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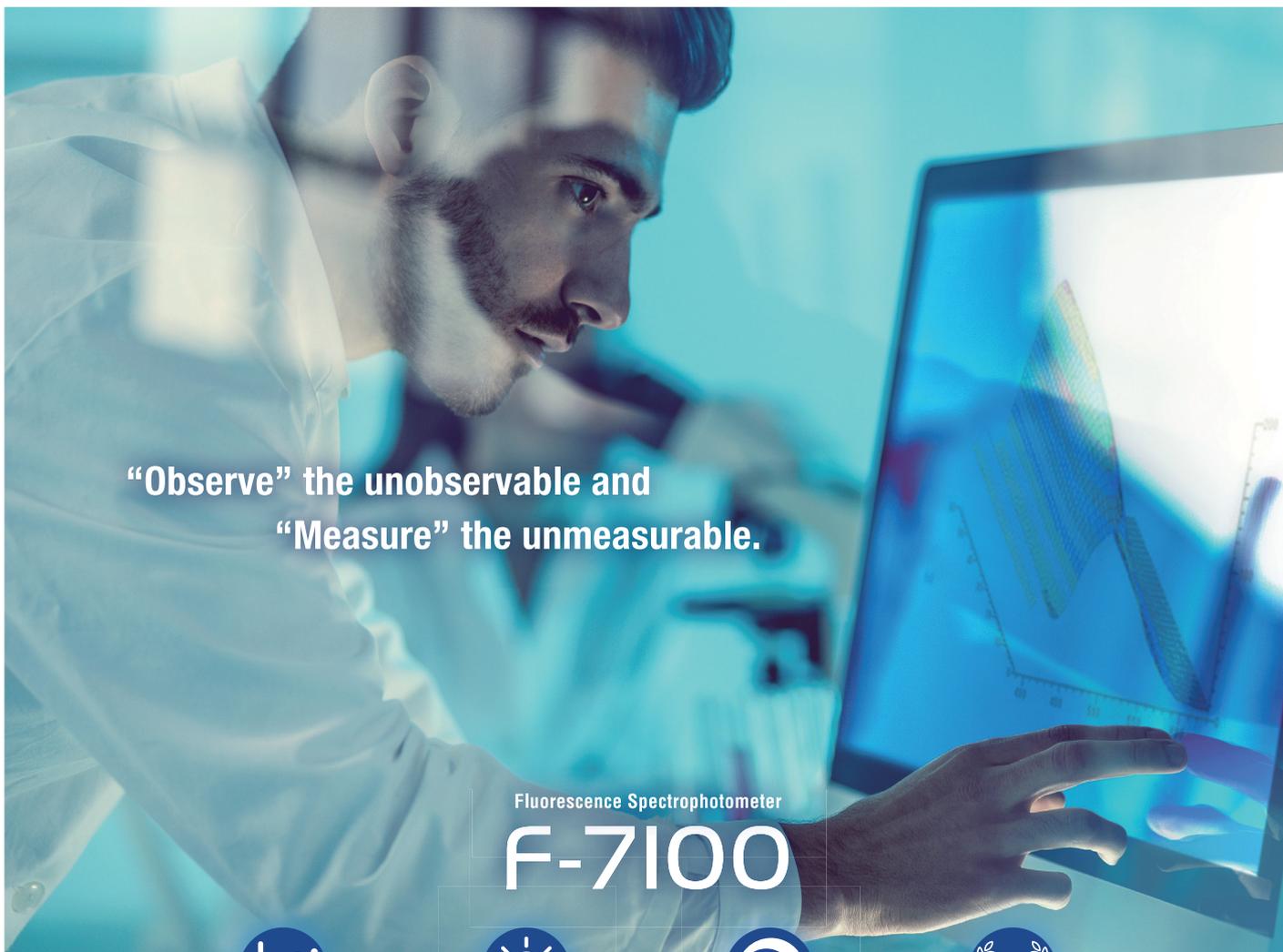
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I would like to take this opportunity to thank John Chalmers, who has retired as Article Editor, for all his work in finding and editing the interesting articles we have published over the last years. John has worked with authors to ensure that the articles are scientifically rigorous as well as a good read. All of us, authors, readers and myself are most grateful to John.

If you would like to contribute an article to a future issue, I would be delighted to hear from you ([ian@impublications.com](mailto:ian@impublications.com)). Articles in *Spectroscopy Europe* must be of interest to a wide range of our readers, not just those expert in the particular subject of the article.

We all know how spectroscopy and other analytical technologies have played important roles in detecting fraud and in authentication. This is certainly true in the art world and our first article, by Enrico Pigorsch, Matthias Finger, Johanna Kerber and Michael Fleck, describes "Investigation of paper collages by near infrared imaging

techniques". Paper collages, or photomontages, are part of the art market that is seeing much interest amongst collectors. It is difficult to detect forgeries just through expertise. The use of NIR imaging offers a number of ways to identify forgeries or authenticate the collage non-destructively; from determining the glue used to the revealing of printing on the back of the pieces or paper, which often have been taken from books and magazines.

"Total reflection X-ray fluorescence technique for multi-elemental analysis of food" is the topic of Rogerta Dalipi, Laura Borgese, Eva Marguí, Emanuele Sangiorgi and Laura Depero. X-ray spectroscopy techniques have some advantages over other atomic spectroscopy techniques in the analysis of foods, for instance in not requiring significant sample preparation. Amongst these, TXRF has higher sensitivity and limits of detection in the ng range. The authors look at the analysis of a number of very different foods, including seafood, honey and vegetables.

Tony Davies and Robert Lancashire ask "How standard are your standards?". They describe a number of the organisations working with standards that affect the spectroscopy field. It looks as if free access to these may be the only way to ensure their longevity.

In the Quality Matters column, Peter Jenks and Alan Nichols plot a path through the accreditation jungle and decide that "Confidence: the key to quality". With increasing numbers of readers' labs requiring auditing, ensuring that the reference materials and standards you are using meet the requirements of the auditors is essential.

Kim Esbensen and Claas Wagner have produced an extensive Sampling column, on "Representative mass reduction in the laboratory: riffle splitting galore (with or without errors)". They guide readers through the choice of mass reduction equipment and what needs to be done to ensure representative sampling.

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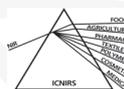
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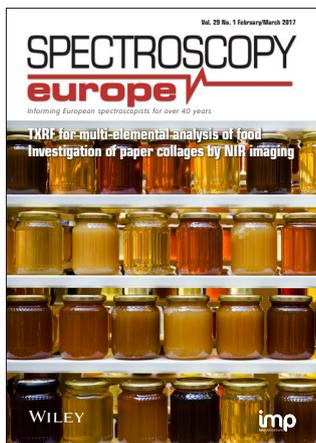
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The analysis of honey by total reflection X-ray spectrometry is described in the article starting on page 12.

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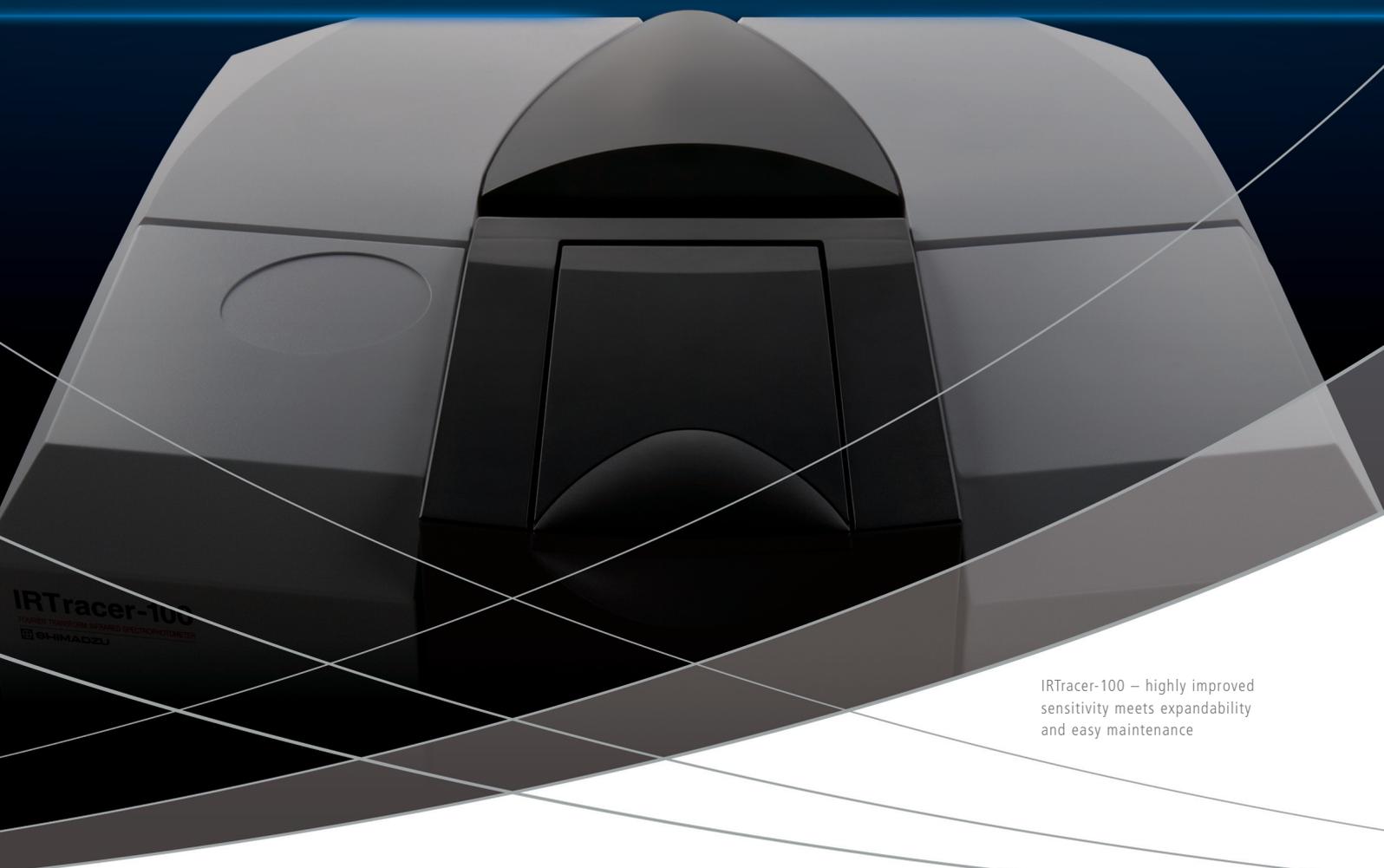
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# Investigation of paper collages by near infrared imaging techniques

Enrico Pigorsch, Matthias Finger, Johanna Kerber and Michael Fleck

PTS Papiertechnische Stiftung, Pirnaer Str. 27, D-01809 Heidenau, Germany

## Introduction

Spectral imaging techniques such as infrared reflectography (IRR) or ultraviolet-visible-near infrared (UV-vis-NIR) spectroscopy are well-established tools for examining works of art. In simultaneously providing both spectral and spatial information, these techniques make it possible to reveal invisible features and structures such as underdrawings or changes that have been made in the pictorial composition of paintings.<sup>1</sup>

Recent advances in sensor technology have extended the available spectral range of NIR imaging systems up to 2200 nm. This makes it possible to add to the existing structural information very specific chemical information and arrive at a chemical imaging analysis in micrometre dimensions.<sup>2</sup>

NIR chemical imaging measurements offer several advantages and create new possibilities in the analysis of works of art or documents on paper. Measurements conducted with a NIR line camera are totally non-destructive. Frame frequencies of up to 300 Hz allow rapid measurements of large areas. The lateral resolution of the chemical images can be 100  $\mu\text{m}$  or less. In addition, the relatively high penetration depth of the NIR radiation of about 250  $\mu\text{m}$  makes it possible to look not only at the surface but also inside the material or even through to the back of a sheet of paper.

Another interesting property of chemical imaging is the huge quantity of individual spectra recorded in one measurement (e.g. about five million spectra within an area of 30  $\times$  30 cm). This enables the extraction of the chem-

ical information from the spectral data set using unsupervised chemometric methods such as principal component analysis (PCA).<sup>3</sup> In this way, chemical structures or individual particles in a material sample can be detected quickly, visualised and eventually identified without much previous knowledge about the sample.

## Analytical approach to paper collages

The purpose of this article is to demonstrate the application of NIR chemical imaging measurements to the characterisation and authentication of paper collages. Paper collages or photomontages from the Dada art movement (1916 to 1930s) and from other artists of the 1920s and 1930s are very popular among today's art collectors, and there exists an active art trade in these works generating high sales values.<sup>4</sup> Hence, paper collages are more and more often the subject of forgeries and fraud. In most cases, the forgeries cannot be detected on the basis of scientific art expertise only. Chemical analysis methods must also be employed. Our institute (PTS) has a special expertise in paper analysis and in several cases has already assisted criminologists in revealing forgeries of paper art works. The analytical methods that are predominantly used are IR, NIR and Raman spectroscopy and corresponding spectral imaging techniques.<sup>5</sup>

In the case of paper collages, NIR imaging measurements can be employed to identify the glue used without have to remove the pieces of paper

from the carrier material. Another advantage of the imaging technique is that, for identification purposes, it is sufficient to find among the millions of spectra only a few with a sufficiently high intensity of the characteristic NIR glue bands. These spectra can easily be detected by chemometric methods.

Furthermore, printed text and pictures on the back of the collage paper pieces can be rendered discernible, although they remain invisible when visualised in transmitted light. In most cases, the pieces of paper used in making the collage were taken from contemporary illustrated magazines. In this way, the revealed text phrases or pictures are able to provide hints regarding a specific magazine and the time period or even the exact date of its publication. This in turn allows the given dating of the paper collage to be verified.

## Methodology

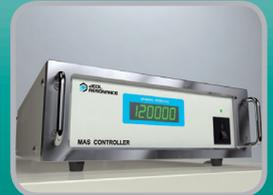
By way of a demonstration, a small paper collage (15  $\times$  20 cm) was prepared on a thick carton board (300  $\text{g m}^{-2}$ ) with eight different photo motifs and eight different synthetic and natural glues (Table 1).

The NIR imaging measurements were performed using a self-built measuring system consisting of a NIR camera KUSTA2.2MSI (LLA Instruments GmbH) and a sample table positioned on two movable axes. The NIR camera has a spectral range from 1229 nm to 2157 nm, from which the range between 1300 nm and 1866 nm was used. The InGaAs detector of the camera has 320 spatial pixels and 256 spectral pixels resulting in a spectral resolution of about 4 nm.



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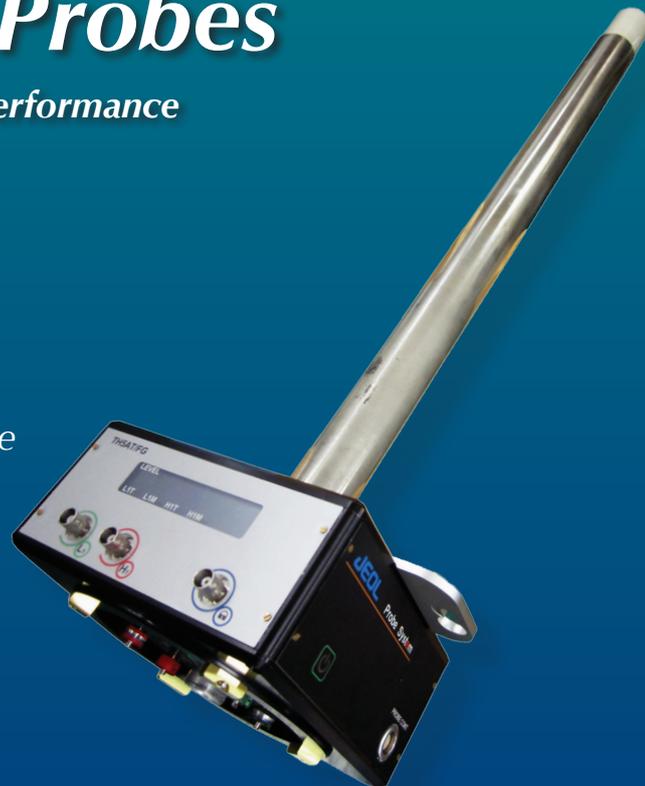


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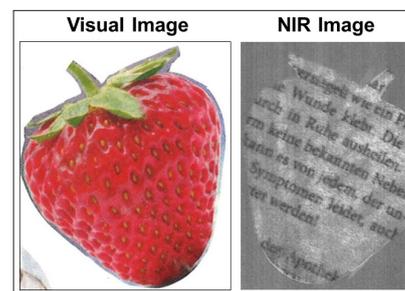
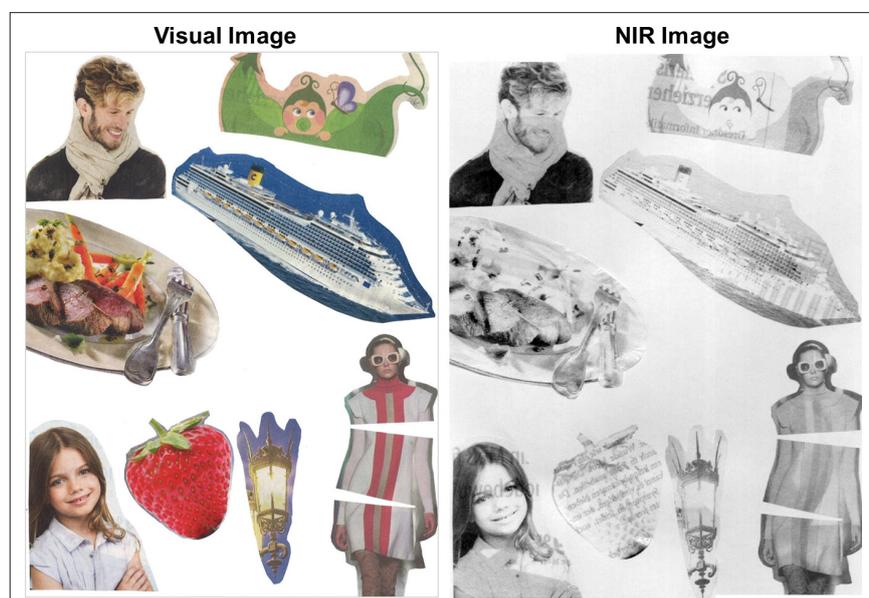
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**Table 1.** Collage motifs and the glues used.

