in samples ($\mu\text{g kg}^{-1}$)	0.31–0.56	0.41–2.34	1.32–1.88		0.017–0.058	0.023–0.042

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ity for forming complexes with organic molecules present in sea water, which are not detected by *in situ* voltammetry.

Conclusion

The direct analysis of estuarine waters without prior dilution is possible using AGD on the iCAP TQ inductively coupled plasma mass spectrometer. The results obtained for the CASS-6 CRM indicate accurate and precise quantification is possible at very low concentration levels. The results obtained for the samples collected in the high salinity range of the Gironde Estuary show that historical metal contamination in the estuarine waters persists, although at lower levels (especially for elements such as Cd or Zn) as compared to earlier studies conducted in the 1990s.³ This observation fits with the continuous decrease of Cd concentrations determined in wild oysters from the Gironde Estuary mouth.⁴ Comparing the data to the results of an earlier study, it is clear that both ICP-MS and voltammetry methods provide similar results at trace and ultra-trace levels. Slight differ-

ences in results may occur due to metal species/complexes not detected in voltammetry. If submersible voltammetry systems allow for *in situ* measurements of a number of trace metals, ICP-MS is a time-efficient alternative and allows the analysis of a wider range of elements in one aspiration of the sample due to its inherent multi-elemental capability. Future work on multi-element analysis in seawater should include an even wider range of elements, including emerging metal contaminants.

Acknowledgements

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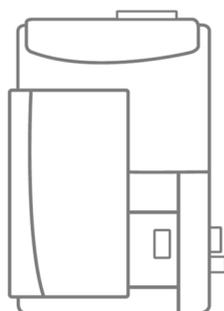
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Spectroscopic data supporting investment decisions

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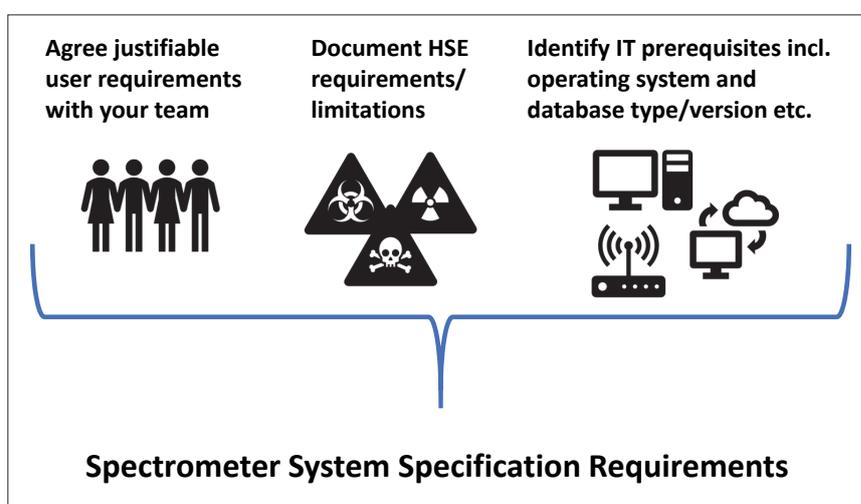
^bSERC, Sustainable Environment Research Centre, Faculty of Computing, Engineering and Science, University of South Wales, UK

So, you are lucky enough to have some capital equipment funds available for a badly needed new spectrometer. You are under pressure to decide how best to invest this hard-won bounty. More importantly, you need to make sure that the investment will fulfil all the expectations of your team. Your justification for a particular instrument must stand up to tough scrutiny by your managers. Cold, hard, factual, analytical data can remove any impression of personal bias in the selection process and make the job of defending any decision, even to friends in unsuccessful supplier companies, much easier.

Why do we need the new spectrometer?... No... Really WHY do we need it?

Fundamental to all spectrometer selection actions are the underlying user requirements and business case for the purchase. Without going into a long Fundamentals of Good Project Management sermon, it is good to get clear documentation of your needs. These can then be used as an impartial base for testing the various offerings in the market place. In fact, going through the process of arriving at these requirements is often extremely useful in firming up various colleagues' opinions (and challenging their prejudices). It sets clear expectations at the beginning of the selection process.

This is the time when you may also need to focus hard on the "business case" in terms of cash numbers such as: we will be more efficient as we can run 10-times as many samples per week using a fifth of



A few considerations BEFORE starting your search for your next spectrometer.

the expensive consumables. Quite often it is only these cash "return-of-investment" numbers which will interest senior decision makers. If they are not convincing, it will be hard to convince finance-oriented controllers just that a 10% increase in optical resolution is a sound case for investment!

If you are looking to replace an existing spectrometer, checking what the real workload has been, what methods have been run and what was the range of samples analysed will be a good source of performance specifications. A second benefit of this check is that it can easily provide you with a source of real-world test samples which you can use later when assessing the performance of your shortlist of spectrometer candidates.

If the initial justification is work overload rather than the impending failure of an outdated model, it is often revealing to check the instrument logs. It is possi-

ble that the spectrometer itself is not measuring 24/7, so a good challenge would be to see if investing in a better/larger autosampler would suffice.

These arguments may vary depending on whether your company or university has good access to capital expenditure, but is light on staff/student numbers to use the equipment. If so, increasing the sample throughput per man-hour of work makes less sense than focusing on the quality of the outputs, as you do not have the numbers to keep your new investment running at optimal capacity. On the other hand, where you have excellent numbers of good analysts currently queueing to feed the spectrometer, but are short of cash to buy instruments, perhaps the speed of the individual measurement will be a critical factor and maybe the use of autosamplers to keep it running 24/7 will be the appropriate solution.

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Having your requirements well documented at an early stage can also help to focus discussions later in the process, when some fancy add-on or additional software package is added to the considerations, but which was not part of the original arguments for the investment. This is not to say they should be ignored, but this does allow some impartial defence of excluding them from the assessment if required.

Computer control and deployed software

As you would expect from this column, the computing aspects of your requirements should be given particular attention from a number of different angles. Annoyingly, the dream spectrometer solution from the point of view of functionality may well come with an utterly hopeless user interface which will drastically reduce its efficient integration and operation in your team. It is also important to check your current computing specifications. If you have a well-run IT department, they should be able to supply you with a good roadmap of what and when compulsory changes to the environment are to be expected. If, for example, your chosen solution can only run with a specific database version and client operating system, this needs to be identified and checked against your internal IT deployment guidelines and roadmap. If you are working in a large organisation, especially in a heavily regulated industry, this is a must.

Again, it is important to think of your analysts and their interaction with the spectrometer software. Having a lab full

of similar instrument types from a range of different vendors does mean, unfortunately, that it is hard to cover for staff holidays, absences for other reasons or when a particular spectrometer is out of commission for some reason. It is not great when the entire workload falls on a system which only half the staff can operate efficiently. This situation was worse in the past when vendors picked different operating systems from each other and even from instrument to instrument—maybe for good commercial reasons—but which made laboratory management a nightmare. Fortunately, this happens less now, but is still something to keep in mind. This is also a great area to get input from your team, as they may well have tales to tell about particular issues or instabilities they or their network of friends have found with particular solutions.