No.	Motifs	Glue compounds	Glue products
1	Cruiser Ship	Polyvinylpyrrolidone	UHU stic
2	Young Man	Cellulose nitrate based	UHU Schnellkleber, hart
3	Lanterne	Polyacrylate	UHU Vielzweckkleber
4	Model	Polyvinylacetate	Ponal, wasserfest (Henkel)
5	Strawberry	Gum arabic	Boesner (Germany)
6	Plate	Starch	Potato starch
7	Girl	Animal glue (skin)	Zank (Germany)
8	Baby	Animal glue (bones)	Zank (Germany)

**Figure 2.** Visual and NIR images of strawberry motif. The grey scale of the NIR image represents score values of the fourth principal component.**Figure 1.** Visual and NIR images of the self-designed paper collage. The grey scale of the NIR image represents absorbance at 1300 nm.

A frame frequency of 161 Hz was used. The paper collage was scanned with a field of view (width) of 53 mm by moving the  $y$ -axis at a speed of  $13 \text{ mm s}^{-1}$ , resulting in a lateral resolution of about  $170 \times 80 \mu\text{m}$ .

The data was analysed using spectral imaging software developed in-house in Matlab (The Mathworks Inc.).

### Measurements on the self-designed paper collage

Figure 1 shows the visual image and the grey colour-coded NIR image of the paper collage. The different grey colours of the NIR image represent different grades of reflection of the NIR radiation

at a wavelength of 1300 nm. Hence, the picture is not a real chemical image, but it gives an impression of the high lateral resolution of the NIR imaging measurement.

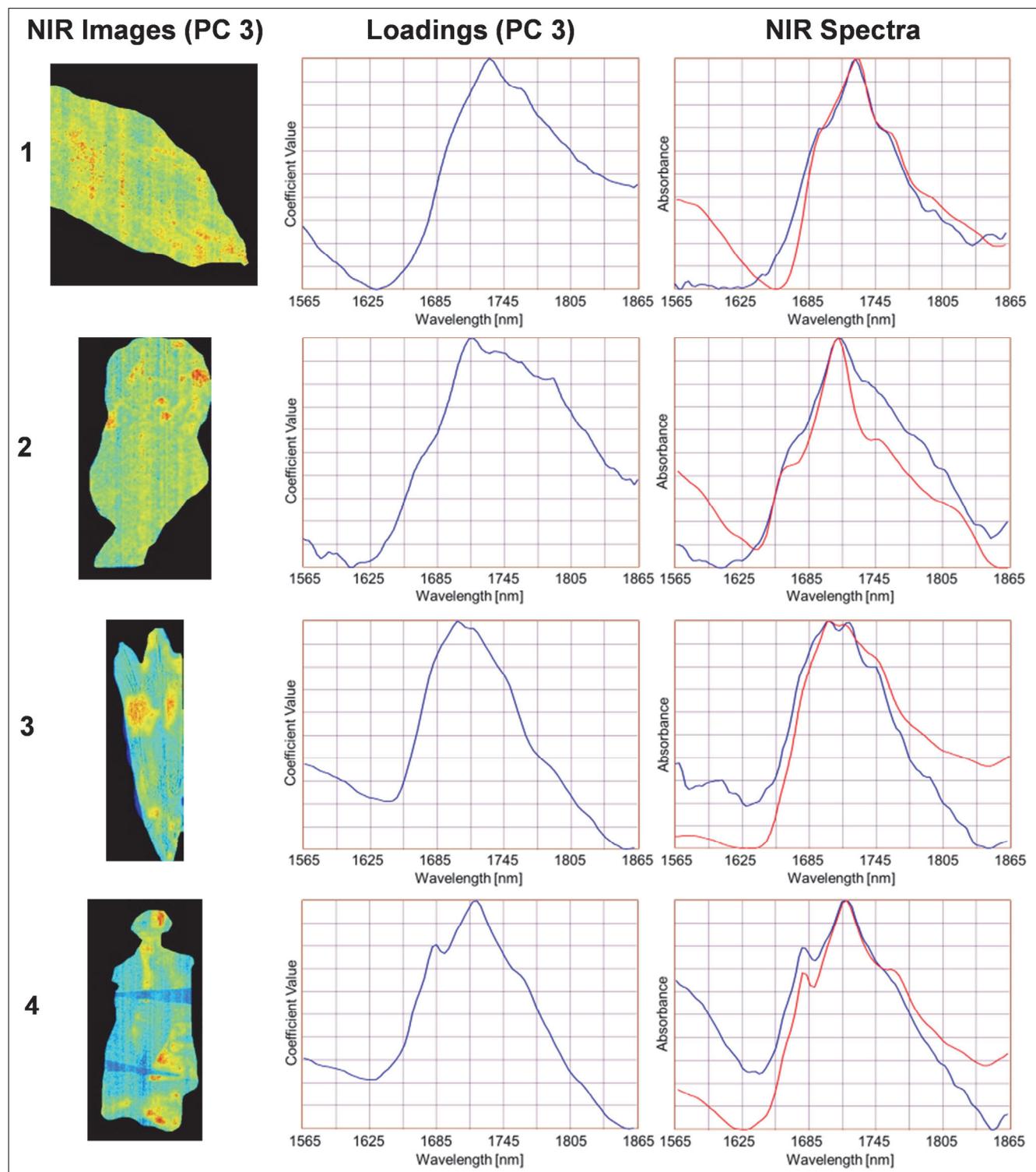
In the strawberry motif of the NIR image, some printed text is already discernible on the back of the piece of paper. The German text is rendered legible by using the fourth principal component of a PCA and changing the orientation of the characters (Figure 2).

In order to identify the different glues, it is necessary to find NIR spectra with characteristic NIR bands of the glue that have sufficient intensity. This was done using an unsupervised PCA to examine the NIR imaging data

for spectral differences. The PCA was performed for each motif separately. The corresponding images in Figures 3 and 4 show in red colour those points with the highest scores for the principal components that represent the spectral differences due to the glue. In some cases, the PCA loadings already show the characteristic NIR absorptions of the glue compounds. But to achieve a more precise match with the reference spectra for all glues, the average spectra of the respective 10–20 spectra with the highest scores were calculated and finally subtracted with a weighted paper spectrum. The results are shown in Figures 3 and 4. The comparison of the difference spectra with the reference spectra shows very good agreement for all eight glue samples including the natural glues.

### Measurements on paper collage art works

Measurements on real paper collage artworks yielded results which are highly illustrative of the information that can be extracted from hidden printed text on the back of the pieces of paper. The paper collages which were investigated belong to a collection of paper collages made by Karl Waldmann.<sup>6</sup> The origin of the works and even the real existence of an artist by the name of Karl Waldmann are a continuing subject of debate that will not be discussed here.<sup>7</sup> Another unresolved question is the time period during which the paper collages might have been made. The period assumed ranges from 1930 to 1958.

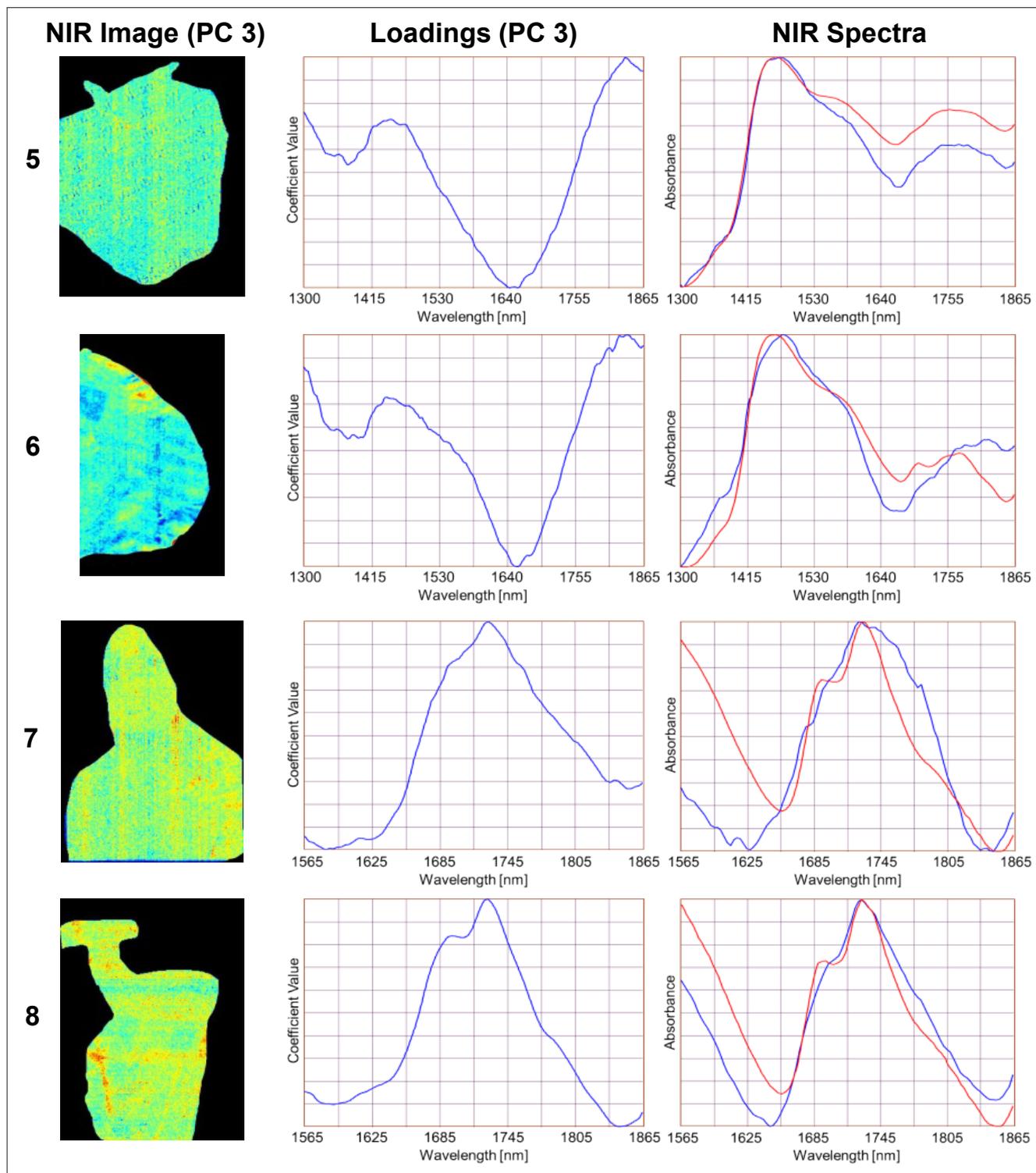


**Figure 3.** NIR images, loadings PC 3 and NIR spectra of the glue for collagen motifs 1 to 4. Colour coding represents score values of third principal component. Red spots indicate higher concentrations of the glue substance. The NIR spectra are the reference spectra of the glue (red) and the average spectra of red spots subtracted with a weighted paper spectrum (blue).

In all examined works, the glue that was found was gum arabic which is not in contrast to the assumed dating of

the works. Figures 5 and 6 show two visualisations of hidden prints in two of the paper collages. A printed text

and picture were revealed on the back of the brown paper background in the paper collage in Figure 5. Further inves-



**Figure 4.** NIR images, loadings PC 3 and NIR spectra of the glue for collagen motifs 5 to 8. Colour coding represents score values of third principal component. Red spots indicate higher concentrations of the glue substance. The NIR spectra are the reference spectra of the glue (red) and the average spectra of red spots subtracted with a weighted paper spectrum (blue).

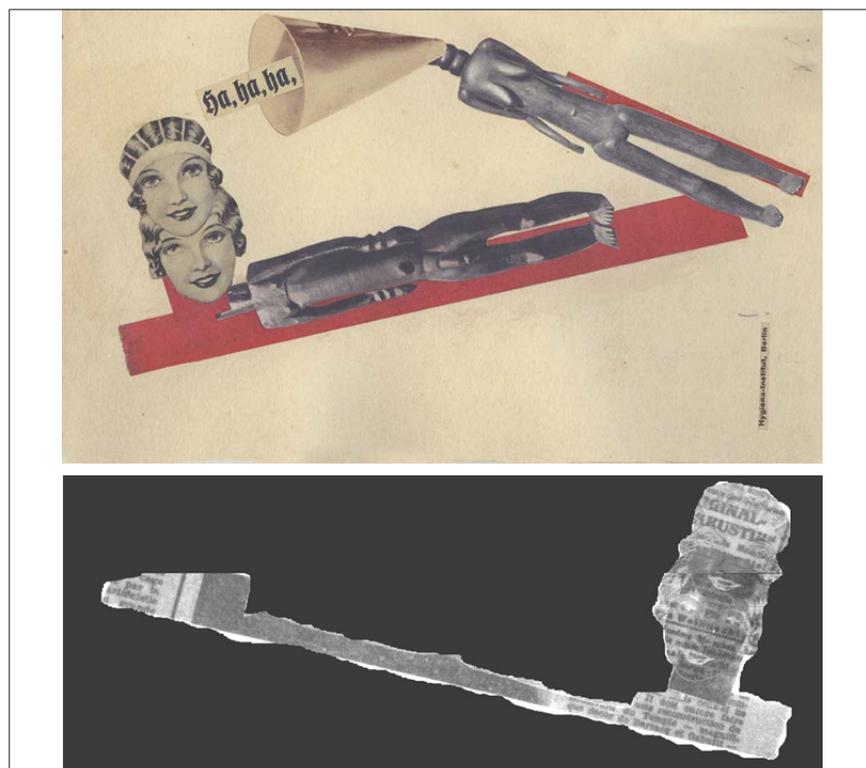
tigation showed that the brown paper was the cover of an ethnological book entitled “*Neu-Guinea*”, *Schriften-Reihe*

“*Kulturen der Erde*”, Folkwang Verlag by Ernst Fuhrmann published in 1922. This was a significant result, because it

turned out that photos from this book were used in at least 14 paper collages in the Waldmann collection.



**Figure 5.** Visual (top) and NIR images (bottom) of the paper collage “Kinder am Strand von Merauke” (No. 902) from the Karl Waldmann collection.<sup>6</sup>



**Figure 6.** Visual (top) and NIR images (bottom) of the paper collage “Ha, ha, ha” (No. 824) from the Karl Waldmann collection.<sup>6</sup>

The following text in French was found on the back of the red paper strip in the paper collage in Figure 6: “Il doit encore faire ... juste reconstruction du ... du Temple – magnifique décor de Brasacq et Gabutti-...”. This text can be linked to the famous French film “Les Enfants du Paradis”. The boulevard du Temple in Paris was the major setting of the film, and Léon Brasacq and Raymond Gabutti were the set designers. The film was shot in 1943/44 and first publicly shown on 9 March 1945 in Paris. Therefore, the paper collage could not have been made before 1945.

## Conclusions

The investigations showed that NIR imaging measurements are a powerful tool for characterising and authenticating paper collage art works. They can provide decisive clues regarding the material used and the sources of the pictorial motifs which in turn can confirm dates and eventually assist in authenticating works of art.

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# Total reflection X-ray fluorescence technique for multi-elemental analysis of food

Rogerta Dalipi,<sup>a</sup> Laura Borgese,<sup>a</sup> Eva Marguí,<sup>b</sup> Emanuele Sangiorgi<sup>c</sup> and Laura E. Depero<sup>a</sup>

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## Introduction

Elemental analysis is needed more and more in the food industry; from a nutritional point of view and for safety and quality purposes. Food is our most significant source of major (Ca, Cl, C, H, Mg, N, O, P, K, Na, S), minor-trace (F, I, Fe, Si, Zn) and ultra-trace (Cr, Co, Cu, Mn, Mo, Ni, Se, V) essential elements. However, non-essential and/or potentially toxic trace elements, like Al, As, Cd, Hg, Pb, Sb and U, may also contaminate food, entering the food chain from the environment, processing and storage.

Spectroscopic techniques like inductively coupled plasma mass or atomic emission spectrometry (ICP-MS or ICP-AES) are usually selected for elemental determination in foodstuffs, due to their multi-elemental capabilities and low limits of detection. These techniques require some additional sample treatment for the total destruction of organic matrices: mainly acidic digestion, especially for solid samples. This is a critical step involving the use of dangerous reagents. Moreover, losses of analytes by volatilisation are common, and the procedure itself is very time-consuming. The use of other methodologies, including X-ray fluorescence (XRF) spectrometry, for direct analysis of solid food samples has increased

over the last few years. Among XRF techniques, total reflection X-ray fluorescence (TXRF) is preferred, having higher sensitivity and a limit of detection at the nanogram level.