Finally, if you intend to purchase a spectrometer solution which is running a hyphenated method such as UHPLC-MS/MS with an autosampler, it is critical to see how the various components interact with one another. In some solutions, vendors fail to fully integrate the software to control the various components with each other. Maybe the autosampler runs as the main experiment control component and the spectrometer only receives a “Go” signal from the autosampler when a sample injection takes place. This can mean double the work for your analysts—programming the autosampler separately from the spectrometer and often having to use two

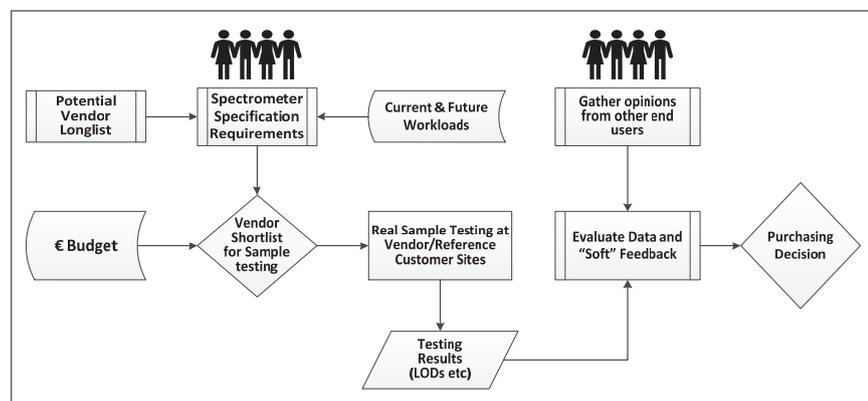
separate interfaces. This can also be a potential source of serious errors if, for example, the sample lists are held separately from one another and not shared by both software packages. This sort of configuration can also make carrying out Design-of-Experiments based new method optimisation work a complete nightmare.

Required analytical figures of merit and your own “reference” samples

One mistake often made in looking at the choice of analytical instruments to invest in is to rely on measurements of reference sample mixtures measured by the vendor. Quite often, depending on the technique under investigation, reference mixtures of pure chemicals are commonly used to measure and check instrument performance in the laboratory. These might be a starting point, for example in checking that hyphenated chromatographic/spectroscopic instruments in a particular configuration is performing correctly. However, they should only be used as a starting point.

It is far more important to use the information from your current workload discussed above to identify “typical samples” from your own laboratory and standard methods that your team apply. Make good use of your team’s experience of how these samples behave and what performance to expect from them. Your standard matrix may contain high concentrations of salt, for example, so comparing measurements carried out on samples made up only in ultra-pure water may mask issues a candidate spectrometer has with high-salt content samples.

These test samples should be identified and enough material available for you to carry out your own round-robin amongst the vendors’ instruments that you are short-listing. In the past, really good data have been supplied by a manufacturer for industry-standard reference materials, but local requirements for the spectrometer were not to carry out sample quantification studies on the major components, but rather to look for and quantify very low level toxic contaminants in a complex and changing matrix



High level input decision-making criteria for your investment decision.

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where only tiny amount of sample were available. So, the performance of the complete analytical system right down at the limits of detection were critical. In one case, a vendor chose to collect scattered photons from a sample surface at a smaller solid angle than the competitor's designs. This meant that although the main spectrometer itself was superb, it couldn't match the Limit of Quantitation figures of the competitors—a key knock-out criterion for the selection.

Operational requirements and assessing the vendors

Armed with clear requirements, a range of typical, different and challenging reference test samples and, of course, some idea of your budget, you are now well prepared to venture outside your organisation and contact the various vendors in the market place. In some countries this may actually mean having to go through an "independent" systems integrator rather than the vendor themselves. This may be important when you come to assess the availability of future support for the instrument you intend to buy. This construct may not necessarily be a bad thing, experts who speak your own language and who are based more locally may well provide better support than thinly spread vendor engineers who are travelling from country to country every day. Local support may well be much cheaper if your maintenance contract requires you to pick up the cost of travel for a service visit.

Some spectrometers also contain, for example, radioactive sources and here it is important to identify your in-house rules/regulatory requirements around the movement of these sources. If your instrument needs to be returned to the vendor for maintenance or repair, how much additional trouble/expense will there be in the future, especially if that journey requires the instrument to cross national borders.

Having used your requirements to come to a shortlist of instruments and vendors on paper (remember any one vendor may actually have several models which can meet your needs), it is very desirable to actually see the

spectrometers in action and your vendors—if they are seriously interested in selling you a solution—will only be too happy to oblige. This might be at their own sites or with good existing customers with whom they have an agreed relationship. Be very wary of a vendor with no demonstration facilities close enough for you to visit where you can run your samples and who also doesn't have any reference customers in your area. For some instrument types, it is perfectly reasonable for the vendor to request your samples in advance of a visit, especially if you have chosen challenging samples of a type they have not seen before, as they will probably need to carry out the same method optimisation work in advance of the required measurements as you would do yourself. Saying that, a vendor delivered a very impressive performance with some samples one of our teams had been struggling to analyse consistently for several months, only to have the vendor's engineer solve the problem in about 30 minutes! Definitely a lesson learnt!

Always ask for a reference customer list with named individuals who a vendor believes will be happy to talk to you. These may be too far from your location to visit in person, but a quick telephone call to gather their longer-term experiences with a particular vendor or solution. You may get excellent insights into the different options on the market place that you can never gain when running a few test samples over a few days. These contacts are a good source of confirmatory evidence on such topics as long-term cost of ownership, around replacing parts that wear out or solvent usage. Since the contact introduction comes from the vendor and you expect them to be positive, a good question can be "what do you wish you had known or had asked about before buying"?

Test the most important requirements on your own samples in a demo on the vendor's instrument and compare the results of different vendors very closely. Do not rely on results only generated by the vendor of the instrument on standard reference mixtures.

Conclusions: the future

Having run all your reference samples on the various short-listed spectrometers, gathered all the feedback from other customers with the same solutions and, very importantly, listened hard to the thoughts of your own analysts, you also need to look beyond the current time point and into the future.

We have had issues in the past with the instrument selection process opting for a clearly superior product from a particular vendor, only for them to pull out of the market, de-listing the product we purchased within months of us installing it. An extremely annoying position to find yourself in and one which can be hard to foresee. You should be open with the vendor about their plans with this spectrometer type or field of analysis. The person you are talking to may themselves be unaware of thought processes higher up in their organisation, especially if decision-making control is from a headquarters located on another continent, but it is worth probing. Do they have a signed-off release plan, for example, for newer versions of the control software? Most major vendors will have this information and if you are under a non-disclosure agreement with them—which I would strongly recommend as they have been measuring your samples—they should be free to let you know. If they show you a good development roadmap, ask about their history of keeping to this roadmap in the past. This is a good indicator of the commitment of the company as a whole to this technology area.

Finally, it is in your interests and those of your vendor community to keep the results of your assessment and the data that drove the final decision confidential. Even for vendors you do not choose to purchase from, it is respectful to give honest and open feedback on your decision criteria as you will want them to be open and honest with you when the next purchase comes around and supporting you during your decision-making journey will have cost them money. Remember the feedback you provide can potentially help them improve their product offering which in the long-run can only help us all!

A look at the reference material industry

Peter J. Jenks B.Sc, FRSC

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How do analysts and quality managers source and use the reference materials, certified reference materials, pharmaceutical and clinical reference substances and proficiency testing services they need to meet the challenges of working in an ISO/IEC 17025 accredited laboratory?

This is question I first asked in 2001, when I had just started The Jenks Partnership. At that time the testing world was coming to terms with the release of ISO/IEC 17025 in 1999. Initial traction was slow, but with the release of the first revision in 2005 the world changed. From that point the production and supply of RMs started to change from being predominantly an activity of National Metrology Laboratories to a straightforward commercial business, regulated by a combination of ISO/IEC 17025 and ISO Guide 34.

We published our findings in the long defunct *Reference Material Report* newsletter, published by IM Publications and which I edited. The aim of the newsletter was to provide users and producers of RMs with industry background and news to help them better meet the requirements of the growing Quality Management world. You can read that article in the online version of this column.

In the 18, almost 19 years, that have passed, it seems I've become deeply embedded within the RM producer and user community. All through the years I've been a regular contributor to *Spectroscopy Europe*, observing and commenting on the ever-evolving world of chemical metrology: this is my 66th column!

Much has changed since 2001, back then in the RM world it was clear that laboratory accreditation and the supply of certified and other RMs was but a small part of the overall lab supply business.

In early 1999 I returned from Germany to work for LGC, my role was to put together a plan that would convert the "Office of Reference Materials", partly funded by the UK Government, into the LGC Standards business. In 2000 LGC acquired Promochem GmbH and its network of suppliers and distributors. Since then LGC has grown its standards business so that it now forms a major part of LGC's activities. After privatisation, LGC has been owned by a series of Private Equity investors, the most recent, KKR Inc. has put LGC on the market and it is attracting a very considerable interest.

Why should so many be interested in what may seem to be an obscure corner of the life science sector? Because over the last 20 years the RM business has grown, massively. Some estimates put the global market for all types of RMs, across all sectors, as close to \$2billion. That is a big business and it is getting bigger. The growth rate is hard to pin down, it varies considerably across the many sectors, but overall is probably 7% a year. Add to that the very high levels of profitability and it is very attractive for investors looking for a safe place in and increasingly challenging world. The growth is due a couple of factors: first the accreditation of testing laboratories to ISO/IEC 17025, and for clinical laboratories to the related ISO/IEC 15189 standard and until every possible lab has been accredited the growth will continue.

The second driver is the increasing concerns of society about the pollution of food, water and air and the consequential regulation by Governments: this increases the amount of testing, which increases the demand for RMs and PT.

So, in the light of so much change, has the way RMs are used changed?

We concluded it would be worthwhile to re-run the survey. This time it is web-based, a simple "tick box" process. This link (<https://www.spectroscopyasia.com/reference-material-survey-2019>) will take you to the survey, which will run until the end of January 2020.

It is just five pages and should take no more than 10 minutes of your time to complete.

Once closed, we will analyse the responses and compare with the results from 2001. The results we will publish in *Spectroscopy Europe* as an extended Quality Matters column, later in 2020.

In the rich, vibrant market that the RM market has become, one might expect to see new entrants, investors seeking to build a new Promochem or LGC. But although there are new entrants, mostly exploring new niches such as RMs for next generation molecular biology analytical techniques, we do not see any major developments: why?

The simple answer is that starting a CRM producer from scratch is a long, costly and expensive process. The main challenge is achieving Accreditation, both ISO/IEC 17025 and ISO/IEC 17034 are essential and if PT is required, ISO/IEC 17043 is also required. All the businesses that make up the RM market were set up in the 1970s and 80s, many have been acquired and are part of the LGC, Merck, Agilent, Thermo Fisher and Waters businesses. The question has to be "Is this a positive development"?

The answers from the questionnaire, and in particular Sections 3 and 5, will help show if the level of service, of support and advice available has improved, or worsened, as a result of the changes over time.

Revisiting the Replication Experiment

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Here starts a second round of Sampling Columns, which have been a fixture in almost every *Spectroscopy Europe* issue since its inauguration in 2014. The first series, which concluded in the last issue, provides a stand-alone collection for easy, free access to a first curriculum of the Theory and Practice of Sampling. The second series of Sampling Columns will focus on sampling in practice, special issues and features (left out of the first series), educational gems and other bits-and-bobs. The last two items comprise a mixture of topics and issues that also will illustrate and educate readers, but specifically only after a first minimum of TOS competence has been acquired.

Introduction

The Replication Experiment (RE) was introduced and applied to different sampling contexts in an earlier column.¹ Here we want to show its features and usefulness in the context of evaluating a possible new sampling + analytical approach for raw material characterisation in a demanding industrial context: "Representative sampling and use of handheld X-ray fluorescence (HHXRF) to characterise lot and sample quality of quartzite in a pyro-metallurgical ferrosilicon plant".² The issue has a very sharp focus: Is the HHXRF approach applied to field

samples able to quantify very troublesome, minute amounts of pollutant trace compounds in quartzite for the ferrosilicon process with the necessary accuracy and precision? We here focus on the application of the RE only in the context of the full evaluation, a much broader study.³

Industrial setting (Elkem Metal Canada ferrosilicon plant)

Elkem Metal Canada Inc. is a producer of ferrosilicon alloy. Production relies on two main pathways characterised, respectively, by a base of 50% or 75%

silicon, with several customer-specified options, e.g. foundry and steel plants. For this type of production, the plant reduces quartz (SiO_2) by a combination of melting in a blast furnace and Söderberg electrolysis. For this process to be successful, the composition of quartz (in the form of the raw material quartzite) must be carefully monitored and controlled. Contaminants, such as aluminium, can dilute the silica phase. Although quartz would appear to be a simple matrix, the low concentrations of impurities, for example, in the form of discrete minute grains of ilmenite (FeTiO_3) or magnetite (Fe_3O_4) increase



A big job awaiting MSc student Danny Desroches; photo taken just before it all began in earnest in front of a newly arrived raw material lot.



Close-up of the typical dimensional manifestation of quartzite blocks (safety glove for scale). Block surface flatness, which influences the quality of HHXRF analysis, is commented upon further in the text.

SAMPLING COLUMN

the difficulty of accurate analysis. These impurities exhibit an extreme degree of spatial heterogeneity in the raw quartzite lot and thus make proper sampling before, or associated with, analysis highly challenging. As the matrix is essentially SiO_2 (>98 % m/m), Elkem Metal does not analyse individual whole *shipments* of quartz but instead relies on traditional *grab samples* over contractually specified volume/time periods. This protocol is a major weakness for proper QC/QA, which was one of the main reasons for the present industry-academe collaborative studies.^{2,4} Can HHXRF be deployed in the field directly at the incoming shipments (truckloads) of run-of-the-mine quartzite (Figure 1)?

Bearing in mind the fundamental relationship: Global Estimation Error (GEE) = TSE + TAE. There are two aspects of evaluating such a possible alternative approach, Total Sampling Error (TSE) and Total Analytical Error (TAE).

Previous TAE evaluation of HHXRF⁴

Chemical analysis in environmental and mineral exploration is increasingly carried out with the aid of portable instruments such as HHXRF. However, use of HHXRF in an industrial context such as quality control for mineral transformation or processing has been rarely documented. Desroches *et al.* tested such an instrument on quartz (quartzite) as it is a chemically simple mineral (SiO_2) where critical analytical performance indicators (detection limits, precision and accuracy, instrumental drift etc.) are easily delineated. The TAE has been evaluated on trace elements (contaminants) in quartz. The limit of detection of the quartzite matrix was lower than $70 \mu\text{g g}^{-1}$ for TiO_2 , Fe_2O_3 and CaO on matrix-matched reference materials pressed pellets.⁴ TiO_2 , Fe_2O_3 and CaO gave results similar to certified values, while low concentration light elements such as Al_2O_3 and MgO gave less accurate results. *In situ* determinations using HHXRF, although faster, are restricted by the quality of the analysed surface, and even small mineral inclusions can cause seriously erratic results. This is typically coun-

teracted by increasing the number of measurements, for example to five (on a single 10-cm-sided quartzite block). Considering the diminished workload of using a HHXRF in this industrial context, the analytical evaluation was interpreted as “fit-for-purpose”.² Here we want to continue this evaluation by including all the preceding sampling, sub-sampling and other sample preparation steps before analysis.