## Basic principles and analytical capabilities of TXRF

TXRF is a variation of energy dispersive XRF spectrometry (EDXRF). It differs from EDXRF mainly in the experimental setup geometry. An angle lower than  $0.1^\circ$  is required in TXRF in order to obtain the reflection of the whole beam on the reflector, instead of the conventional angle of  $45^\circ$ , as illustrated in Figure 1.

TXRF is primarily used for chemical micro and trace analyses. For these purposes, small quantities, mostly solutions and suspensions, are deposited

on optical flat reflectors, i.e. quartz glass, and evaporated to dryness. After that, the residue is measured in a spectrometer equipped with energy dispersive detector positioned a few millimetres above the reflector surface. Most of the incident beam radiation is reflected and, as a consequence, the spectral background is reduced. In this way, the fluorescence yield is very high and absorption effects minimised. These characteristics allow better detection limits ( $10^{-7}$  to  $10^{-12}$ g) compared to those of conventional EDXRF. Matrix effects are negligible if the thin film requirements are fulfilled, i.e. samples that are thinner than the critical thickness.

Several TXRF spectrometers are commercially available. The main producer companies are Bruker (S2 Picofox, S4 T-Star), Rigaku (Nanohunter), GNR (TX 2000) and

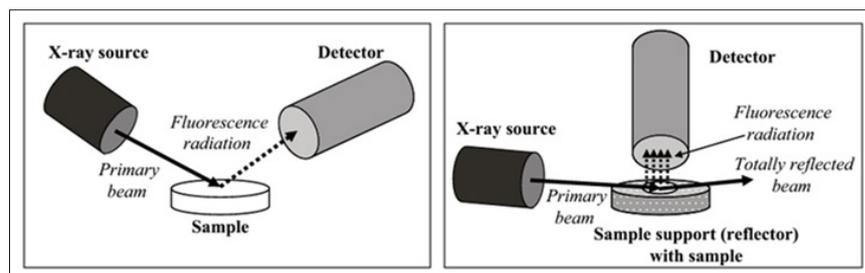


Figure 1. Instrumental setup for conventional XRF (left) and TXRF (right).

**Table 1.** Instrumental parameters of TXRF spectrometers.

S2 PICOFOX TXRF benchtop spectrometers		
Anode	Mo	W
X-ray tube	Air-cooled metal ceramic	Air-cooled metal ceramic
Maximum power	40W	50W
Optics	Multilayer monochromator (17.5 keV)	Multilayer monochromator (35 keV)
Detector	Silicon drift detector, Area: 30 mm <sup>2</sup> , FWHM: 139.43 eV (Mn K $\alpha$ )	Silicon drift detector, Area: 10 mm <sup>2</sup> , FWHM: 146.72 eV (Mn K $\alpha$ )
Filter	Mo 10.00 $\mu$ m	Ni 50.00 $\mu$ m
Sample changer	Manual version for single samples	Automatic version with cassette for up to 25 samples
Atmosphere	Air	Air
Voltage	50 kV	50 kV
Current	750 $\mu$ A	1000 $\mu$ A
Live time	600 s	2000 s

ATI (Wobistrax). The most recent TXRF spectrometers are benchtop instruments equipped with low-power, air-cooled X-ray tubes.

In the next sections several analytical TXRF methodologies for the analysis of solid food samples are described. In all cases, benchtop systems have been used (Bruker S2 Picofox TXRF spectrometer). Table 1 shows the instrumental setups and measurement parameters.

### Application of TXRF in foodstuff analysis

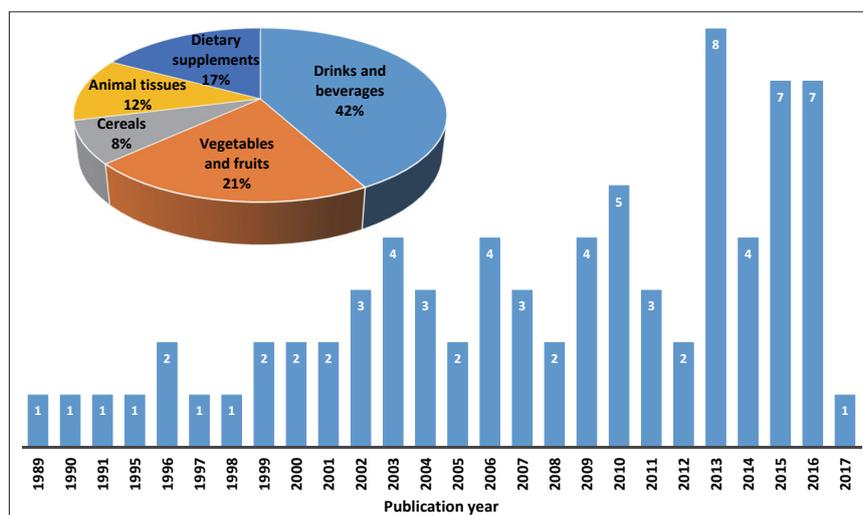
The first paper about elemental analysis of foodstuff by means of TXRF dates back to 1989. The number of publications in this field has increased during the last ten years. Today, TXRF is emerging as a powerful tool for food analysis, especially where a holistic approach is followed. Drinks, beverages, vegetables, fruits, herbs, spices, cereals, animal derivatives and dietary supplements as foodstuff samples have been analysed by TXRF for safety and quality purposes. Figure 2 shows the trend of publications related to TXRF analysis of foodstuff and the percentages of the fields of application. In this paper we will focus on some examples of TXRF analysis of seafood, vegetables and honey, considering sample preparation procedures and methods developed to obtain accurate and reliable results.

More details can be found in the literature.<sup>1–3</sup>

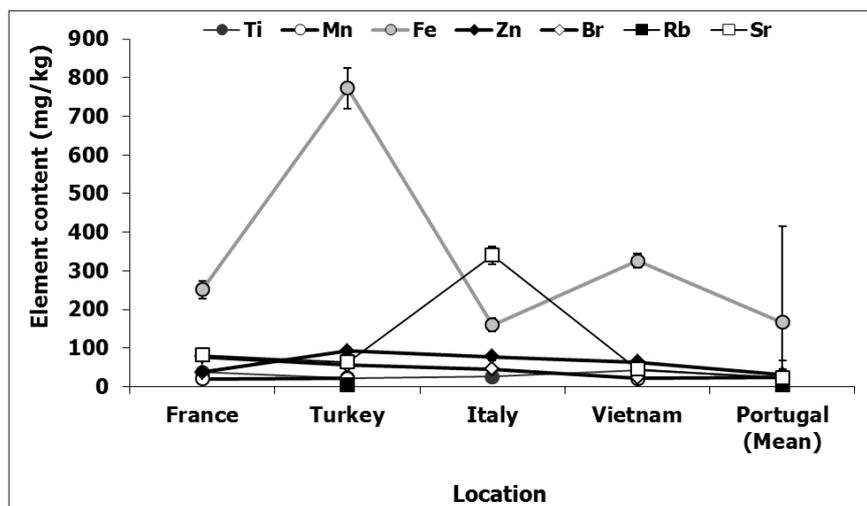
### Seafood analysis

It is well known that bivalves may accumulate large amounts of metals and for this reason they are widely used as bio indicators, but they are also analysed for food safety purposes. Here we show the analysis of different commercial clam species from Portuguese markets for the determination of elements in the high–medium mg kg<sup>-1</sup> range (higher than 5–10 mg kg<sup>-1</sup>), comparing two different sample preparation procedures.<sup>1</sup> For this purpose, the soft

tissues were freeze dried and ground to a particle size less than 100  $\mu$ m. The conventional microwave digestion with a mixture of HNO<sub>3</sub> + H<sub>2</sub>O<sub>2</sub> and sample suspension in a disperser solution were compared as sample treatments for TXRF analysis. Different amounts (20, 50 and 100 mg) and dispersant solutions (ultrapure water and 0.1% Triton<sup>®</sup> X-114) for the preparation of suspensions were tested. For quantification, Y was used as internal standard. All the tests were performed using the reference material GBW08571 “mussel muscle tissue”. Measurements were performed with W excitation.



**Figure 2.** Number of publications related to TXRF analysis of foodstuff and the fields of application (source ISI Web of Knowledge).



**Figure 3.** Minor elemental determination in the studied edible clams from different locations.

It was found that the best conditions for the analysis of solid biological samples are 100 mg of sample and 1 mL of ultrapure water as disperser agent. The comparison between acid digestion and suspension showed comparable results. The results of the analysis of commercial edible clams from different locations by means of TXRF are shown in Figure 3. As we can see, similar concentrations were obtained for all the locations, except for Fe in Turkish and Sr in Italian clams. The measured concentration levels agree with literature data.

### Honey analysis

Elemental determination in sugar-rich foodstuff samples has been a challenging analytical task for researchers due to matrix effects. Among all such foods, honey is the most studied, both for environmental and food safety reasons. In this work, we show a quick and simple analytical method for multi-elemental analysis of different honey samples by means of a TXRF system equipped with a Mo anode X-ray tube.<sup>2</sup> For this purpose, 45 honey samples with different botanical and geographical origins were selected. Each sample solution was prepared by mixing about 0.5 g of honey and 10 mL of ultrapure water. The volume of 1 mL of each sample solution was added with Ga and thoroughly homogenised, to prepare the specimen for

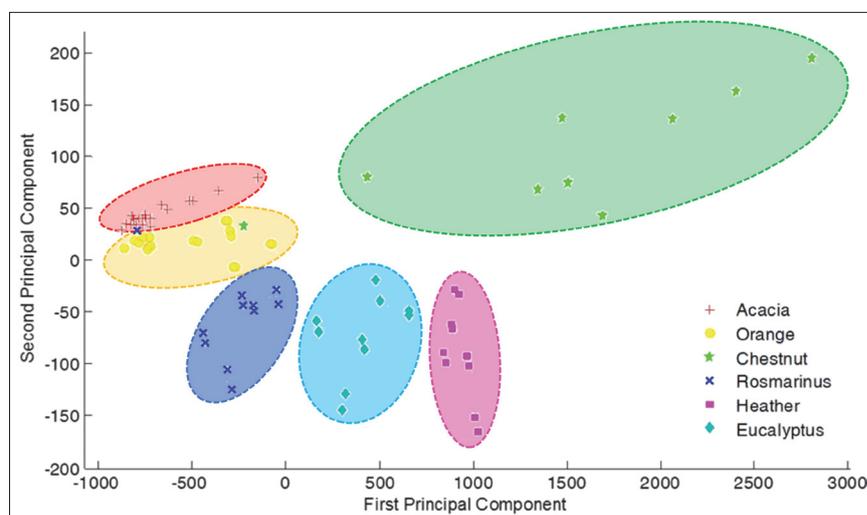
TXRF analysis with a final Ga concentration of  $0.5 \text{ mg L}^{-1}$ . Three replicates were prepared depositing  $10 \mu\text{L}$  of specimen on each quartz glass sample carrier and dried.

Our data are in agreement with those reported in the literature for similar studies and different spectrometric techniques, such as AAS and ICP-MS. All these data were used together for chemometric investigation. Single botanical origin honey samples were considered. Principal component analysis (PCA) allowed a clear differentiation according to botanical origin (Figure 4), where K, Mn, Zn and P give the main contribution. These results clearly show that TXRF

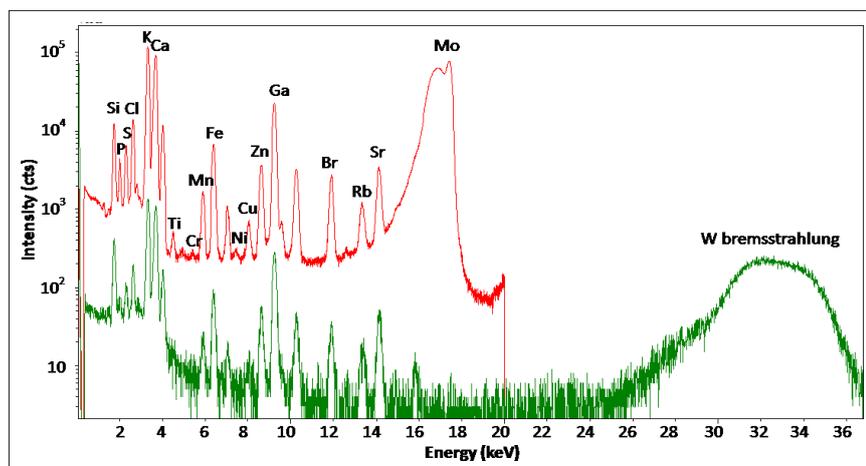
provides results comparable to the other techniques.

### Vegetal foodstuff analysis

Vegetables are of fundamental importance in the human diet and are primary recipients of essential and potentially toxic elements from the environment. These elements are transferred to humans directly through eating, or indirectly through milk and meat from animals fed with vegetables. In this work we demonstrate that TXRF allows accurate and precise analysis of lyophilised vegetables, with a simple and fast procedure for sample preparation. Six certified reference materials provided by the National Institute of Standards & Technology (NIST) were selected: SRM 1515 (apple leaves), SRM 1547 (peach leaves), SRM 1570A (spinach leaves), SRM 1572 (citrus leaves), SRM 1567A (wheat flour) and SRM 1568A (rice flour). The solid samples were prepared by suspending about 20 mg of powder in 1 mL of dispersant solution (ultrapure water or 1% Triton X-100). Gallium was added as internal standard with a final concentration of  $10 \text{ mg L}^{-1}$ . Duplicates were prepared for each sample and  $10 \mu\text{L}$  were deposited on a siliconised quartz reflector and dried. Measurements were performed with two low-power benchtop TXRF systems equipped with Mo and W X-ray tubes.<sup>3</sup>



**Figure 4.** PCA Scatter plots of TXRF and other spectroscopic techniques data found in the literature with different botanical and geographical origins.



**Figure 5.** TXRF raw spectra for the analysis of SRM 1570A sample obtained using Mo (red spectrum) and W (green spectrum) as excitation sources.

The typical raw spectra of SRM 1570A are shown in Figure 5. The main differences between Mo and W excitation are in the measurement energy (keV) range and the background produced. The Mo TXRF system has better sensitivity and detection limits are lower compared to the W system,

with some exceptions. Indeed, the W system is more suitable for the determination of high Z elements like Cd and Hg that cannot be determined with the other system.

Results are summarised in Figure 6 and compared with certified values. Most of the values are in agreement.

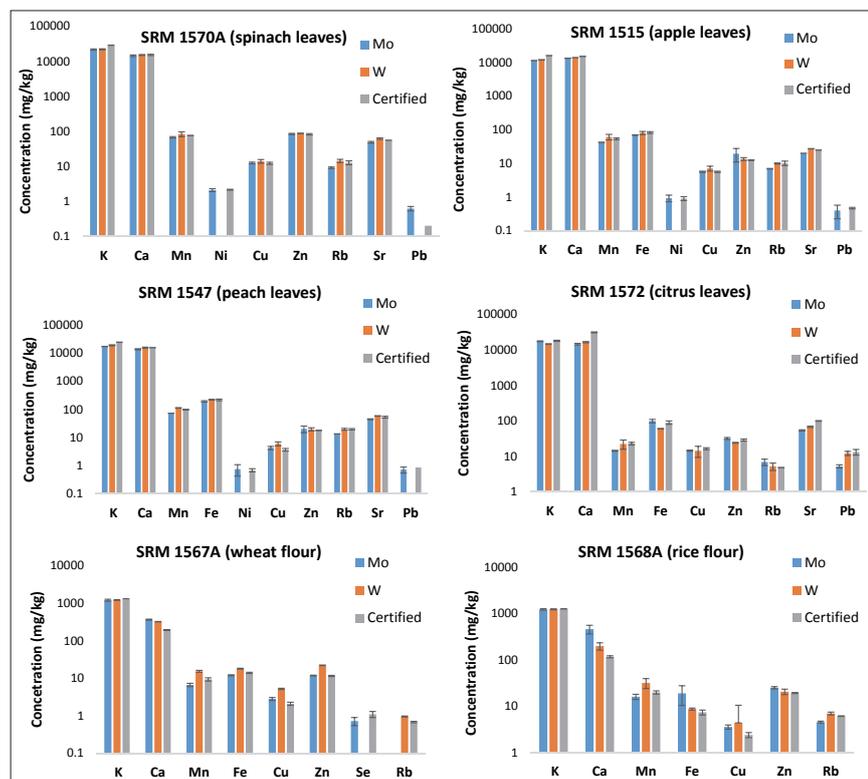
Differences for light Z elements like K and Ca are due to the absorption effects when measurements are performed in air (not under vacuum conditions). Moreover, due to the limited sensitivity of the W TXRF system, elements in very low concentrations could not be determined.