Experimental design

The study employed an elaborate experimental design complying with the fact that sampling errors and analytical errors, “lot-to-analysis”, are related in a logical chain-wise fashion: Primary Sampling Errors (PSE) → Sample Preparation Errors (SPE) → Total Analytical Errors (TAE). In the studies, particular care was taken to include the many labour-intensive *sample preparation steps* involved in the current “primary-sample-transportation-to-laboratory-for-extensive-preparation-and-analysis” pathway in order that comprehensive, valid comparisons could be made with the new HHXRF alterna-

tive. What can be gained if the current laborious approach can be scrapped?

It is especially interesting to know *where* in the pathway to find the most dominating sampling/preparation errors in a *no-nonsense* industrial context: “Improvements in final analytical accuracy and precision—at what practical and economical costs?” There is always a stern cost/benefit filter for comprehensive academic studies in industry. No gain from a relative minor total Measurement Uncertainty (MU) benefit if the new (or extra) workload goes in the opposite direction! Striking an optimal balance here is critical, for which reason the project team was comprised by i) the analytical chemistry student (the most important workforce for the study), university professors galore (of both analytical geochemistry and sampling expert types) and the backstop industrial representative (the ferrosilicon process expert, *no less*). With this top-heavy supervisory group, the student was eventually let loose on the rather complex study, in which several objec-



Figure 1. Overview of the experimental design (note plastic bags each containing 10 kg, see text). Student and supervisors hard at work on a typical industrial lot of raw material, quartzite. Note that all inspection and sampling takes place on the lot surface, following years of experience with the overall heterogeneity of truckload lots transported from the mine. Photo courtesy of Dany Desroches.

SAMPLING COLUMN

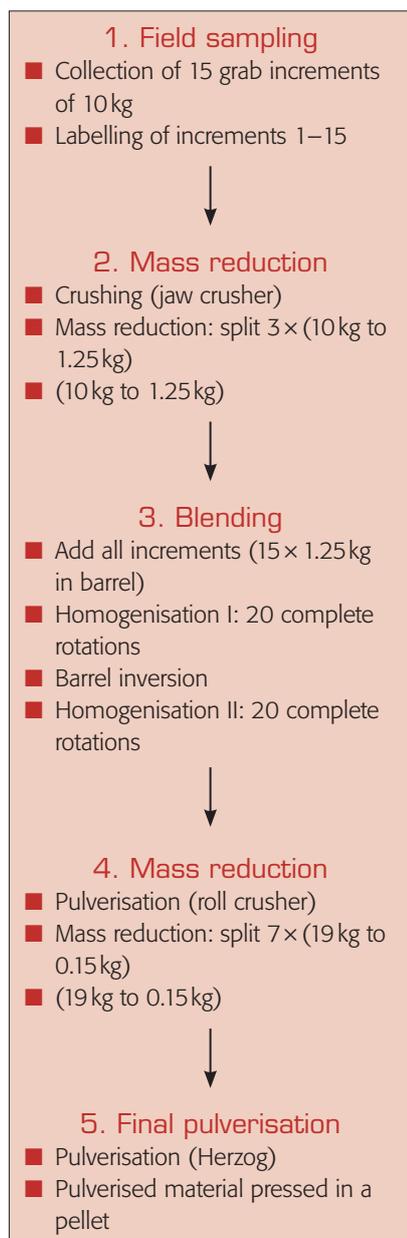


Figure 2. Simplified illustration of the compound experimental design used; see Reference 2 for the full design.

tives were to be integrated (estimation of TSE, optimisation of “practical field same mass”, variographic characterisation of lot surface transects, comparison of grab vs composition sampling approaches).^{2–4}

We here outline only RE-based decoupling of *all* pathway error contributions (a first for the plant, as concerns the full sampling-preparation stages). Several new insights followed ...

The most important critical success factor was quickly identified in the full lot-to-analysis pathway, by performing a hierarchical RE. Figure 2 is a re-drawn, simplified version of the resulting schematics.

Replication experiment

The variation (the spread) between samples that have been sampled by the exact same procedures (including all sub-sampling and preparation procedures in the laboratory) can be quantified by fitting a standard normal distribution. This will be a reflection of the inherent heterogeneity of the lot material, information that was of particular interest to the present industrial plant. The precise number of replicates needs to be defined after careful consideration.⁵

Hierarchical Replication Experiment

REs can quantify the effects of variation in *total* sampling, processing and analytical error. RE can also determine whether the alternative HHXRF procedure can be declared fit-for-purpose when also here applied “from the top”, i.e. when replication starts with the primary sampling. When applied hierarchically RE can precisely identify those pathway stages that contribute most to the overall uncertainty. RE can be applied to any new, or to any existing sampling procedure, where RE must *always* begin at the primary sampling stage to ensure that *all* sampling and/or preparation errors are included in the estimate of the total MU. The evaluation of each step also requires a minimum of ten replicate sampling operations.

With five identified stages in the present ferrosilicon production process, this translated into fifty analytical samples. Beginning with ten primary samples (PSE), the RE experiment then prepared, in an identical manner, ten secondary sub-samples from one of the primary samples, selected at random. Further repetitive application of this protocol, ten next stage sub-samples from a randomly selected sample were prepared, cascading hierarchically down through all sample preparation steps,

ending with the strict analytical variance TAE. It is imperative that all sets of ten replicates samples, from whatever stage are subsequently subjected to identical downstream processing in the laboratory, thus producing comparable analytical results pertaining to each stage where the hierarchical RE was initiated, Figure 3.^a

To identify the stage(s) that most contribute to the GEE, it is only necessary to compare the empirically estimated variances from all stages (as summarised to 100%) based on the identified sampling stages: PSE (field sampling), primary crushing error (jaw crusher), secondary crushing error (roll crusher), pulverisation/homogenisation error ..., whereas the TAE is known in minute detail from the previous TAE characterisation.⁴ Side-stepping the many detailed particulars in the way the full study was carried out, Figure 4 shows the final quantitative results of the hierarchical RE.

Decoupling of the empirical error hierarchy

Figure 4 then shows the key information to be gained by the hierarchical RE. For the current laboratory approach, the primary sampling contributes 35%, jaw crushing 25%, roll crushing 25% of the overall variance, while pulverisation and analysis strictly account for only 5% and 2.5%.

Based on other experiences, the latter two, especially, have been somewhat *overestimated* before the present study, while the sampling and the two sample preparation/mass-reduction steps functioned as alarm bells from which to build important new experience; much was learned for future process optimisation.^{2–4}

^aN.B. This approach is different from a conventional Design of Experiment approach with the intention of variance decomposition by ANOVA. One is in no way able to control all factors involved in the present kind of practical design, which instead follows the lot-to-aliquot pathway in a straight, realistic fashion.

SAMPLING COLUMN

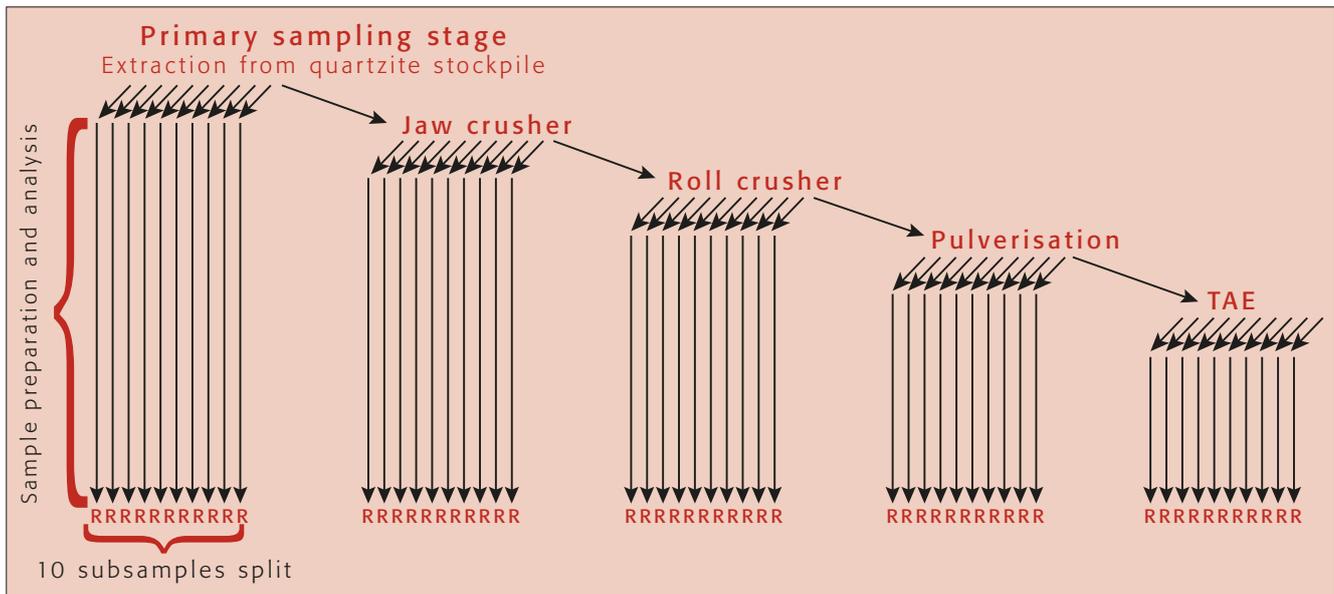


Figure 3. Hierarchical RE for the ferrosilicon plant assessment of traditional laboratory method vs the new HHXRF. At all stages, a sample is randomly selected from which is split off ten sub-samples with the current protocols.

Figures 3 and 4 shows a perfunctory *decoupling* of the individual error sources in the traditional sampling + analysis approach, typical of very many process industrial cases.

Which can now be compared with the much less laborious, direct HHXRF approach as applied directly to individual

truckloads carrying run-of-the-mill quartzite (or to stocks piled up from serial truck off-loads). For the interested reader, the *specific* conclusions as to the fate of HHXRF in the specific plant contexts were:

Although typical grab sampling followed by laboratory XRF instrumentation provide useful results (today's

status quo), composite sampling coupled with in situ analysis provide more accurate results. The portability and speed of analysis of HHXRF give the opportunity to carry out many individual determinations over a specified lot surface within, say, an hour compared to the much longer current procedure. Considering the larger volume that can be sampled better, more controlled results are obtained from the field. The new HHXRF protocol has some limitations, however, such as lower intrinsic sensitivity compared to laboratory equipment,^{3,4} and analytical results are influenced even by very small mineral inclusions that appear randomly under the X-ray beam of the portable instrument. Lighter elements are strongly influenced by deviations from a suitable flatness of the analysed surface relative to the X-ray beam. These issues are to some degree compensated for by a higher number of HHXRF measurements which are done in a considerably shorter time span. Here the evergreen time vs money vs analytical efficiency balance is exposed in full. Based on these first empirical results, an HHXRF-based approach would appear possible and can likely become well substantiated. However, quantification of the specific lot heterogeneities on a regular basis must be considered as an

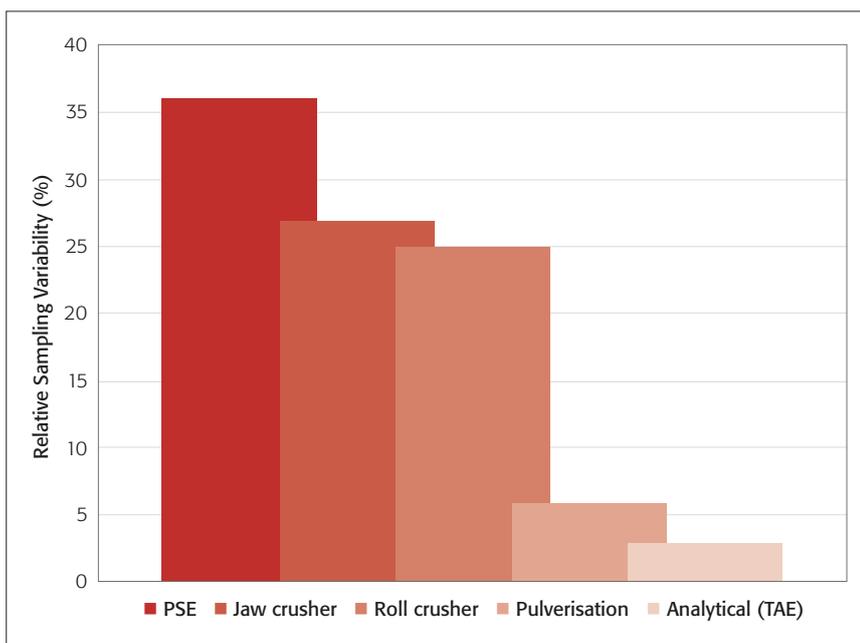


Figure 4. Numerical results of the hierarchical RE, with which can be identified error contributions from each step (primary sampling to analysis). Vertical scale is RSV; %, standard deviation/average of replicated results $\times 100$ for Fe_2O_3 .

SAMPLING COLUMN

necessary quality assurance—which is a task that very much can be best facilitated using HHXRF.

Conclusions

The usefulness of hierarchical RE characterisation has been shown in a demanding industrial context, helping to identify the weak(est) links in the current “lot-to-analysis” pathway. Quantitative results indicate that the HHXRF approach may be able to obtain a status as “fit-for-purpose”. The hierarchical RE reigns supreme.

Acknowledgements

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industrial setting. Also, Elkem Chicoutime for permission to summarise from the original publication (Reference 2).

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Paul Bédard obtained his PhD from Université du Québec à Chicoutimi followed by a postdoctoral fellowship at Université de Montréal. Subsequently, he did a few years of consulting, then became laboratory manager at LabMaTer (Université du Québec à Chicoutimi). He obtained a tenured professor position at Université du Québec à Chicoutimi in 2011. His main experience is in geochemistry of igneous rocks. He worked on Archean granites, platinum group element analysis, HHXRF in industrial context and more recently on niobium and rare earth elements deposits in carbonatites.