### Concluding remarks and future perspectives

In this work, we have shown the suitability of low-power, benchtop TXRF instrumentations equipped with Mo and W X-ray tubes for multi-elemental analysis of different foodstuff samples. TXRF offers a fast and simple way to perform screening and reliable quantitative analysis of food samples with complex matrices. TXRF may be successfully used for food safety, traceability and quality control. Moreover, TXRF has some advantages over other spectroscopic techniques, such as the possibility to get simultaneous multi-elemental information, the low amount of sample required to perform the analysis and the possibility to get quantitative results without external calibration. The use of TXRF is still limited due to the lack of recognised standard methods, and overcoming this will require a significant collaborative effort to develop guidelines for experimental procedures. Future improvements in TXRF devices and sample pre-treatments are expected to offer further approaches for low-cost routine and on-line analysis.

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**Figure 6.** Comparison of the results obtained by Mo and W TXRF systems and certified values. Error bars represent the standard deviation of duplicates.

# How standard are your standards?

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Berlin, February 2017... and high level representatives of a number of consortia are together on the podium during the SmartLab Exchange conference discussing the latest developments in standards, which include initiatives in some spectroscopic fields. In the last ten years or so, several well-funded initiatives, strongly supported by the pharma companies, have formed consortia to deliver to their members utilities and "standards". Of course, this column greatly welcome initiatives towards more standardisation,. However, as again shown in Berlin, the use of the term "standard" itself is being widely used as a marketing tool and risks delivering the wrong message to those hearing presentations around these initiatives for the first-time.

## International standards bodies

One of the very first bodies to recognise and adopt standardisation in the support of international trade was the International Electrotechnical Commission (IEC). I like the definitions they use:

"A standard is a document, established by consensus and approved by a recognized body, that provides, for common and repeated use, rules, guidelines or characteristics for activities or their results, aimed at the achievement of the optimum degree of order in a given context."

"International Standard"

"An International Standard is a standard adopted by an international standards organization and made available

to the public." The definition given in all IEC standards reads: "A normative document, developed according to consensus procedures, which has been approved by the IEC National Committee members of the responsible committee in accordance with Part 1 of the ISO/IEC Directives."

The IEC is a not-for-profit, quasi-governmental organisation, founded in 1906, whose members are National Committees, and they appoint experts and delegates from industry, government bodies, associations and academia to participate in the technical and conformity assessment work of the IEC.

ISO, the International Organization for Standardization, was the result of an international meeting in 1946 when delegates from 25 countries met at the Institute of Civil Engineers in London. They decided to create an international organisation "to facilitate the international coordination and unification of industrial standards". With remarkable speed, by 23 February 1947, the new organisation was ready and ISO began operations. Today, the ISO Central Secretariat is based in Geneva, Switzerland.

They cover many of the areas outside of the remit of the IEC and nicely improve on the IEC's rather oblique "...optimum degree of order in a given context" to something everyone can identify and understand "fit for purpose".

"A standard is a document that provides requirements, specifications, guidelines or characteristics that can be used consistently to ensure that materials, products, processes and services are fit for their purpose."

Now these organisations were established because of a similar perceived need and threat to international trade and development.

The International Union of Pure and Applied Chemistry goes back even further. IUPAC was formed in 1919 by chemists from industry and academia, who recognised the need for international standardisation in chemistry. The Union was formed to handle standardisation of weights, measures, names and symbols and is essential to the "well-being and continued success of the scientific enterprise and to the smooth development and growth of international trade and commerce".

The International Association of Chemical Societies (IACS) had met in Paris in 1911 and produced a set of proposals for the work that the new Association should address, including:<sup>3</sup>

- Nomenclature of inorganic and organic chemistry;
- Standardisation of atomic weights;
- Standardisation of physical constants;
- Editing tables of properties of matter;
- Establishing a commission for the review of work;
- Standardisation of the formats of publications;
- Measures required to prevent repetition of the same papers.

In modern times this work of these international standards bodies has necessarily evolved and extended into the digital domain as the delivery of the scientific content has moved into this environment.

# TONY DAVIES COLUMN

## New industrial standardisation efforts

The following bodies presented initiatives at SmartLab. Their initiatives will impact spectroscopic data handling and the presenters were brave enough to sit for a panel discussion and subsequent Question and Answer session.

### SiLA: "Standardization in Lab Automation"

Founded in 2008, driven by Roche, Novartis and Actelion, SiLA is a non-profit organisation whose documentation is only available to members. Downloads of their documents require registration as a "Personal Member" or an upgrade to "Corporate Member". On the content front, the ontologies and taxonomies are agreed amongst members.<sup>4</sup>

SiLA is based on HTTP/2, the successor of the Internet standard HTTP, which is likely to exist for decades as well (HTTP is from 1999). HTTP/2 is an Internet Engineering Taskforce (IETF) standard.

- 2009: Project-based internal implementations of the standard. Release of SiLA Specifications V1.0
- 2010: 1st public implementations of device interface standard. Release of SiLA Specifications V1.1
- 2011: Started to evaluate existing data standards; shared feedback with AnIML
- 2012: SiLA began working on data standard. Release of SiLA Specifications V1.2
- 2013: PoC of updated AnIML standard. Release of SiLA Specifications V1.3
- Oct 2016: SiLA 2 roadmap officially announced (go-live planned for mid-2017)

### Proteomics Standards Initiative

HUPO, the Human Proteomics Organisation, develops data format standards for proteomics, looking at both data representation and annotation standards. They aim to involve data producers, database providers, software producers and publishers etc. Very much aimed at driving the public deposition of analytical data in this field. Main activities are:

- Formats: usually an XML schema (but also tab-delimited files)

- Controlled vocabularies—currently around 2600 terms which are usually an Open Biomedical Ontologies OBO-style, hierarchical controlled vocabulary precisely defining the metadata that are encoded in the formats.

- Minimum information (MIAPE) specifications: format-independent specification of minimum information guidelines.
- Databases and tools: software implementations to make the standards truly useful.
- Community interaction to ensure deposition of data in public repositories.

As they are focussed on mass spectrometry-based proteomics, they have developed the following PSI Standard File Formats for MS:<sup>5</sup>

- mzML (MS data)
- mzIdentML (Identification)
- mzQuantML (Quantitation)
- mzTab (Final Results)
- TraML (SRM, Selected Reaction Monitoring)

This organisation intends in the next five years to focus on improving adoption/support, especially in vendors' software, to move on to handle mass spectrometry metabolomics data. They wish to finalise compatible formats with genomics data, e.g. proBed and proBAM (applicable for proteogenomics studies) and start working with the structural biology community (since MS proteomics is being increasingly used in that context).

### ASTM Subcommittee E13.15 on Analytical Data

XML standardisation effort started in this form in 2003.<sup>6</sup> In recent years, it has been driven hard and championed by the very patient Burkhard Schaefer. Recent publications are available in chromatography and mass spectrometry.<sup>7</sup>

- E1947-98(2014) Standard Specification for Analytical Data Interchange Protocol for Chromatographic Data
- E1948-98(2014) Standard Guide for Analytical Data Interchange Protocol for Chromatographic Data
- E2077-00(2016) Standard Specification for Analytical Data

Interchange Protocol for Mass Spectrometric Data

- E2078-00(2016) Standard Guide for Analytical Data Interchange Protocol for Mass Spectrometric Data

### Allotrope example: semantics provides common meaning

In 2012, the Allotrope Foundation was launched funded by the member companies, mainly from the pharma industries, through annual subscription. The governance of Allotrope Foundation is achieved via consensus within the Foundation, and is administered by a Board of Directors comprised of two individuals from each member company.<sup>8</sup>

- 2012: Allotrope launched, scope and strategy defined
- 2013: Initiate software development, evaluation of existing standards
- 2014: Feasibility studies and POCs, ADF design, testing and due diligence
- 2015: API and taxonomy development, V1.0 released internally, first deployments inside member companies
- 2016: ADF/API updates, V1.1 released internally (March 2016), API testing, Minimum Viable Product (MVP) defined, V1.2 release for internal beta testing (Nov 2016)

In the field of standards development (document standards, metadata and test data), they evaluate existing data standards and define appropriate controlled vocabularies and ontologies to use with the Allotrope Framework. They intend to provide within their membership group test datasets for use in development and map the metadata associated with the test data to existing standard definitions.

The Allotrope Foundation has a number of V1 taxonomies under development. The member companies take on the initial deployments of individual technical areas. These include gas chromatography, Karl Fischer, liquid chromatography, mass spectrometry, nuclear magnetic resonance spectroscopy, thermogravimetric analysis, ultraviolet spectroscopy, capillary electrophoresis, cell counter, cell culture analyser, blood gas analysis, balance and pH.

*continued on page 25*

## Confidence: the key to quality

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That the quality of analytical data matters is accepted; indeed it is the name of this column! At the heart of quality data is a quality system and underpinning that system is the proper use of reference materials. So, if the appropriate reference materials are sourced and used properly to validate an analytical system then all will be well.

Unfortunately, it is not quite so simple. Despite the best efforts of the International Standards Organisation (ISO), the International Laboratory Accreditation Council (ILAC) and numerous national accreditation bodies, all reference materials are not created equal! This means that the wily quality manager must know what to look out for when selecting the right reference material to use. This means that even in a world that seems well regulated "caveat emptor", or the buyer alone is responsible for checking the quality and suitability of goods before a purchase is made, still applies.

This article has been written to help guide and inform reference material users in their selection of appropriate and suitable reference materials. It is based on several talks Alan and I have given over the years. We plan to follow up with an article on choosing the "right" reference material, and will give guidance on what to do when the right reference material simply does not exist. We will go on to introduce the concept of "commutability", which is becoming a hot topic within the reference material community.

Certified reference materials are generally thought to be of the highest metrological status (i.e., scientifically valid).

They can be produced by National Metrology Institutes such as LGC, IRMM, BAM, or by "accredited producers". Whilst National Metrology Institutes do not need accreditation due to their legal status, some chose to become accredited, such as IRMM and LGC.

Any organisations that are doubly accredited to ISO/IEC 17025 and ISO Guide 34, may call themselves a reference material producer and legitimately produce and certify certified reference materials according to ISO definitions and standards. The combination of ISO 17025 and ISO Guide 34 means that the reference material producer has demonstrated that they are capable of competent measurement and have demonstrated competency in the production and distribution of reference materials.

Accreditation documents are available on the accrediting body website and usually on the reference material producer website that details the scope of their accreditation. More on this later.

However, there are no laws or requirements (outside the ISO world, which is not legally mandated) that prevent anyone from calling anything they want (even a yellow dog!) a certified reference material. So how does a Quality Manager know if a product is a legitimate certified reference material and how does a laboratory choose the best one for a particular application?

What about ISO 9001 Certification? Many producers of reference materials state they hold an ISO 9001 Certificate. Whilst this is good, it has no relevance to the production of a reference material or certified reference material. All ISO 9001 Certification does is demonstrate an

organisation has a quality system. An ISO 9001 quality system has no associated published scope and it is not checked or audited by a technical expert from an accreditation body.

The current situation, as quoted from a Chinese representative from APLAC is that it is "Chaos". Why is this?

There are many different representations (and mis-representations) in the market and to make matters worse some reference material producers' certified reference materials violate or stretch the rules. For example, some accredited reference material producers have a small portfolio of certified reference materials and a large portfolio of reference materials or standards that are NOT covered by the scope of their accreditation. Often the producers marketing literature will imply that all the reference materials are certified reference materials because the producer is accredited... in these authors' opinion such behaviour is reprehensible and is verging on dishonesty.

So:

1. Just because a reference material producer is doubly accredited it does not mean that their product is a certified reference material!!
2. If a producer is not doubly accredited, their products cannot be certified reference materials!!

So, what are the "Rules" that mean a certified reference material is actually a certified reference material?

A certified reference material must be accompanied by a Certificate of Analysis that contains, at a minimum:

- Name of the material

# QUALITY MATTERS

- Producer identity and producers code for the material
- General description of the material
- Intended use
- Instructions for proper use
- Instructions for appropriate conditions of storage
- Certified property value(s), each accompanied by a statement of uncertainty
- Method(s) used to obtain property values
- Period of validity, if appropriate

Key words to look for on the Certificate supplied with a certified reference material:

- "Uncertainty Statement", this must be shown and generally appears as a Value  $\pm$  another Value, e.g.,  $105 \mu\text{g mL}^{-1} \pm 1.2 \mu\text{g mL}^{-1}$
- "Traceable", this is generally interpreted as: compared to a standard from an National Metrology Institute or to another certified reference material and must be explained
- "Homogeneity", there needs to be an explanation of how the homogeneity of the certified reference material has been developed, this is normally a component of the Uncertainty Statement
- "Stability", as with Homogeneity this needs to be detailed and this is another component of the Uncertainty Statement
- Accreditation Marks on the Certificate. The name and address of the accreditation body must be shown. Normally an accredited organisation is allowed to use Accreditation Marks from their accrediting body on literature and Certificates of Analysis.
  - Note: in the US there are several Accrediting Bodies. In all other countries there is a single National Accrediting Body, for example UKAS in the UK.

It is also important to examine the reference material producer's Scope, which lists the tests/methods/technologies and type of reference materials that an organisation is accredited to produce and test. Each Accrediting Body maintains on their website a list of accredited organisations and their associated Scopes. Unfortunately,

some accreditation bodies offer this information only in their national language. Most reference material producers also maintain access to their Scopes on their individual websites. In summary:

- A reference material produced and tested outside of an accredited organisation's Scope cannot be legitimately called a certified reference material.
- Accreditation marks may not legitimately appear on the associated Certificate of Analysis for out of Scope tests and reference materials.
- The ISO standards require that all tests and reference materials that are not within Scope be clearly distinguished.

It is also important to read the wording on a Certificate carefully, and particularly marketing and other commercial literature. If a product is claimed to be tested in an ISO 17025 laboratory but without any mention of ISO Guide 34 it is not a certified reference material.

Also, look out for claims that the product is tested "according to ISO 17025" and manufactured "according to ISO Guide 34", but this is not supported by any accreditation marks or evidence of accreditation on any documentation. Such a product will not be a certified reference material.

- Anybody can say this and they might actually be doing it.
- However, YOUR accrediting body will not accept this as legitimate.

In conclusion, not all certified reference materials are created equal. A certified reference material is only as good as the accompanying documentation: Certificate of Analysis (COA). There is a lot of variation amongst accredited producers. Some do a great job, some not so good.

So, to be certain you are not called out by your accreditation body auditor on their next visit you must evaluate each certified reference material based on:

- Intended use
- Completeness of documentation
- Clarity of documentation
- Company reputation

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# Representative mass reduction in the laboratory: riffle splitting galore (with or without errors)

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While this series is presenting the universal principles behind representative sampling of all types of lots and composition, the focus has studiously been kept outside the analytical laboratory. This is because many are of the opinion that applying the Theory of Sampling (TOS) at all such large(r) scales (primary sampling) is different from the work thought to belong the analytical realm, which indeed takes place at much smaller scales. However, if the systematics of TOS shall be in a position to be used to its full power and reach, this division needs careful attention—it is time to enter THE LAB (see also the last column of 2016<sup>1</sup>).

## Introduction

Truth be told, for the many operations falling under the term “sample processing” or “sample preparation”, very nearly all contain straight-forward sampling processes—only **writ small**, but *bona fide* TOS operations nevertheless. As is shown below, it pays well to follow TOS’ universal application scope all the way to its ultimate stage, that of selecting (sampling for) the analytical aliquot (the analytical mass). It is very advantageous to view all sampling operations, spanning the entire “from lot-to-analysis” pathway, as a scale-invariant theatre; in which the sampling operations are identical, in principle as well as in practice. It is indeed **only** the scale that varies. Thus a spatula – is a laboratory spoon – is a shovel – is a spade – is a backhoe grabber – is a crane grabber... All these tools are used to select and extricate an increment, or a sample, it is only the **scale** that varies. The choice of which sampling tool dimension to choose is only related to the lot size vs the desired increment size, all of which is strongly related to the grain size characteristics of the lot. The objective of collecting an increment, or several, is in practice always

related to only two possible objectives: to perform grab sampling **or** composite sampling (see previous columns). The last column of 2016<sup>1</sup> dealt with some of these systematics in detail from the perspective of a particularly popular tool, the sampling spear, or the sampling thief. Following directly this avenue, the present column deals exclusively with the by far most often used method for mass reduction in the lab—riffle splitting.

## Riffle splitting

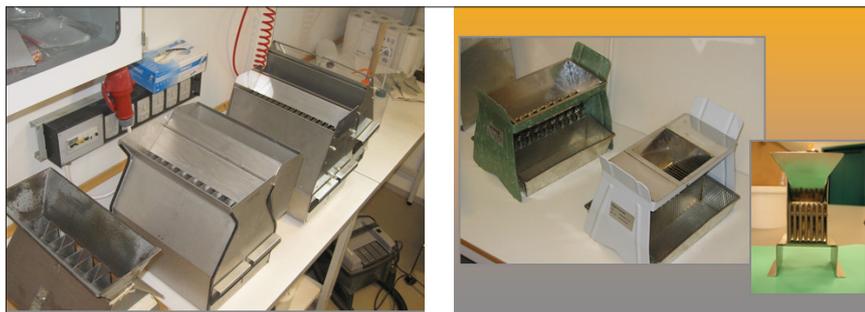
There are a few requirements in order for riffle splitting to be the perfect way to do mass reduction in the lab, by which is meant the most effective way to obtain representative mass reduction in the lab. The sample material must be free-flowing in order to be able to pass through the riffles, driven by gravity. Other than that, there are obvious requirements related to the largest particle size (in some less frequent cases also related to the sorting of the material). In general, it is obvious whether a target material is suitable for riffle splitting or not. It is the largest particle size that determines the operative requirements of the riffle splitters. A well-known rule of thumb is

that the individual riffle opening must be three times the largest particle diameter +  $\epsilon$ , in order to prevent all possibilities of *clogging* a riffle chute. With these few requirements in place, riffle splitting is completely *scale-invariant*, and one may pick the splitter tool that fits the practical and logistical conditions and requirements best, see Figure 1.