Kim H. Esbensen, PhD, Dr (hon), has been research professor in Geoscience Data Analysis and Sampling at GEUS, the National Geological Surveys of Denmark and Greenland (2010–2015), chemometrics & sampling professor at Aalborg University, Denmark (2001–2015), professor (Process Analytical Technologies) at Telemark Institute of Technology, Norway (1990–2000 and 2010–2015) and professeur associé, Université du Québec à Chicoutimi (2013–2016). From 2015 he phased out a more than 30-year academic career for a quest as an independent consultant from 2015: www.kheconsult.com. But as he could not terminate his love for teaching, he is still very active as an international visiting, guest and affiliate professor.

A geologist/geochemist/data analyst of training, he has been working 20+ years in the forefront of chemometrics, but since 2000 he has devoted most of his scientific R&D to the theme of representative sampling of heterogeneous materials, processes and systems (Theory of Sampling, TOS), PAT (Process Analytical Technology) and chemometrics. He is a member of several scientific societies and has published over 250 peer-reviewed papers and is the author of a widely used textbook in *Multivariate Data Analysis* (35,000 copies), which was published in its 6th edition in 2018. He was chairman of the taskforce behind the world's first horizontal (matrix-independent) sampling standard DS 3077 (2013). He is editor of the science magazine *TOS forum* (<https://www.impopen.com/tos-forum>) and for the Sampling Column in *Spectroscopy Europe/Asia*.

Esbensen is fond of the right kind of friends and dogs, swinging jazz, fine cuisine, good wine, contemporary art and classical music. His has been collecting science fiction novels for more decades than what he is comfortable contemplating, still, as ever, it's all in the future ...

NEW PRODUCTS

ATOMIC

New handheld LIBS analyser

The lightweight Thermo Scientific Niton Apollo handheld LIBS analyser enables users in the field to test metals for carbon content in environments where previous technologies were too cumbersome. Results can be returned in as few as 10s and the portability of the Niton Apollo is especially useful for operators at complex jobsites who have historically manoeuvred large equipment into small or difficult spaces to perform analysis.

In addition to quantifying carbon concentrations in low alloys and L+H grade steels, the Niton Apollo also more accurately measures Al, Cr, Cu, Fe, Mn, Mo, Ni, Si, Ti, V, W, Carbon Equivalency (CE) and pseudo elements. Additional features and benefits of the Niton Apollo include: third-party-validated interlocks designed to keep users and bystanders safe from laser exposure; a tapered nose to attain more field coverage of awkward corners, joints and tight welds; micro and macro cameras to support sample positioning and record keeping documentation; wireless data transfer, remote operation and software updates enabled by NitonConnect; IP54 rating to safeguard against environments containing splashes or dust; two hot-swappable Milwaukee® batteries with a battery life of 3–4h each; tilting, colour touchscreen to allow viewing from multiple angles.

Thermo Fisher Scientific

► <http://link.spectroscopyasia.com/31-096>



IMAGING

Hyperspectral 20 Mpx video camera

Cubert has introduced the ULTRIS hyperspectral light field camera. During image acquisition the object is recorded with a multitude of images, each with its own optical bandpass filter with different centre wavelength. This is made possible by combining a continuously variable bandpass filter with a lenslet array. The camera features an Ultra-HD CMOS sensor with 20 Mpx. It has a native resolution of 400 × 400 px, resulting in 160,000 spectra, each with 100 spectral bands covering 450–850 nm, taken in a single snapshot.

Cubert

► <http://link.spectroscopyasia.com/31-107>



Nanoscale IR spectroscopy and chemical imaging SNOM/AFM microscopy system

Bruker has released the nanoIR3-s Broadband™, a nanoscale FT-IR spectroscopy system that combines the nanoIR3-s s-SNOM (scattering Scanning Near-field Optical Microscopy) based platform with femtosecond IR laser technology. This combination had applications in advanced polymeric materials, life science applications and in nanoscale optical imaging of 2D materials, including plasmonic fields and nanophotonic structures.

The nanoIR3-s Broadband system covers the entire mid-infrared spectral range (2.5–15 μm/4000–670 cm⁻¹) by coupling



NEW PRODUCTS

with a broadband light source based on a femtosecond OPO/DFG laser. While featuring high laser power and wide spectral

range, this laser source can also switch its linewidth for imaging and spectroscopy.

Bruker

► <http://link.spectroscopyasia.com/31-104>

INFRARED

New INVENIO X FT-IR research spectrometer

The INVENIO platform was introduced in 2018 and offers MultiTect™ technology for mounting up to five detectors, as well as FM functionality for simultaneous mid- and far-infrared spectroscopy. The new INVENIO X is available with an automated three-position beam splitter changer, and its wear-free, novel INTEGRAL™ interferometer combines high spectral resolution of $<0.09\text{ cm}^{-1}$ with the accuracy of cube corner mirrors. In addition, INVENIO X benefits from other features of the INVENIO platform: its novel beam path improves spectroscopic sensitivity, and system-on-a-chip (SoC) built-in intelligence ensures faster data acquisition and processing. Its optional Transit™ channel enables quick measurements without removing longer-term experiments from the main sample compartment. Coded beam windows with magnetic mounts, an automated internal attenuator, an eight-position validation wheel and an optional integrated touch panel.

Bruker

► <http://link.spectroscopyasia.com/31-105>



MASS SPEC

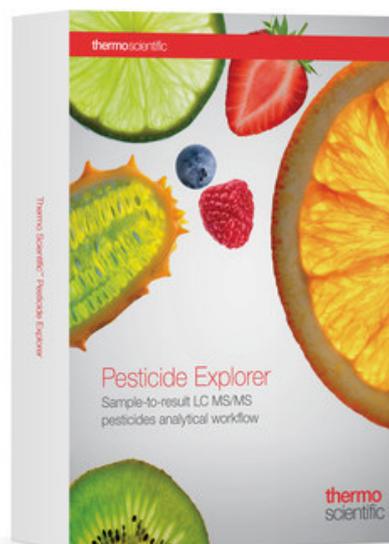
Workflow for routine determination of pesticides in food

A new workflow from Thermo Fisher Scientific provides a validated analytical method combining HPLC and triple quadrupole MS for the reliable and sensitive quantitation of pesticides in complex sample matrices. This is designed to help food testing laboratories more efficiently comply with global regulatory requirements. The Thermo Scientific Pesticide Explorer workflow brings together the sample preparation, hardware and software capabilities required by food testing laboratories for the targeted analysis of multi-class pesticides. It can be easily configured to meet the needs of individual laboratories.

The workflow consists of the Thermo Scientific Vanquish Flex Binary UHPLC system with the Thermo Scientific TSQ Quantis triple quadrupole mass spectrometer, verified consumables and analytical software, including Thermo Scientific TraceFinder and Thermo Scientific Chromeleon chromatography data system software.

Thermo Fisher Scientific

► <http://link.spectroscopyasia.com/31-100>



NEAR INFRARED

Lightweight handheld NIR spectrometer

Si-Ware Systems is introducing a handheld material analysis scanner with “plug-and-play development” capability for rapid deployment in the field or on the factory floor. The NeoSpectra-Scanner is built around Si-Ware’s NeoSpectra spectral sensor technology, with a spectral range of 1350–2500 nm. The NeoSpectra-Scanner has a five-step application development process that requires the independent characterisation of materials, measurement of the materials with the scanner and developing analysis models that correlate the two. The reseller or user develops the custom user application and display for smartphone, mobile device or laptop. The plug-and-play development package includes the scanner, reference material, data collection software and a software development kit.