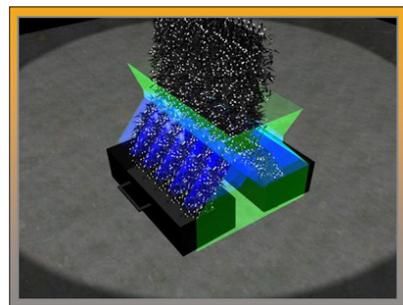
The riffle splitting principle can be implemented in a great variety of scales and ways, and realised in a wide range of tools, but the principle behind all is intuitively simple and easy to comprehend: the objective is to split an incoming mass into two *equal* sub-samples both with respect to mass and (which is decidedly most important) with respect to the analyte concentration to be found in each. There also exist variations aimed at different splitting ratios, see further below. The universal riffle splitting principle is illustrated in Figure 2.

Perhaps surprising, it is fully possible to conduct riffle splitting in a non-representative manner. Thus there are rules governing riffle splitting if this is to be representative. Below is illustrated some of these as but a first foray into the subject. For complete coverage of

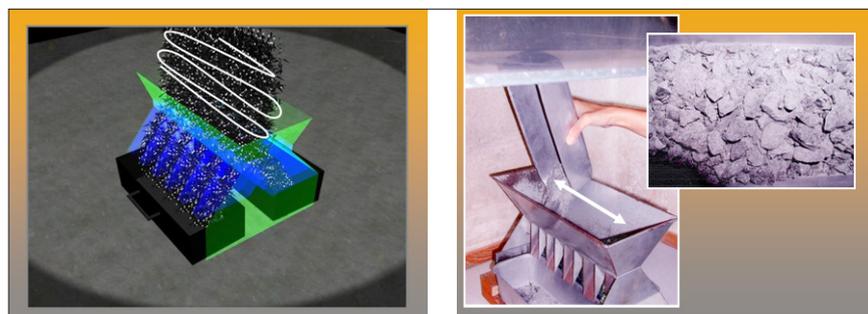
# SAMPLING COLUMN



**Figure 1.** Size does not matter. Riffle splitters are available in a large range of sizes, determined by the effective opening of the riffles (chutes). The smallest met with so far is illustrated on the far right, managing to compress 14 juxtaposed chutes along a linear distance of only 5 cm. The resulting chute width is just about the smallest opening that can accommodate very fine grained aggregate material and powders without a serious danger of clogging. So riffle splitters smaller than this are not relevant, and other ways must be found (other tools) that manage to do sub-sampling in a fashion that achieves the same purpose.



**Figure 2.** The universal riffle splitting principle: a collimated stream of matter is split by a series of juxtaposed riffles (chutes) leading to a number of slices of the stream into two alternative sub-sample reservoirs.



**Figure 3.** Longitudinal loading of the ingoing sample to be split is often an area of major misunderstanding. This “covering all chutes evenly” operation may well seem fair and reasonable at first sight, which upon scrutiny is revealed to be based on a faulty, undocumented, indeed unjustified assumption that the material in the loading tray is fully homogenous. As is very well known from TOS (see all previous columns), this never occurs in the world of science, technology and industry, and will always result in an unnecessarily inflated TSE.<sup>4</sup>

this critically important curriculum, see Petersen *et al.*<sup>2</sup> or Pitard,<sup>3</sup> but these basic

issues are also covered in many of the background TOS literature references,

see, for example, in the standard DS 3077.<sup>4</sup>

There is always a danger of that *some* of the component particles may accidentally bounce and rebound upon contact with chute walls etc. and thus may, accidentally, be propelled out of the active chute splitting zone. Such components are *lost* from the splitting products, i.e. an Incorrect Extraction Error (IEE) has been committed (by a structural Incorrect Delineation Error, IDE). Figure 5 illustrates why riffle splitters always must be closed or encapsulated. This is not too much to demand from any manufacturer.

In the right-hand image of Figure 5, a serious effort has been made to prepare the loading tray so as to deliver all the material along the longitudinal splitter axis in a controlled, even fashion (see also Figure 6). This, combined with the

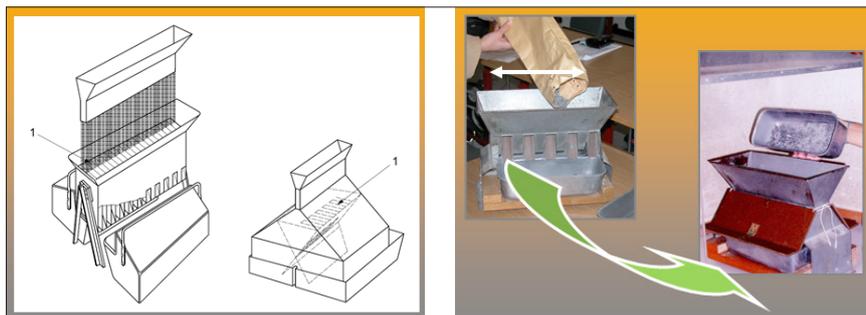
### Riffle Splitters

- The sample is fed to a (preferably large) number of chutes leading to two separate reservoirs collecting (ideally) 50 % of the material each.
- The sample must be spread evenly over all chutes
- Chutes must be equal in number, every second leading to each reservoir
- The chutes must be equal in size and shape

- Equal number of equally sized chutes
- Feeding tray has the right size
- Equal number of equally sized chutes
- Feeding tray is too wide (both sides)
- Unequal number of equally sized chutes
- Feeding tray has the right size
- Equal number of equally sized chutes
- Feeding tray is too wide (one side)

**Figure 4.** Left: riffle splitter design principles that must be observed. Right: the many ways to break these rules (most often unknowingly). However, it is the easiest thing to become an expert in all matters riffle-splitting.<sup>2-4</sup>

# SAMPLING COLUMN



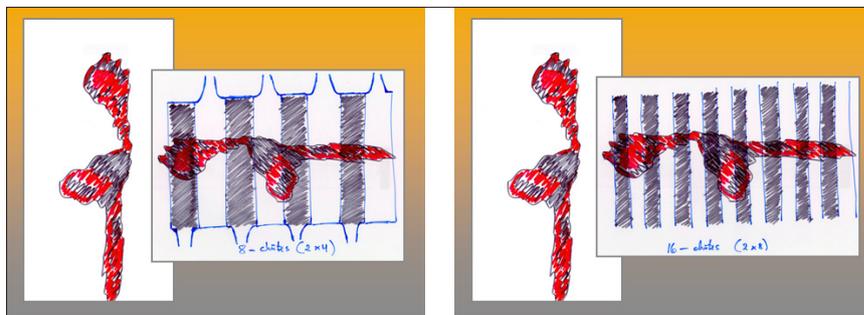
**Figure 5.** Left: how the closed equipment requirement can be easily realised, or **not**. Right: how the misunderstood “covering” loading is replaced by a carefully prepared loading tray being used so as to deliver all the material along the longitudinal splitter axis simultaneously in a controlled, even fashion. There has also been a serious effort to mix the material in the tray thoroughly before loading. All such operations help!



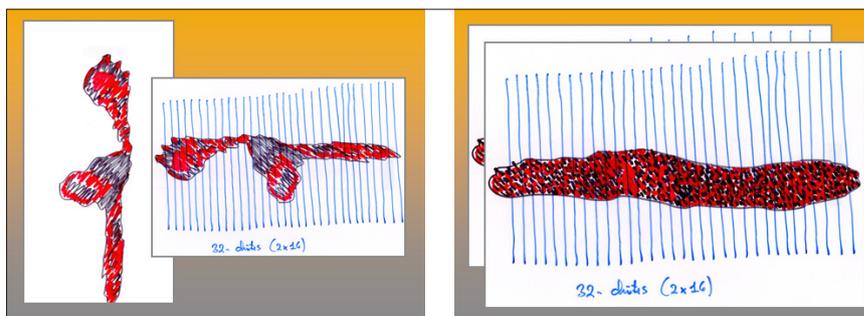
**Figure 6.** Albeit using only primitive and simplistic prototypes, in this analytical laboratory, the riffle splitting operations shown do everything correctly, indeed in a representative fashion: no IDE, IEE because of correct pre-loading mixing, correct loading, using a correct enclosed splitter. And should there be a residual dust fraction escaping the operations (hopefully reduced to the absolute fit-for-purpose level), a plexiglass hood has been installed (operated by a powerful exhaust fan) taking care of health risks to workers.



**Figure 7.** More efficient crushing leads to a much more uniform material in the loading tray, especially when combined with a conscious effort for better mixing as well.



**Figure 8.** Illustration of a very sloppily prepared, extremely inhomogeneous material laid up in a loading tray (red/black), and subjected to an increasing number of active splitting chutes.



**Figure 9.** Left: illustration of a very sloppily prepared, extremely inhomogeneous material in a loading tray (red/black) subjected to a very high number of active splitting chutes. Right: an identical number of chutes, splitting the same material that has been subjected to **proper** crushing and mixing unit operations; this also increases the splitting efficiency significantly.

Figure 7 shows how a modest improved comminution results in a significantly improved loading tray material constitution, much better suited for improved splitting efficiency.

It is in the *interaction* between optimised material constitution and the number of riffle chutes brought to bear that riffle splitting mass reduction really comes to the fore—with a significantly improved (i.e. reduced) TSE. In Figures 8 and 9, a very heterogeneous, very unevenly distributed material (almost a caricature) in the loading tray (red/black)

critical effort to mix the material in the tray thoroughly, results in subsequent sampling (splitting) procedures with a significant reduction in both Incorrect as well as Correct Sampling Errors (ISE, CSE).

The above precautions can always be observed, it is only a matter of TOS meeting with GLP, so, many, repetitive mass reduction operations can be carried out even in quite extensive scales with only small efforts (there is always relevant equipment to be had).

As an example with great carrying-over effect: more thorough crushing is a very effective sampling unit operation that can be used with significant effect.

# SAMPLING COLUMN

is subjected to a series of different splitting chutes (8, 16, 32), making it obvious that an increased number of chutes always offers better sub-sampling, everything else being equal. The last illustration shows the situation in which the most effective splitter (32 chutes) is brought to bear on a much improved material constitution (much better crushed and very well mixed material). The essential feature is that it is the exact same material subjected to four very different riffle splitting operations. There is absolutely no doubt that when crushing, mixing and effective riffle splitting are brought together with a well-considered, TOS-informed plan pertaining to the material characteristics at hand, the largest reduction in TSE can be obtained almost at no extra effort.

Observe how proper riffle splitting (using, say,  $G$  chutes) acts like a very through composite sampling—each of the two identical sub-samples were constructed by aggregating  $G/2$  increments covering the entire lot (the ingoing load sample). This composite sampling effect is not always recognised.

Figures 8 and 9 clearly illustrate the advantages obtainable when calling in three of the four Sampling Unit Operations (SUO) in their right order (crushing, mixing, composite sampling) leading to the most efficient (least TSE) mass reduction possible in the analytical laboratory. Compare this to the plethora of sub-optimal, indeed often fatal, applications of grab sampling which can be observed in many of the world's laboratories in which the spatula still rules (see also the last Sampling Column of 2016<sup>1</sup>).

## Automation—enter the rotary divider

If not already, at one time or other, the advantages of using riffle splitters for effective TOS-correct mass reduction will become obvious, indeed pressing. All the necessary, but repetitive, manual work will at first be a blessing because of the dramatically reduced TSE involved. Soon, however, *all* this work will begin to look like a burden—"if only this work could be automated...".

Well, no problem: enter the *rotary divider*. Rotary dividers act and function precisely *like* a riffle splitter, in fact they *are* riffle splitters through and through, only designed for a much more efficient *throughput*. Figure 10 shows two versions of the rotary divider, one with fixed opening widths for the number of chutes chosen (32), and one with a variable chute width for the number of chutes chosen (12). For both there are now no limitations regarding the weight of the sample to be loaded, because any (large) sample mass can be loaded in successive parts without changing the sum-total splitting operation; this is a huge advantage both for the high-throughput laboratory as well as with respect to on-line process implementation. Both the rotary dividers shown here operate on the basis of the same framework with a loading hopper and a rotating nozzle that delivers a steady stream of material hitting the splitting chutes which are arranged in a circular fashion.

By carefully balancing the loading flux in relation to the rotating nozzle speed it is simple to arrange for the sample mass

to be split and distributed over a very large number of chutes; every new 360° turn of the nozzle allows the stream flux to be distributed over a new multiple of the fixed number of chutes (here 32, 64, 96 ...); the number of operative chutes multiplication factor is staggering, making rotary dividers very much more efficient compared to their stationary, linear cousins. There are many other advantages associated with rotary dividers, see References 2–4.

It is fair to say that many other types of implementation of the same rotary splitting principle can be found; some of these will be covered when this column turns its attention to process sampling.

## Benchmark study

There are an almost infinite set of variations on the theme of laboratory mass reduction approaches and methods, which type of equipment to use etc. Upon scrutiny and reflection, however, there are only a limited number of *types* of procedural approaches: grab sampling (spatula, spoon etc.), riffle splitting (linear, rotary), coning-and-quartering... A little systematics will clear the way for clear appreciation.

Figure 11 presents a graphical overview of the gamut of what is being used today in science, technology and industry laboratories—starting with grab sampling, i.e. using *one* extraction to get the analytical mass directly (TOS: obviously fatally wrong if/when homogeneity has not been documented beyond reasonable doubt), via "shovelling methods" with various fractional shov-



**Figure 10.** The rotary divider—the ultimate mass reduction equipment. Many variants of this principal solution can be automated.

# SAMPLING COLUMN



**Figure 11.** Overview of the principally different mass reduction methods and typical types of equipment in the authoritative benchmark study by Petersen *et al.*<sup>2</sup>

elling ratios (akin to simplistic composite sampling), to the well-known “spoon method” (used extensively in the seed industry) and the “Boerner divider” (a well-nigh brilliant invention from the same realm, Figure 12), to linear as well as rotary dividers.

And then there is *coning-and-quartering*, which turns out to be the world’s most misunderstood combination of inferior mixing followed by a fatal four-riffle splitting—to be avoided at all costs. Coning-and-quartering (C&Q) was treated in full detail in a paper that could have had the title: “Why we killed C&Q and why it had it coming”—but which has a more scientifically acceptable title (with the exact same content, however), see Reference 5 for the full story.

At the other extreme is the “Boerner divider”, which is named after its designer, Herr Professor Doktor Boerner (no doubt as to the nationality of its inventor). The functional principle is gravity driven, azimuthal cone-dispersion, sectorial chute splitting (34 chutes) without any moving parts. The principle is sheer genius, and is illustrated briefly in Figure 12. Better still “Look it up, look it up – Google it”. Not only is the design brilliant, its appearance is often also a thing of beauty (such balanced use of brass and copper).

## The ultimate method/ equipment ranking for the laboratory

The present foray through the typical objectives, methods, equipment design and means-of-operation for mass reduction in the laboratory has been swift, but manages to be comprehensive. In fact, all the principal types of mass reduction methods used in today’s laboratories in science, technology and industry are covered, as are their typical practical manifestations with a necessary focus on “how to perform wrongly” (there is so much to learn, and to learn most effectively, from *mistakes*).

Figure 13 is the summary *representativity ranking* of all methods and approaches.

From TOS’, from Referene 2, definition of representativity:  $r^2 = (\text{bias})^2 + (\text{imprecision})^2$  suffice to say that the *smaller* the  $r^2$  the *better* the sampling, i.e. the splitting approach/method/equipment! Detailed scrutiny of the plot reveals the general conclusions of this extensive benchmark:

- shovelling methods off all kinds are *unacceptable* (excessive TSE, excessive  $r^2$ );
- the riffle splitting *principle* reigns supreme, rotary over linear when possible, but both variants works



**Figure 12.** The famous “Boerner divider”, functioning exactly like a rotary divider but **without** moving parts. Every second chute leads to two separated collecting funnels (inner and outer), allowing complete separation into two identical sub-samples.

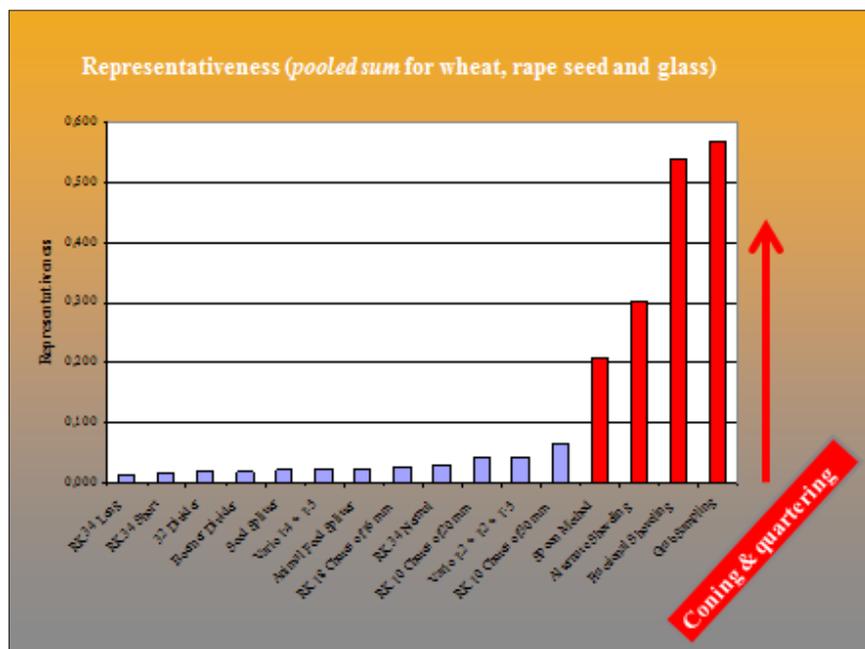
exceedingly well—critically dependent on proper eradication/reduction of all ISE, CSE);

- the “Boerner divider” is superior to pretty much anything else.