The instrument has a large spot size (up to 10 mm) for measuring non-homogenous materials such as grains and soils. It is also ruggedised for in-field use, conforming to IP65 protection standards. The Scanner is designed not only for point-and-shoot capability but also to rest on flat surfaces for above-scanner sampling, or for below-scanner sampling from a suspended position. It works with any Bluetooth-enabled device to transfer



information. The Neospectra-Scanner runs on two 18650 batteries and when fully charged can perform more than 1000 scans.

NeoSpectra

► <http://link.spectroscopyasia.com/31-101>

NMR

Broadband multinuclear benchtop NMR spectrometer

Oxford Instruments has launched X-Pulse, a high-resolution 60 MHz benchtop NMR system with true multinuclear capability. The system can be tuned to any nucleus from ^{29}Si to ^{31}P easily, without having to change NMR probes. A unique flow cell and variable temperature probe allow dynamic chemical reactions to be continuously monitored, with the variable temperature capability from 20 °C to 70 °C. New shimming technology delivers lineshapes of less than 0.35 Hz/10 Hz as standard, making it easier to separate overlapping peaks and identify smaller concentrations of compounds. A traditional magnet design with high thermal mass makes X-Pulse insensitive to sample temperature variations, whether static or flowing, so that sample temperature artefacts are eliminated.

Oxford Instruments

► <http://link.spectroscopyasia.com/31-106>

Bruker introduces 80 MHz benchtop FT-NMR system

Bruker has introduced the Fourier 80 system, an 80 MHz Fourier Transform nuclear magnetic resonance (FT-NMR) benchtop spectrometer. The Fourier 80 has been designed for organic or medicinal chemistry research, routine analysis, teaching or synthesis verification in any chemistry laboratory. Equipped with a novel, ultra-stable 80 MHz permanent magnet, it requires no cryogenics, magnet power supply, water cooling or special labora-



tory infrastructure. The Fourier 80 can be installed on a bench or in a fume hood without the need for new infrastructure.



NEW PRODUCTS

The Fourier 80 has been designed for high data quality and stability at 80 MHz, with excellent lineshape, resolution and sensitivity in 80 MHz homonuclear ^1H or heteronuclear $^1\text{H}/^{13}\text{C}$ FT-NMR experiments. It runs Bruker's TopSpin™ software, giving users access to the extensive TopSpin library of 1D and 2D homonuclear and proton-carbon heteronuclear experiments and pulse programs. In addition, the Fourier 80 offers the new, easy-to-use GoScan™ software module for NMR novice users.

SOFTWARE

Software updates to ACD/Labs' Spectrus

ACD/Labs has introduced updates for their Spectrus software platform, including an expansion of instrument vendor data format support. Several enhancements have been introduced across the analytical techniques and workflows supported by Spectrus applications. For NMR, "targeted profiling" has been introduced offering streamlined analysis of 1D mixture spectra. The speed and accuracy of ACD/Labs' NMR Predictors have also been enhanced, along with improvements to data processing tools. For LC/MS, v2019 software offers greater flexibility in the

The Fourier 80 offers workflows and protocols for academic, pharma and industrial chemistry research, forensics and organic synthesis control. It can be incorporated into chemistry education to introduce students to FT-NMR. An optional teaching package with recommended experiments and spectra interpretation guidance is available.

Bruker

▶ <http://link.spectroscopyasia.com/31-097>

analysis and review of MS^n data, more efficient spectral searching for dereplication workflows and more intuitive management of LC/UV/MS information.

The software architecture of ACD/Labs' entire line of software applications has been updated to 64-bit, eliminating any technological limitations scientists may face such as the import/export of large LC/MS datasets.

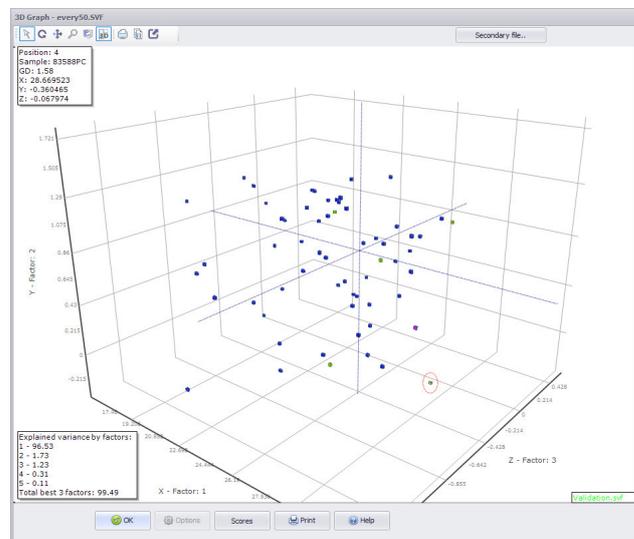
ACD/Labs

▶ <http://link.spectroscopyasia.com/31-103>

NIR chemometric and database management software

Unity Scientific has announced the release of UCal 4™, a complete chemometric toolbox for the creation, optimisation and validation of NIR calibrations. UCal 4 includes five chemometric algorithms and a built-in independent validation routine for rapid determination of the best classification parameters. The resulting conformity models can be configured in parallel with quantitative models for simultaneous good product and compositional analysis from Unity SpectraStar analysers. For standard quantitative calibrations, UCal 4 improves calibration accuracy and precision with new regression options. Variance scaled PLS is now available for improved performance and more robust predictions from most applications. A new locally weighted regression algorithm delivers accurate predictions from large, diverse or non-linear calibration sets. Calibration managers will also appreciate the new batch processing system which allows for an automated iterative evaluation of standard calibration parameters for a rapid and thorough optimisation of data sets.

Enhanced graphical displays and an easy-to-use interface make it easy to interpret complex data and statistical relationships for novices and experts alike. The new Project Manager tool provides rapid access and simplified file management across multiple project and applications, while the new population structuring routines identify important samples for calibration, improving robustness and saving reference laboratory costs. Deploying new calibrations is simplified with the newly-released CommUnity™ Networking Suite from Unity Scientific. Simply load the calibration in CommUnity and publish it to your network. Complete product configurations will be delivered from the cloud, and in seconds, the instrument is ready for operation.



Alternatively, calibrations can be manually uploaded to individual SpectraStar analysers.

Unity Scientific

▶ <http://link.spectroscopyasia.com/31-102>

Conferences

2019

3–7 November, Toronto, Canada. **SETAC North America 40th Annual Meeting**. ✉ <https://toronto.setac.org/>

5–8 November, Prague, Czech Republic. **9th International Symposium on Recent Advances in Food Analysis (RAFA 2019)**. ✉ jana.hajslova@vscht.cz, ✉ <http://www.rafa2019.eu/>

1–6 December, Boston, United States. **Materials Research Society 2019 Fall Meeting (MRS 2019)**. ✉ <https://www.mrs.org/fall2019>

9–13 December, San Francisco, United States. **2019 American Geophysical Union (AGU) Fall Meeting**. ✉ meeting-info@agu.org, ✉ <https://www.agu.org/Plan-for-a-Meeting/AGUMeetings>

2020

12–18 January, Tucson, Arizona, United States. **2020 Winter Conference on Plasma Spectrochemistry**. Ramon Barnes, ✉ wc2020@chem.umass.edu, ✉ <http://icpinformation.org>

27–29 January, Liege, Belgium. **Chemometrics 2020 Conference**. ✉ chemom2020@sciencesconf.org, ✉ <https://chemom2020.sciencesconf.org/>