## Conclusions

So, mass reduction in the laboratory is anything but the easy matter of acquiring a piece of equipment that *claims* to be able to do a representative splitting job. Far from it: performance documentation is needed! Well there is one exception, which unfortunately cannot be applied to all types of material, but

# SAMPLING COLUMN



**Figure 13.** Ultimate representativity ranking of the 17 + 1 methods assessed in Reference 2. (The coning-and-quartering method was added after publication.<sup>5</sup>)

when this is the case, just order the Boerner divider ;-)

For all types of equipment that have passed muster in the representativity

ranking<sup>2</sup> there exists a rational set of rules that *must* be honoured in full in order for any alleged “splitter” to be representative. The most important of these have

been introduced and illustrated above. An authoritative benchmark study allows anybody to perform a comprehensive audit of the state of TOS application in the analytical laboratory, greatly recommended.<sup>2,3</sup> A severe warning is sounded about coning & quartering,<sup>5</sup> incidentally at all scales.

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continued from page 17

The first release of a technique will be an ADF format file for HPLC-UV detection planned as below:

- Allotrope Data Format (ADF) Instance Data (end Q2 2017)
- Allotrope Foundation Ontologies (AFO) Classes and Properties (end Q3 2017)
- Allotrope Data Models (ADM) Constraints (end Q4 2017)

## Conclusions

As it is clear to see, there is currently a number of overlapping well-funded alliances driving forward “standardisation” in their own interest areas. Unfortunately, much of the development paperwork is only visible to paying members or by paying for the documentation.

This issue was highlighted during a very good question and answer session at Smartlab<sup>9</sup> which included some pretty strong statements around the

lessons learnt from the failure of previous lab connectivity initiatives. This was highlighted as being due to the closed nature of those developments. The general conclusion was that in order for any standards initiative to succeed they should be “...extremely open!” It is well worth repeating that we welcome the strength of these initiatives but it is clear that if these are not to end up in the graveyard of failed “standards” more care needs to be taken that the recognised standardisation bodies are involved to ensure that multiple different uses of the same controlled terminology take place and that movement between these different formats is ensured.

This is not new—the longevity of the Adobe PDF standard—initially defended and a company confidential format has been ensured by its documentation and adoption as an ISO standard—with Adobe relinquishing control and rights to the standard.<sup>10</sup>

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## Tomas Hirschfeld Award 2017

The International Council for Near Infrared Spectroscopy (ICNIRS) are pleased to invite nominations for the 2017 Tomas Hirschfeld Award, which will be sponsored by Foss. Nominations should be sent to the ICNIRS Chair-Elect, Tom Fearn, to arrive by 28 February 2017. Full details can be found on the ICNIRS website ([www.icnirs.org](http://www.icnirs.org)).

## SIFT-MS of breath could help detect stomach and oesophageal cancers

Together, stomach and oesophageal cancer account for around 1.4 million new cancer diagnoses each year worldwide. Both tend to be diagnosed late, because the symptoms are ambiguous, meaning the five-year survival rate for these two types of cancer is only 15%. New research, reported at the European Cancer Congress 2017, has shown that a test using selected ion flow-tube mass spectrometry (SIFT-MS) could diagnose cancer with an overall accuracy of 85%. (See previous article in *Spectroscopy Europe* for background on SIFT-MS: <http://bit.ly/SIFT-MS>.)

Dr Sheraz Markar told the Congress: "At present the only way to diagnose oesophageal cancer or stomach cancer is with endoscopy. This method is expensive, invasive and has some risk of complications. A breath test could be used as a non-invasive, first-line test to reduce the number of unnecessary endoscopies. In the longer term this could also mean earlier diagnosis and treatment, and better survival."

The trial was based on the results of previous research that suggested differences in the levels of specific chemicals (butyric, pentanoic and hexanoic acids, butanal and decanal) between patients with stomach or oesophageal cancer and patients with upper gastrointestinal symptoms without cancer.

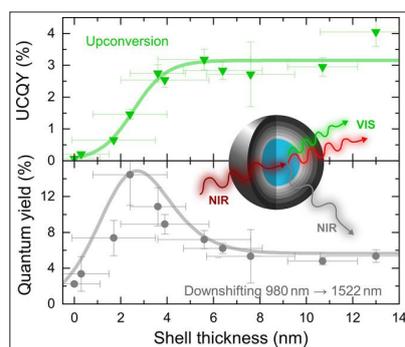
In the new study, breath samples were collected from 335 people. Of these, 163 had been diagnosed with stomach or oesophageal cancer and 172 showed no evidence of cancer when they had an endoscopy.

All the samples were analysed with SIFT-MS; researchers measured the levels of the five chemicals in each sample to see which ones matched to the "chemical signature" that indicated cancer. The results showed that the test was 85% accurate overall, with a sensitivity of 80% and a specificity of 81%.

The team is also working on breath tests for other types of cancer, such as colorectal and pancreatic, which could be used as first-line tests in general practice surgeries.

## Enhancing the luminescence of core-shell lanthanide-doped nanocrystals through surface modifications

Researchers at the University of California at Berkeley and Lawrence Berkeley National Laboratory, have demonstrated a mechanism that exploits surface quenching processes to magnify the luminescence of core-shell lanthanide-doped nanocrystals. The work has been published in *Nano Letters* (doi: [10.1021/acs.nanolett.6b03683](https://doi.org/10.1021/acs.nanolett.6b03683)). The research involved precisely tuning the shell thickness around upconversion and downconversion  $\beta$ -NaYF<sub>4</sub>:Yb,Er nanoparticles with undoped  $\beta$ -NaLuF<sub>4</sub> shells, to quantify, understand and enhance the luminescence properties associated with optimising surface quenching through spectral and lifetime analysis. Thanks to



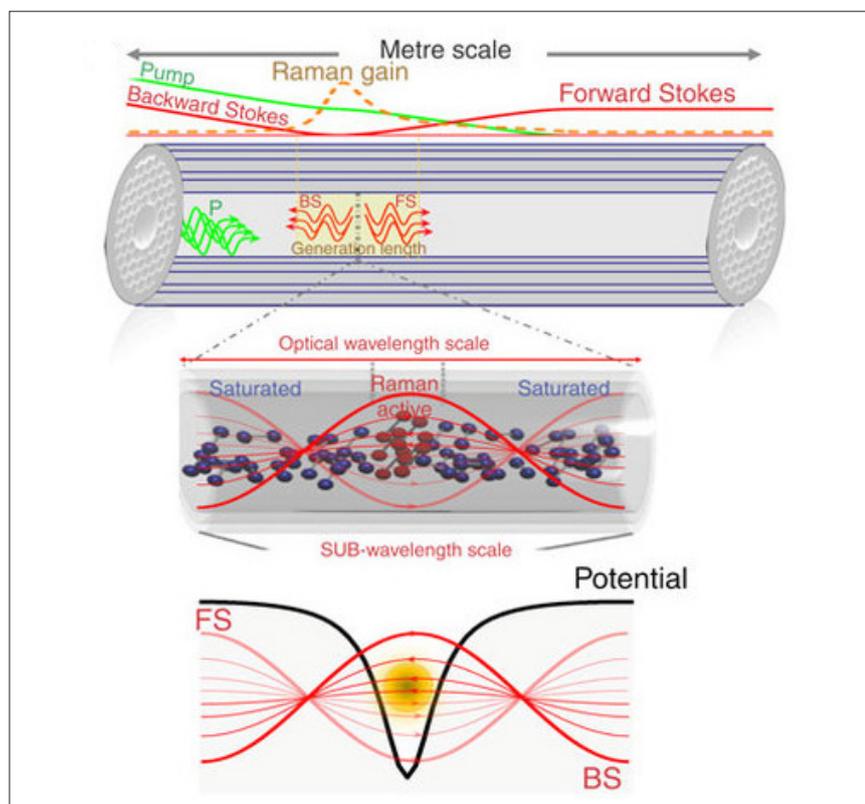
their unique ability to shift wavelengths of light to higher or lower energies based on the lanthanide dopants and crystal host, upconversion and downconversion nanocrystals have broad applications in areas such as quantum cutting, securities, biomedical imaging and novel light emitting diodes.

For this study, the researchers used an Edinburgh FLS980 luminescence spectrometer coupled to an Opotek Opolette laser to acquire spectral and lifetime properties of the enhanced upconversion and downconversion mechanism of these shell-enhanced lanthanide-doped nanocrystals. One of the authors of the paper, Dr Stefan Fischer said, "The Edinburgh FLS980 allowed us to look at the time-dependent dynamics of every single transition of Er<sup>3+</sup> and Yb<sup>3+</sup> in our nanocrystals on a very reproducible and quantitative level which eventually enabled us to derive surface quenching rates for every energy level involved in the complex up- and downconversion mechanism. This enhanced understanding of the system's dynamics allowed us to model a novel surface quenching assisted downshifting mechanism with a distinctive peak for a certain thickness of the inert shell. This distinctive peak is a result of the interplay between the different surface quenching rates which we have determined by exploiting the sensitivity of the FLS980 over a broad spectral range from the UV to the NIR."

## Lattice of nanotraps and line narrowing in Raman gas

Decreasing the emission linewidth from a molecule is one of the key aims in precision spectroscopy. One approach is based on cooling molecules to near absolute zero. An alternative way is to localise the molecules on the sub-wavelength scale. A novel approach in this direction uses a standing wave in a gas-filled hollow fibre. It creates an array of deep, nanometre-scale traps for Raman-active molecules, resulting in linewidth narrowing by a factor of 10,000.

The radiation emitted by atoms and molecules is usually spectrally broadened due to the motion of the emitters, which results in the Doppler effect. Overcoming this broadening is a difficult task, in particular for molecules. One possibility to overcome the molecular motion is by building deep potential traps with small dimensions. Previously, this was done, for example, by arranging several counter-propagating beams in a complicated setup, with limited success.



On the macroscopic scale, the pump light transforms into forward-propagating Stokes (FS) radiation, which is partially reflected from the fibre end and becomes backward-propagating Stokes radiation (BS) which is also amplified by the pump. In the region where both FS and BS are strong, they form interference pattern of standing wave, which is shown on the microscopic scale. In the low-field regions (denoted by red-colour molecules) the molecules are in the ground state and strongly trapped, as shown by the potential in the bottom panel. These trapped molecules are Raman-active, leading to line narrowing.

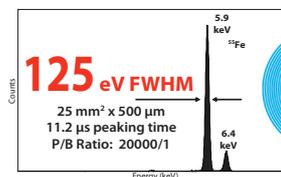
since they are close to the ground state. These low-field regions form an array of roughly 40,000 narrow, strong traps, which contain localised Raman-active molecules. The size of these traps is around 100 nm, which is much smaller than the light wavelength of 1130 nm. Therefore, the emitted Stokes sidebands have a very narrow spectral width of only 15 kHz—this is 10,000 times narrower than the Doppler-broadened sidebands for the same conditions. Details of this work have been published in *Nature Communications* (doi: [10.1038/ncomms12779](https://doi.org/10.1038/ncomms12779)).

The self-organisation of the gas manifests also on the macroscopic scale. First, the calculations show that the Raman process mainly happens exactly in the fibre section where the standing wave is formed, as shown in the top panel of the Figure. Second, the macroscopic gradient of the potential leads to the gas flow towards the fibre end, which is observed by eye in the experiment. This strong localisation and the linewidth narrowing have various uses, e.g. in spectroscopy. However, it can also be used as well as a method to periodically modulate the density of the gas, which is naturally suited for developing quasi-phase-matching schemes for other

In a cooperative effort between the Max Born Institute and Xlim Institute in Limoges, researchers show that sub-wavelength localisation and line narrowing is possible in a very simple arrangement due to self-organisation of Raman gas (molecular hydrogen) in a hollow photonic crystal fibre. Due to Raman scattering, the continuous-wave pump light transforms into the so-called Stokes sideband, which travels back and forth in the fibre due to reflections from fibre ends and forms a stationary interference pattern—a standing wave with interchanging regions of high and low field (see Figure). In the high-field regions, the Raman transition is saturated and is not active, and the molecules have high potential energy since they are partially in the excited state. In the low-field region, the molecules are Raman-active, and they have low potential energy

## Silicon Drift Detectors

### XRF Experimenter's Kit



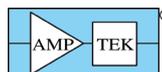
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non-linear processes, such as effective generation of high harmonics.

### Ultrahigh sensitivity graphene infrared detectors for imaging and spectroscopy

Graphene's benefits are opening possibilities in high-performance infrared (IR) imaging and spectroscopy. Researchers from the Graphene Flagship (the EU's biggest ever research initiative), have developed a graphene-based pyroelectric bolometer that detects infrared (IR) radiation by measuring tiny temperature changes with an ultra-high level of accuracy. The work, published in *Nature Communications* (doi: [10.1038/ncomms14311](https://doi.org/10.1038/ncomms14311)), demonstrates the highest reported temperature sensitivity for graphene-based uncooled thermal detectors, capable of resolving temperature changes down to a few tens of  $\mu\text{K}$ . Only a few nano-

Watts of IR radiation power are required to produce such a small temperature variation in isolated devices, about 1000 times smaller than the IR power delivered to the detector by a human hand in close proximity.

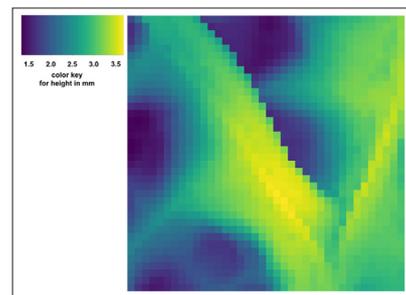
The high sensitivity of the detector is of great use for spectroscopic applications beyond thermal imaging. With a high-performance graphene-based IR detector that gives a strong signal with less incident radiation, it is possible to isolate different parts of the IR spectrum. This is of key importance in security applications, where different materials—such as explosives—can be distinguished by their characteristic IR absorption or transmission spectra.

Typical IR photodetectors operate either via the pyroelectric effect, or as bolometers, which measure changes in resistance due to heating. The graphene-based pyroelectric bolometer combines both approaches with the excellent

electrical properties of graphene, for maximum performance. Graphene acts as a built-in amplifier for the signal, removing the need for external transistors—meaning no losses from parasitic capacitance and remarkably low noise. The high conductivity of graphene also offers a convenient impedance matching with the external readout integrated circuit (ROIC) used to interface with the detector pixels and the recording device. With the continuous improvement in the quality of graphene (e.g., higher mobility), robust devices with an extended dynamic range (temperature range over which the device will operate reliably) can be fabricated while maintaining the same excellent temperature responsivity.

### Mass spectrometry imaging of non-flat surfaces

Researchers at the Max Planck Institute for Chemical Ecology in Jena, Germany, have improved mass spectrometry imaging in such a way that the distribution of molecules can also be visualised on rippled, hairy, bulgy or coarse surfaces. The source of the laser-based technique was custom-built to accommodate the topography of non-flat samples. By employing a distance sensor, a height profile of the surface is recorded before



Height profile of a piece of savoy cabbage ( $4 \times 4$  mm). The maximum difference in height is 2.38 mm. Image: Benjamin Bartels, Max Planck Institute for Chemical Ecology.

the actual chemical imaging. The new tool can be used for answering ecological questions from a new perspective, as reported in *RSC Advances* (doi: [10.1039/C6RA26854D](https://doi.org/10.1039/C6RA26854D)).