29–31 January, Ghent, Belgium. **16th International Symposium on Hyphenated Techniques in Chromatography and Separation technology**. ✉ <https://kuleuvencongres.be/htc16/>

6 February, Guildford, United Kingdom. **6th BMSS Ambient Ionisation Special Interest Group (SIG) Meeting**. Andrew Ray, ✉ andrew.ray@astrazeneca.com, ✉ <https://www.bmss.org.uk/bmss-ambient-ionisation-sig-meeting/>

16–21 February, San Diego, United States. **2020 Ocean Sciences Meeting (OSM)**. ✉ meetinginfo@agu.org, ✉

<https://www2.agu.org/ocean-sciences-meeting/>

17–22 February, Anaheim, California, United States. **2020 American Academy of Forensic Sciences (AAFS) 72nd Annual Scientific Meeting**. ✉ <https://www.aafs.org/home-page/meetings/future-past-aafs-meetings/>

23–27 February, San Diego, United States. **The Minerals, Metals & Materials Society (TMS) 2020 150th Annual Meeting**. ✉ mtgserv@tms.org, ✉ <https://www.tms.org/tms2020>

22–26 March, Philadelphia, United States. **259th American Chemical Society National Meeting**. ✉ natimtg@asc.org, ✉ <https://www.acs.org/content/acs/en/about/governance/committees/cwd/meetings.html>

4–7 April, San Diego, United States. **Experimental Biology 2020**. ✉ eb@faseb.org, ✉ <https://experimentalbiology.org>

26–29 April, Oviedo, Spain. **The 5th International Glow Discharge Spectroscopy Symposium**. Peter Robinson, ✉ pete@masscare.co.uk, ✉ <https://www.ew-gds.com/>

3–8 May, Vienna, Austria. **2020 European Geosciences Union (EGU) General Assembly**. ✉ secretariat@egu.eu, ✉ <https://www.egu2020.eu/>

24–28 May, Chiba City, Japan. **Japan Geoscience Union Meeting 2020**. ✉ <http://www.jpгу.org/>

24–26 May, Rome, Italy. **8th CMA4CH Meeting, Measurements, Diagnostics, Statistics in Environment and Cultural Heritage Fields**. ✉ infocma4ch@uniroma1.it, ✉ <http://www.cma4ch.org>

24–28 May, Winnipeg, Canada. **103rd Canadian Chemistry Conference**. ✉ <http://www.ccce2019.ca/>

24–28 May, Chiba, Japan. **Japan Geoscience Union (JpGU) Meeting**

2020. ✉ <http://www.jpгу.org/en/articles/20171208meetingplan.html>

27–28 May, Graz, Austria. **chii2020**. ✉ <http://www.chii2020.com/>

31 May–4 June, Houston, Texas, United States. **68th ASMS Conference**. ✉ <https://www.asms.org/conferences/annual-conference/future-annual-conferences>

4–5 June, Münster, Germany. **2nd Workshop on Laser Bioimaging Mass Spectrometry**. Michael Sperling, ✉ ms@speciation.net, ✉ <https://bit.ly/2VbCvoH>

7–10 June, Loen, Norway. **10th Nordic Conference on Plasma Spectrochemistry**. Yngvar Thomassen, ✉ yngvar.thmassen@stami.no, ✉ <http://nordicplasma.com/>

21–26 June, Honolulu, Hawaii, United States. **2020 Goldschmidt Conference**. ✉ helpdesk@goldschmidt.info, ✉ <https://goldschmidt.info/2020/>

21–26 June, Courmayeur, Italy. **18th Chemometrics in Analytical Chemistry Conference (CAC2020)**. ✉ ludovic.duponchel@univ-lille.fr, ✉ <https://cac2020.sciencesconf.org/>

24–26 June, Warsaw, Poland. **European Symposium on Atomic Spectrometry 2020**. Ewa Bulska, ✉ esas2020@uw.edu.pl, ✉ <http://www.esas2020.uw.edu.pl/>

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

29 June–1 July, Manchester, United Kingdom. **The 20th Biennial National Atomic Spectroscopy Symposium (BNASS 2020)**. Dr Phil Riby, ✉ phillip.riby@manchester.ac.uk, ✉ <http://www.rsc.org/events/detail/40623/bnass-2020-the-20th-biennial-national-atomic-spectroscopy-symposium>

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5–8 July, Skagen, Denmark. **International Association for Spectral Imaging (IASIM) 2020**. ✉ 2020@iasim.net, 🌐 <https://2020.iasim.net/>

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

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/>

8–10 September, Sheffield, United Kingdom. **41th British Mass Spectrometry Society Annual Meeting 2019-BMSS41**. Mark Mcdowall, ✉ mark_mcdowall@icloud.com, 🌐 https://www.bmss.org.uk/41st-bmss-annual-meeting/

9–17 September, Reno, NV, United States. **47th Annual Conference of Federation of Analytical Chemistry and Spectroscopy Societies (SciX2020)**. ✉ scix@scixconference.org, 🌐 <https://www.scixconference.org/index.php/scix-home/future-conferences>

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>

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

www.ukaachen.de/kliniken-institute/institut-fuer-immunologie/institut.html

4–8 October, Pittsburgh, United States. **2020 Materials Science and Technology Conference (MS&T20)**. ✉ netsoc@cim.org, 🌐 <http://www.matc-itech.org/>

25–28 October, Montreal, Canada. **2020 GSA Annual Meeting**. 🌐 <http://www.geosociety.org/>

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

2021

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/>

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

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

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

2021

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

Courses

2019

4–7 November, Berlin, Germany. **17th European Short Courses on Principles and Applications of Time-resolved Fluorescence Spectroscopy**. ✉ trf@picoquant.com, 🌐 <https://www.picoquant.com/trfcourse>

22–23 November, Leipzig, Germany. **7th Workshop on Field-Flow Fractionation-Mass Spectrometry (FFF-MS)**. Dr. Björn Meermann, ✉ nanoanalytics@univie.ac.at, 🌐 <https://www.ufz.de/index.php?en=46025>

Exhibitions

2019

18–20 November, Princeton, NJ, United States. **Eastern Analytical Symposium (EAS) and Exhibition**. ✉ askEAS@eas.org, 🌐 <http://www.eas.org/>

2020

1–5 March, Chicago, United States. **Pittcon 2020—Conference on Analytical Chemistry and Applied Spectroscopy**. ✉ pittconinfo@pittcon.org, 🌐 <https://pittcon.org/>

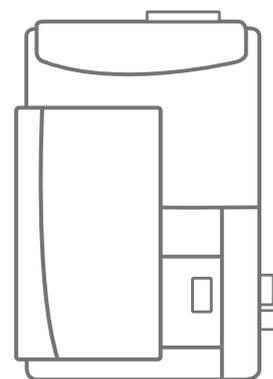
16–18 March, Dubai, United Arab Emirates. **ARABLAB 2020**. ✉ info@arablab.com, 🌐 <https://www.arablab.com/>

31 March–3 April, Munich, Germany. **analytica 2020: 27th International Trade Fair for Laboratory Technology, Analysis, Biotechnology and Analytical Conference**. 🌐 <https://www.analytica.de/>

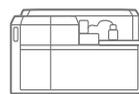
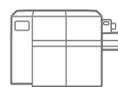
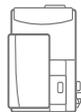
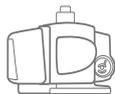
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