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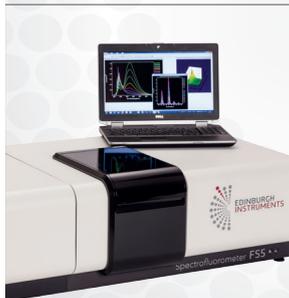
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**PRODUCT:** 20/30 PV Microspectrophotometer

**APPLICATIONS:** Materials science • Energy industry • Flat panel displays • Semiconductors

**KEY FEATURES:** UV-visible-NIR spectral range • Transmission and reflectance • Raman • Luminescence

**PRODUCT:** 508 PV Microscope Spectrometer

**APPLICATIONS:** Materials science • Energy industry • Flat panel displays • Semiconductors

**KEY FEATURES:** UV-visible-NIR spectral range • Transmission and reflectance • Luminescence

**Edinburgh  
Instruments**

Tel: +44 (0)1506 425 300  
[sales@edinst.com](mailto:sales@edinst.com)  
[www.edinst.com](http://www.edinst.com)



**PRODUCT:** FS5 Spectrofluorometer

**APPLICATIONS:** Material research • Photochemistry • Biophysics • Quantum yields • Rare earth minerals

**KEY FEATURES:** Time resolved and steady state fluorescence measurements • Single photon counting for the highest sensitivity • Two emission ports as standard • NIR upgradeability • Ultrafast data acquisition

**PRODUCT:** FLS980 Fluorescence Spectrometer

**APPLICATIONS:** Material research • Photochemistry • Biophysics • Quantum yields • Rare earth minerals

**KEY FEATURES:** Time resolved and steady state fluorescence measurements • 25,000:1 water Raman SNR • Large sample chamber with 6-axis access to your sample • High performance monochromators with high straylight rejection

**PRODUCT:** LP980 Transient Absorption Spectrometer

**APPLICATIONS:** Singlet transient absorption • Triplet transient absorption • Structural analysis • Photochemistry • Solar cells

**KEY FEATURES:** All-in-one spectrometer for transient absorption, Laser-induced fluorescence, Raman and laser-induced breakdown spectroscopy (LIBS), Kinetic and Spectral data acquisition • Transients measured up to 2.55 μm • Integrated ICCD allows transient spectra to be measured within one laser flash

**Technospex  
Pte Ltd**

Tel: (65) 6276 6928  
[weelee@technospex.com](mailto:weelee@technospex.com)  
[www.technospex.com](http://www.technospex.com)

**PRODUCT:** uSight-X

**APPLICATIONS:** Photo-luminescence •

Raman and/or fluorescence measurement and mapping  
**KEY FEATURES:** Empowers an upright microscope with laser induced luminescence micro-spectroscopy functionalities • Mapping at high spatial resolution down to (less than) 1 μm measuring spot • Allows user concurrent imaging such as fluorescence, DIC, polarisation dark field and bright field without moving the samples

The distribution of chemical compounds in flowers, leaves, stalks and other parts of a plant are of major importance in ecological research. Many of these compounds are plant secondary metabolites which are produced by plants to attract pollinators or to fend off herbivores or pathogens, for example. It is important that a plant produces these substances, but it can also be crucial where the molecules are accumulated in the plant tissue.

"The biggest challenge in analytics is preserving the constitution of a sample throughout the analytical process. More often than not, sample preparation influences the result by altering the sample's chemical constitution. Typical prepara-

tion steps include sectioning a sample into thin, flat slices because flatness is required to guarantee optimal laser focus, a key parameter in reliable analysis", Benjamin Bartels points out.

Most samples encountered in chemical ecology have surfaces which are far from flat: plant leaves often have hairy structures or they are rippled. Caterpillars can also be hairy, and they are generally rather bulgy than flat. Benjamin Bartels and Aleš Svatoš have adapted the laser ablation electrospray ionisation (LAESI) technique to non-flat surfaces to open up the possibility of performing chemical imaging of samples with pronounced three-dimensional shapes while maintaining the reliability of classical measurements.

The new instrument measures the height profile of the surface in question prior to the actual mass spectrometry imaging. The recorded height profiles can be used to correct the distance between the focusing lens of the laser and the sample's surface. In this way one of the essential parameters for reliable laser probing is kept constant throughout the experiment on samples with three-dimensional structure which were previously not subjectable to such analysis.

In the near future the researchers would like to implement further improvements and refinements. It is their goal to use LAESI for routine measurements of non-flat surfaces.

# NEW PRODUCTS

## ATOMIC

### Versatile ICP-MS for trace elemental analysis

PerkinElmer's new ICP-MS system, the NexION 2000, has been designed to handle any sample matrix, address any interference and detect any particle size. Universal Cell Technology removes interferences and there is a choice of three gas channels including the ability to run pure reactive gases such as ammonia). Intelligent dilution of samples is achieved through integrated All Matrix Solution systems and an extended dynamic range detector. The NexION 2000 also has a new RF coil, LumiCoil, that requires no water or gas cooling.

PerkinElmer

► [link.spectroscopyeurope.com/29-01-100](http://link.spectroscopyeurope.com/29-01-100)

### Arc/spark metals analyser

Spectro Analytical Instruments' new SpectroPort portable arc/spark optical emission spectroscopy metals analyser is as easy to use as a handheld analyser, whilst having many of the advantages of



The SpectroPort from Spectro, a portable arc/spark metals analyser.

their top of the range SpectroTest OES analyser. SpectroPort is as fast as a handheld XRF analyser, with many analyses taking a few seconds, and can accurately analyse C, S, P, B, Li, Be, Ca, Si, Mg and Al at low and critical levels. Its new optical system covers a wide range of elemental wavelengths. A number of options to maximise mobility are available, includ-

ing large and small transport trolleys and portable batteries; SpectroPort can also be used cordlessly with a rechargeable battery pack.

Spectro Analytical Instruments

► [link.spectroscopyeurope.com/29-01-101](http://link.spectroscopyeurope.com/29-01-101)

## IMAGING

### Miniaturised spectral sensing system

Viavi Solutions and ESPOS Photonics have worked together to produce a miniaturised spectral sensing system on a chip. This combines Viavi's 64-channel, micro-patterned bandpass filter array and ESPOS' hybrid CCD-CMOS imager to make a spectral sensor that is less than  $2.7 \times 2.7 \times 1.1$  mm in size. The sensor will be produced in two versions: visible (385–900 nm) and NIR (775–1065 nm). The size and projected cost means that the device has potential application in consumer mobile devices such as smartphones. A developer kit, the SPM64 Developer Kit, is available which includes a prototype sensor mounted on a USB dongle-style circuit board with a microcontroller and several light sources. When the board is connected via USB to a computer running the supplied software, it sends pre-processed spectral information to the GUI and optionally a data file. An API allows customers to write their own software to control the sources and sensor, and to acquire and analyse data.

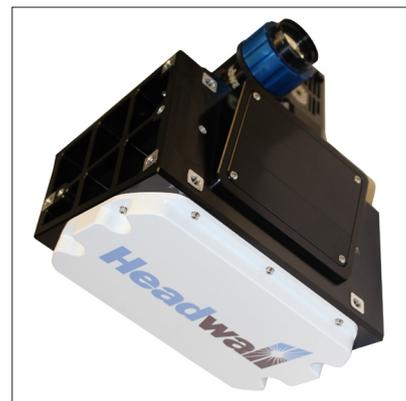
Viavi Solutions

► [link.spectroscopyeurope.com/29-01-102](http://link.spectroscopyeurope.com/29-01-102)

## LUMINESCENCE

### Chlorophyll fluorescence imaging sensor

Headwall has introduced the Hyperspec Chlorophyll Fluorescence sensor, which collects data from 670 nm to 780 nm, which allows both the important "Oxygen-A" and "Oxygen-B" bands to be measured. The sensor uses an all-reflective approach and Headwall's own precise diffraction gratings, providing high signal-to-noise performance. The sensor is small and light enough for use



Headwall's Hyperspec Chlorophyll Fluorescence sensor.

onboard commercially available UAVs, and can be used in aircraft and satellites. It weighs about 6 kg and measures  $300 \times 200 \times 200$  mm.

Headwall Photonics

► [link.spectroscopyeurope.com/29-01-103](http://link.spectroscopyeurope.com/29-01-103)

### Water treatment software

Horiba Scientific have developed new software for their Aqualog to facilitate monitoring and optimisation of water treatment processes. Aqualog analyses organics in water, measuring simultaneously both absorbance spectra and fluorescence excitation–emission matrices. The Aqualog Datastream Dashboard, which was designed with EigenVECTOR Research, integrates with Aqualog and automates the analysis and reporting of a wide range of organic matter parameters that are critical for managing and optimising the drinking water treatment process. These have been selected to target disinfection by-product issues, algal issues and other contamination components. The dashboard also displays independently uploaded data for parameters such as pH, alkalinity, turbidity and  $\text{Cl}_2$ .

Horiba Scientific

► [link.spectroscopyeurope.com/29-01-104](http://link.spectroscopyeurope.com/29-01-104)

## MASS SPEC

### LC-MS for routine analyses

Shimadzu has released a new member of its UFMS (ultra-fast mass spectrometry) family in Europe: the LCMS-8045 triple quadrupole mass spectrometer.

# NEW PRODUCTS



Shimadzu's LCMS-8045 is now available in Europe.

This has a modified ion sampling device and collision cell technology, which provides greater efficiency and high quantitative accuracy and reliability. The ion source has a cable-less, tube-less housing and the desolvation capillary can be replaced without breaking vacuum. It shares, with other UFMS members, a scan speed of  $30,000 \text{ u s}^{-1}$  without loss of mass accuracy and a polarity switching time of 5 ms. The LCMS-8045 can be upgraded, like the LCMS-8050, to the high-sensitivity LCMS-8060.

Shimadzu

▶ [link.spectroscopyeurope.com/29-01-105](http://link.spectroscopyeurope.com/29-01-105)

## High-resolution trace gas analyser

Ionicon Analytik has introduced a new compact, high-resolution VOC analyser, the PTR-TOF 4000. This uses new hexapole "Ion-Guide" technology and a novel,



Ionicon's PTR-TOF 4000 high-resolution VOC analyser.

high-resolution TOF analyser. Mass resolution is up to  $4000 \text{ m}/\Delta \text{m}$  and sensitivity is 200 cps/ppbv with a detection limit below 5 pptv.

Ionicon Analytik

▶ [link.spectroscopyeurope.com/29-01-106](http://link.spectroscopyeurope.com/29-01-106)

## QTOF for biotherapeutics

Sciex has announced the latest introduction to its X-Series quadrupole time-of-flight platform. The X500B QTOF was developed to provide ease-of-use for high-resolution standardised workflows for biotherapeutic developers, who need to characterise fully biologic products in large numbers during the development process. To simplify complex data processing, the X500B comes with BioPharmaView Software 2.0, which also offers the ability to interpret SWATH 2.0



The X500B QTOF has been developed for biotherapeutics.

data independent acquisition. SWATH acquisition allows comprehensive peptide mapping data to be acquired in a single injection, with high-resolution MS and MS/MS data for every peptide in the sample.

Sciex

▶ [link.spectroscopyeurope.com/29-01-107](http://link.spectroscopyeurope.com/29-01-107)

## Ultra high throughput screening for MALDI

Analytik Jena and Bruker Daltonics have developed a new ultra-high throughput screening product for biopharmaceutical labs. uHTS MALDI Sample Preparation is a bridge between matrix-assisted laser desorption/ionisation (MALDI) and sample preparation using automated ultra-high throughput screening (uHTS). Analytik Jena's CyBio® Well vario automated simultaneous pipettor is used

in the preparation of the MALDI plates, and Bruker Daltonics' rapiflex MALDI Pharma Pulse is used to analyse them. The throughput of the sample prep and spotting of the MALDI target is timed to produce plates on demand for input into the rapiflex MALDI Pharma Pulse, without interruption. The entire process for each 1536-well pipetting plate requires less than ten minutes. Over one million tests are now possible each week. The modular system allows customers to choose the degree of automation which best suits their application and workflow.

Analytik Jena

▶ [link.spectroscopyeurope.com/29-01-108](http://link.spectroscopyeurope.com/29-01-108)

## NEAR INFRARED

### Extended range miniature sensor

Spectral Engines has introduced a new addition to their NM Sensor Module series. The NM2.5 has an extended wavelength range to 2500 nm. These miniature sensors weigh <math><15 \text{ g}</math>, are smaller than a 1-inch cube and have only a single physical connector for simple OEM integration. It includes two light sources, collection optics and comes factory-calibrated for a wide temperature range. The NM modules are also part of Spectral Engines' cloud-based Spectral Scanner platform for industrial, portable and consumer use.

Spectral Engines

▶ [link.spectroscopyeurope.com/29-01-109](http://link.spectroscopyeurope.com/29-01-109)

## PHOTONICS

### InAsSb infrared detector

Hamamatsu has introduced an uncooled InAsSb photovoltaic detector, P13894-011MA, that offers high-speed, high-sensitivity detection in the 3–11  $\mu \text{m}$  wavelength range.

Hamamatsu

▶ [link.spectroscopyeurope.com/29-01-110](http://link.spectroscopyeurope.com/29-01-110)

### Deep-cooled CCD camera

Horiba Scientific have added the Sincerity VUV to their family of deep-cooled scientific CCD cameras. It is

# NEW PRODUCTS

sensitive in the far ultraviolet, with 58% quantum efficiency at 120 nm without an anti-reflective coating. It is available in a standard format of 2048 × 70 pixels, with special formats of 2048 × 256, 2048 × 512 and 1024 × 128 as options. The Sincerity can be deep TE-cooled to –50°C or to –30°C with N<sub>2</sub> purge, and offers low dark current and low noise. The TE-cooled CCD vacuum camera version includes a Viton-sealed flange for compatibility with high vacuum chambers. The CCDs can be supplied with an anti-reflective coating for 115–200 nm or without the coating for 135–1100 nm. The camera can also come with or without a magnesium fluoride window.

*Horiba Scientific*

▶ [link.spectroscopyeurope.com/29-01-111](http://link.spectroscopyeurope.com/29-01-111)

## Laser-driven light source

Energetiq Technology has introduced the fifth generation of their ultra-bright, laser-driven light source (LDLS), the EQ-77. This has a compact lamp house with a wavelength range from 170 nm to >2000 nm, and a choice of dual-beam output or single-beam output with a retro-reflector.

*Energetiq Technology*

▶ [link.spectroscopyeurope.com/29-01-112](http://link.spectroscopyeurope.com/29-01-112)

## Flexible picosecond diode lasers

Omicron has introduced the QuixX series of compact laser modules with integrated driver electronics, high-precision temperature regulation and beam shaping optics. The laser can emit pulses down to 50 ps with user-adjustable pulse shape as well as continuous wave in combination with fast analogue modulation and digital gating/shuttering in the MHz range. The QuixX modules can be integrated into new or existing applications via RS-232 and USB-2.0 interfaces. Control software is included.

*Omicron Laser*

▶ [link.spectroscopyeurope.com/29-01-113](http://link.spectroscopyeurope.com/29-01-113)

## Single mode 1064 nm laser diodes

Laser Components has introduced 1064 nm single-mode laser diodes with output powers of 200 mW or 300 mW from a single emitter chip,

which exhibit typical line widths of 0.5 nm (FWHM). Single mode chips are also available at wavelengths from 785 nm to 1064 nm. Packaging options include a 9 mm TO can or C2 2.1 mm chip on sub-mount package; others are available on request.

*Laser Components*

▶ [link.spectroscopyeurope.com/29-01-114](http://link.spectroscopyeurope.com/29-01-114)

## RAMAN

### Particle module for Raman microscopes

Horiba Scientific has introduced a new version of their ParticleFinder module for their LabSpec 6 Spectroscopy Suite, which works with all their Raman microscopes. The workflow-inspired interface has been improved, offering a step-by-step process to locate particles, generate size/shape statistics, select specific particles based on size/shape parameters and acquire their Raman spectra. Particle classification now uses the full capabilities of LabSpec 6. Univariate and multivariate analysis, as well as the KnowItAll database, allow users easily to classify each particle according to its spectral fingerprint. The chemical ID of particles is easily visualised with a colour code versus the Raman ID. Three types of Raman spectral acquisition on a targeted particle are possible. Single spectrum at the centre of the particle, average spectrum and full mapping.

*Horiba Scientific*

▶ [link.spectroscopyeurope.com/29-01-115](http://link.spectroscopyeurope.com/29-01-115)

### Remote functionality for B&W handheld spectrometer

B&W Tek has released the latest version of TOS Remote, which provides remote operation for their TacticID product line of handheld Raman spectrometers. The new version adds GPS mapping functionality and picture taking capabilities, both of which enhances the evidence available.

*B&W Tek*

▶ [link.spectroscopyeurope.com/29-01-116](http://link.spectroscopyeurope.com/29-01-116)

## X-RAY

### High speed, low noise camera

Princeton Instruments' SOPHIA-XO:2048 is a high-speed, ultra-low-noise camera with a 2048 × 2048, 15 μm<sup>2</sup> pixel CCD, which provides >3fps with 16 MHz read-out speed at full resolution. New proprietary ArcTec ultra-deep-cooling technology minimises dark noise by thermoelectrically cooling the CCD to less than –90°C using only air assist. A back-illuminated CCD without an anti-reflective coat-



The SOPHIA-XO:2048 camera from Princeton Instruments.

ing is used to allow direct detection of the widest range of X-rays (~10 eV to 30 keV). A 6" rotatable ConFlat flange with a high-vacuum seal design provides interfacing to UHV instrumentation.

*Princeton Instruments*

▶ [link.spectroscopyeurope.com/29-01-117](http://link.spectroscopyeurope.com/29-01-117)

### Energy dispersive spectrometer

EDAX has added a new Octane Elect energy dispersive spectroscopy (EDS) system to its existing line of EDS instruments. The new instrument has increased functionality and higher resolution than the existing Element system and sits below the Octane Elite. It can be paired with electron backscatter diffraction (EBSD) analysis as part of the integrated Pegasus EDS-EBSD Analysis System, and includes an EDS detector with a manual slide. All EDAX EDS systems include high-resolution silicon drift detectors.

*EDAX*

▶ [link.spectroscopyeurope.com/29-01-118](http://link.spectroscopyeurope.com/29-01-118)

## Conferences 2017

2–7 April, Vitoria, Espirito Santo, Brazil. **14<sup>th</sup> Rio Symposium on Atomic Spectrometry.** Maria Tereza Weitzel Dias Cameiro Lima, Laboratorio de Espectrometria Atomica, LEA, DQUL/CCE/UFES, ✉ [riosymposium2017@ufes.br](mailto:riosymposium2017@ufes.br), 🌐 [www.riosymposium.com](http://www.riosymposium.com).

2–6 April, San Francisco, California, USA. **253<sup>rd</sup> American Chemical Society National Meeting & Exposition.** Department of Meetings, American Chemical Society, 1155 16<sup>th</sup> St, NW, Washington, DC 20036-4899, ✉ [natlmmts@acs.org](mailto:natlmmts@acs.org), 🌐 <https://www.acs.org/content/acs/en.html>.

3–5 April, Dresden, Germany. **XXVII International EPR Seminar.** Evgenia Dmitrieva, IFW Dresden, Helmholtzstrasse 20, 01069, Dresden, Germany. ✉ [EPR27@ifw-dresden.de](mailto:EPR27@ifw-dresden.de), 🌐 <https://www.ifw-dresden.de/de/institute/institut-fuer-festkoerperforschung/events/xxvii-international-epr-seminar/>.

4–5 April, Barcelona, Spain. **Fifth Paperless Lab Academy (PLA).** Peter J. Boogaard, ✉ [peterboogaard@industrialabautomation.com](mailto:peterboogaard@industrialabautomation.com), 🌐 [www.paperlesslabacademy.com](http://www.paperlesslabacademy.com).

23–28 April, Vienna, Austria. **European Geosciences Union (EGU) General Assembly 2017.** EGU Executive Office, Luisenstr. 37, 80333, Munich, Germany, ✉ [secretariat@egu.eu](mailto:secretariat@egu.eu), 🌐 [www.egu2017.eu](http://www.egu2017.eu).

7–11 May, Konstanz, Germany. **15<sup>th</sup> European Workshop on Modern Developments and Applications in Microbeam Analysis (EMAS 2017) and 7<sup>th</sup> Meeting of the International Union of Microbeam Analysis Societies (IUMAS VII).** Mike Matthews, ✉ [matthm@hotmail.com](mailto:matthm@hotmail.com), 🌐 <http://www.microbeamanalysis.org/emas-2017>.

8–12 May, Faro, Algarve, Portugal. **III International Conference on Applications of Optics and Photonics (AOP 2017).** Eduarda Jesus, Viagens Abreu, S.A., Av. Da República, 124, 8000-079 Faro, Portugal, ✉ [eduarda.jesus@abreu.pt](mailto:eduarda.jesus@abreu.pt), 🌐 <http://www.optica.pt/aop2017/>.

10–12 May, Potsdam, Germany. **4<sup>th</sup> European Conference on Process Analytics and Control Technology (EuroPACT 2017).** Nina Weingärtner, DECHEMA e.V., Theodor-Heuss-Allee 25, 60486 Frankfurt am Main, Germany, ✉ [weingaertner@dechema.de](mailto:weingaertner@dechema.de), 🌐 <http://dechema.de/europact17.html>.

10–11 May, Parma, Italy. **Food Integrity Conference FI2017.** Michele Suman, Starhotel Du Parc, viale Piacenza 12/C, Parma, Italy, ✉ [info@foodintegrity2017-parma.eu](mailto:info@foodintegrity2017-parma.eu), 🌐 [www.foodintegrity2017-parma.eu](http://www.foodintegrity2017-parma.eu).

19–20 May, Kyoto, Japan. **17<sup>th</sup> Symposium on Molecular Spectroscopy.** Yosuhiko Ohshima, ToykoTech, ✉ [ohshima@chem.titech.ac.jp](mailto:ohshima@chem.titech.ac.jp), 🌐 <http://regulus.mtrtl1.info.hiroshima-cu.ac.jp/~molspec/e-index.html>.

22–26 May, Strasbourg, France. **European Materials Research Society (E-MRS) 2017 Spring Meeting.** ✉ [mrs@european-mrs.com](mailto:mrs@european-mrs.com), 🌐 <http://www.european-mrs.com/meetings/2017-spring-meeting>.

29 May–2 June, Vilnius, Lithuania. **4<sup>th</sup> International Conference on Environmental Radioactivity.** Galina Lujanienė, Center for Physical Sciences and Technology, Vilnius, Lithuania, ✉ [vilnius.lujanienė@ar.fi.it](mailto:vilnius.lujanienė@ar.fi.it), 🌐 <http://envira2017.ftmc.it>.

4–8 June, Indianapolis, Indiana, USA. **65<sup>th</sup> ASMS Conference on Mass Spectrometry and Allied Topics.** ASMS, 2019 Galisteo St., Bldg. 1-1, Santa Fe, NM 87505, USA, ✉ [office@asms.org](mailto:office@asms.org), 🌐 <http://www.asms.org>.

5–7 June, Jerusalem, Israel. **Mediterranean Conference on the Applications of the Mössbauer Effect (MECAME 2017).** 🌐 <http://www.medc.dicp.ac.cn/conference/mecame/>.

5–9 June, Bari, Italy. **2<sup>nd</sup> International Conference on Applied Mineralogy & Advanced Materials.** AMAM-ICAM 2017, ✉ [info@amam-icam2017.org](mailto:info@amam-icam2017.org), 🌐 [www.amam-icam2017.org](http://www.amam-icam2017.org).

7–10 June, Toulouse, France. **14<sup>th</sup> International Symposium on Applied Bioinorganic Chemistry (ISABC).** ✉ [isabc2017@sciencesconf.org](mailto:isabc2017@sciencesconf.org), 🌐 <https://isabc2017.sciencesconf.org>.

11–16 June, Pisa, Italy. **9<sup>th</sup> Euro-Mediterranean Symposium LIBS.** Secretary, Applied and Laser Spectroscopy Laboratory, Area della ricerca CNR, 56124, Pisa, Italy, ✉ [info@emslibs.org](mailto:info@emslibs.org), 🌐 [www.emslibs.org](http://www.emslibs.org).

11–16 June, Victoria, Canada. **9<sup>th</sup> International Conference on Advanced Vibrational Spectroscopy (ICAVS-9).** 🌐 <http://www.icavs.org>.

11–16 June, Pisa, Italy. **Colloquium Spectroscopicum Internationale XL (CSI 2017).** Alessandro D'Ulivo, UOS of Pisa, Area Della Ricerca CNR, via G Moruzzi, 56134 Pisa, Italy, ✉ [segreteria-pi@pi.iccom.cnr.it](mailto:segreteria-pi@pi.iccom.cnr.it), 🌐 <http://www.csi-conference.org>.

11–15 June, Copenhagen, Denmark. **18<sup>th</sup> International Conference on Near Infrared Spectroscopy (ICNIRS 2017).** ✉ [icnirs2017@mci-group.com](mailto:icnirs2017@mci-group.com), 🌐 <http://icnirs2017.com>.

11–15 June, Dresden, Germany. **9<sup>th</sup> International Symposium on Modern Principles of Air Monitoring and Biomonitoring, AIRMON 2017.** Secretariat, Institut für Arbeitsschutz der Deutschen Gesetzlichen Unfallversicherung (IFA), 53757, Sankt Augustin, Germany, ✉ [airmon2017@dguv.de](mailto:airmon2017@dguv.de), 🌐 <http://www.dguv.de/ifa/veranstaltungen/airmon-2017>.

19–23 June, Québec city, Québec, Canada. **61<sup>st</sup> International Conference on Analytical Sciences and Spectroscopy (ICASS).** Diane Beauchemin, Queens University, Dept Chemistry, 90 Bader Ln, Kingston, ON K7L 3N6, Canada. ✉ [diane.beauchemin@chem.queensu.ca](mailto:diane.beauchemin@chem.queensu.ca), 🌐 <http://www.csass.org/ICASS.html>.

19–23 June, Quebec, Quebec, Canada. **Spect'Atom 2017.** Patrick Belanger, Institut National de Sante Publique du Quebec, Sainte-Foy, Quebec, G1V 5B3, Canada. ✉ [patrick.belanger@inspq.qc.ca](mailto:patrick.belanger@inspq.qc.ca), 🌐 [www.csass.org](http://www.csass.org).

19–23 June, Champaign-Urbana, Illinois, USA. **72<sup>nd</sup> International Symposium on Molecular Spectroscopy.** 🌐 <http://isms.illinois.edu>.

19–22 June, Naantali, Finland. **15<sup>th</sup> Scandinavian Symposium on Chemometrics (SSC15).** 🌐 <http://ssc15.jimdo.com>.

24–27 June, Waterloo, Ontario, Canada. **Advances in Stable Isotope Techniques and Applications Conference—ASITA2017.** A. Richar Heemskerck, Environmental Isotope Lab., Dept of Earth and Environmental Sciences, University of Waterloo, Waterloo, ON N2L 3G1, Canada. ✉ [ASITA2017@uwaterloo.ca](mailto:ASITA2017@uwaterloo.ca).

26–29 June, St. Petersburg, Russia. **16<sup>th</sup> International Symposium of Trace Elements in Man and Animals.** Alex Tinkov, General Secretary, ✉ [tinkov.a.a@gmail.com](mailto:tinkov.a.a@gmail.com), 🌐 [www.tema16.org](http://www.tema16.org).

7–10 July, Victoria, Canada. **9<sup>th</sup> International Symposium on Two-Dimensional Correlation Spectroscopy (2DCOS-9).** 🌐 <http://www.icavs.org/icavs-9>.

9–14 July, São Paulo, SP, Brazil. **IUPAC2017, 46<sup>th</sup> World Chemistry Congress.** Brazilian Chemical Society, ✉ [iupac2017@sbq.org.br](mailto:iupac2017@sbq.org.br), 🌐 [www.iupac2017.org](http://www.iupac2017.org).

11–16 July, Victoria, Canada. **9<sup>th</sup> International Conference on Advanced Vibrational Spectroscopy (ICAVS-9).** 🌐 <http://www.icavs.org/icavs-9>.

23–28 July, Québec City, Canada. **20<sup>th</sup> International Society of Magnetic Resonance Conference (ISMAR 2017).** 🌐 <http://www.ismar2017.org>.

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- Medical Sciences
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- Polymers and Peptides
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- Water

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- Atomic Emission
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- MRI
- Near Infrared
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- Photonics and Optics
- Raman
- Separation Science
- Surface Analysis
- UV/Vis
- X-Ray Diffraction
- X-Ray Spectrometry
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14–17 August, Vienna, Austria. **6<sup>th</sup> International Symposium on Metallomics.** Gunda Köllensperger, ✉ [gunda.koellensperger@univie.ac.at](mailto:gunda.koellensperger@univie.ac.at), 📧 [www.metallomics2017.at](http://www.metallomics2017.at).

26–29 August, Bodrum, Turkey. **3<sup>rd</sup> International Turkish Congress on Molecular Spectroscopy (TURCMOS2017).** ✉ [info@leoncongress.com](mailto:info@leoncongress.com), 📧 <http://turcmos.com>.

28 August–1 September, Stockholm, Sweden. **19<sup>th</sup> European Conference in Analytical Chemistry.** Ulrika Örn, ✉ [ulrika.orn@kemisamfundet.se](mailto:ulrika.orn@kemisamfundet.se), 📧 <http://euroanalysis2017.se>.

31 August–2 September, Rhodes, Greece. **15<sup>th</sup> International Conference on Environmental Science and Technology.** Secretariat, University of Aegean, Voulgaroktonou Str. 30, 11472 Athens, Greece, ✉ [cest@gnest.org](mailto:cest@gnest.org), 📧 <http://cest.gnest.org/cest2017/about>.

3–8 September, Berlin, Germany. **International Conference on Ion Analysis (ICIA).** ✉ [wolfgang.frenzel@tu-berlin.de](mailto:wolfgang.frenzel@tu-berlin.de), 📧 [www.icia-conference.net](http://www.icia-conference.net).

3–8 September, Berlin, Germany. **4<sup>th</sup> International Conference on Radioecology and Environmental Radioactivity.** Laureline Fevrier, Institut de Radioprotection et de Surete Nucleaire (IRSN), ✉ [laureline.fevrier@irsn.fr](mailto:laureline.fevrier@irsn.fr), 📧 <http://www.iur-uir.org/en/conferences/id-91-4th-international-conference-on-radioecology-environmental-radioactivity-icrer>.

4–7 September, Manchester, UK. **The 38<sup>th</sup> BMSS Annual Meeting.** Royal Northern College of Music, Manchester, 📧 <http://www.bmss.org.uk/meetings.shtml>.

8–13 September, Santa Fe, New Mexico, USA. **2017 SciX Conference (formerly FACSS): Annual National Meeting of the Society for Applied Spectroscopy (SAS) / The 44<sup>th</sup> Annual North American Meeting**

**of the Federation of Analytical Chemistry and Spectroscopy Societies.** ✉ [facss@facss.org](mailto:facss@facss.org), 📧 <http://www.facss.org>.

10–14 September, Berg en Dal, Netherlands. **7<sup>th</sup> International Chemometrics Research Meeting ICRM2017.** ✉ [ICRM2017@DutchChemometricsSociety.nl](mailto:ICRM2017@DutchChemometricsSociety.nl), 📧 [www.DutchChemometricsSociety.nl/icrm-2017](http://www.DutchChemometricsSociety.nl/icrm-2017).

17–21 September, Heraklion, Crete, Greece. **10<sup>th</sup> International Conference on Instrumental Methods of Analysis.** Konstantinos Simeonidis, National Technical University of Athens, Lab. of Inorganic & Analytical Chemistry, Athens, Greece, ✉ [ima2017@chemistry.uoc.gr](mailto:ima2017@chemistry.uoc.gr), 📧 <http://www.ima2017.gr/index.htm>.

17–22 September, Thessaloniki, Greece. **European Congress and Exhibition on Advanced Materials and Processes (EUROMAT 2017).** ✉ [euromat2017@afea.gr](mailto:euromat2017@afea.gr), 📧 <http://euromat2017.fems.eu>.

25–27 September, Ulm, Germany. **14<sup>th</sup> Confocal Raman Imaging Symposium.** Dr Sonja Breuninger, ✉ [Sonja.Breuninger@WITec.de](mailto:Sonja.Breuninger@WITec.de), 📧 [www.raman.net](http://www.raman.net).

8–13 October, Reno, Nevada, USA. **44<sup>th</sup> Annual Conference of Federation of Analytical Chemistry and Spectroscopy Societies, SciX2017.** ✉ [facss@facss.org](mailto:facss@facss.org), 📧 [www.scixconference.org](http://www.scixconference.org).

## Courses 2017

13–17 March, Gembloux, Belgium. **Vibrational Spectroscopy and Chemometrics.** J Fernandez, CRA-W - Valorisation of Agricultural Products Department, Henseval building, Chausse de Namur, 24, 5030 Gembloux, Belgium, ✉ [j.fernandez@cra.wallonie.be](mailto:j.fernandez@cra.wallonie.be), 📧 <http://www.cra.wallonie.be/en/events/training-in-vibrational-spectroscopy-and-chemometrics-1>.

24–26 April, Glasgow, Scotland, UK. **FLUOROFEST 2017.** Joanne Lowy, Village Hotel Club, 7 Festival Gate, Glasgow, ✉ [joanne.lowy@horiba.com](mailto:joanne.lowy@horiba.com), 📧 [www.fluorofest.org](http://www.fluorofest.org).

10–12 May, Utrecht, Netherlands. **Multivariate Analysis of Spectroscopic Data.** Jaarbeurs Beatrixgebouw, Jaarbeursplein, Utrecht, Netherlands, 📧 <http://bit.ly/2iX3ynx>.

15 May–9 June, Copenhagen, Denmark. **Copenhagen School of Chemometrics (CSC)-2017.** José Manuel Amigo, ✉ [jmar@food.ku.dk](mailto:jmar@food.ku.dk), 📧 <https://phdcourses.ku.dk>.

22–23 May, London, UK. **Quantitative Proteomics.** University of Liverpool in London, 33 Finsbury Square, London, EC2A 1AG, ✉ [conferences@biochemistry.org](mailto:conferences@biochemistry.org), 📧 <https://www.biochemistry.org/Events/tabid/379/MeetingNo/TD015/view/Conference/Default.aspx>.

25–27 May, Austin, Texas, USA. **2<sup>nd</sup> North American Workshop on Laser Ablation.** Paul Sylvester, Department of Geosciences, Tech University, Lubbock, TX 79494-1053, USA, ✉ [paul.sylvester@ttu.edu](mailto:paul.sylvester@ttu.edu), 📧 <http://nalaworkshop.weebly.com>.

18–25 August, Dresden, Germany. **International Summer School Spectroelectrochemistry.** Evgenia Dmitrieva, IFW Dresden, Helmholtzstrasse 20, 01069, Dresden, ✉ [summerschool@ifw-dresden.de](mailto:summerschool@ifw-dresden.de), 📧 <https://www.ifw-dresden.de/institutes/iff/events/4th-summer-school-spectroelectrochemistry/>.

## Exhibitions 2017

20–23 March, Dubai, United Arab Emirates. **Arablab 2017.** Jay Domoney, ✉ [jay@arablab.com](mailto:jay@arablab.com), 📧 <http://www.arablab.com>.

11–13 April, Moscow, Russia. **Analitika 2017.** 📧 <http://www.analitikaexpo.com/en-GB>.

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