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RECENT ADVANCES IN FOOD ANALYSIS
November 5 - 8, 2019 ● Prague, Czech Republic
RAFA 2019

Wallonie recherche CRA-W

# WORKSHOP

## Vibrational spectroscopy and chemometrics:
## "The laboratory moves to the sample"

**Tuesday, November 5th, 9:00 – 13:30**


**Chaired by**

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# Agenda

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| 9:00-9:50 | **Basics of Vibrational Spectroscopy** |
| | Vincent Baeten, Walloon Agricultural Research Centre, Belgium |
| 9:50-10:40 | **Basics of Chemometrics** |
| | Juan A. Fernández Pierna, Walloon Agricultural Research Centre, Belgium |
| 10:40-11:10 | Coffee Break |
| 11:10-11:35 | **Comparison of performances of NIR hand-held devices** |
| | Olivier Minet, Walloon Agricultural Research Centre, Belgium |
| 11:35-12:00 | **Miniaturized Near-Infrared devices: application to skimmed milk powder authenticity** |
| | Yannick Weesepoel, Wageningen Food Safety Research, Netherland |
| 12:00-12:25 | **Model transferability: An interlaboratory study using SCiO devices to test oregano authenticity** |
| | Terry McGrath, Queen's University of Belfast, United Kingdom |
| 12:25-12:50 | **NIR Applications in a Food Industry: from the lab to the sample** |
| | Beatriz Carrasco Gomez, BlendHub, Spain |
| 12:50-13:30 | **Questions & Discussion** |

# Introduction

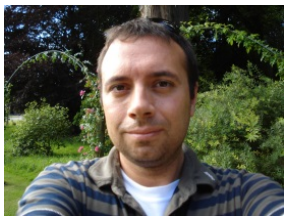
Vibrational spectroscopy, as Near infrared (NIR) or Raman, is the most widely used non-destructive technology in the food and feed industries for the daily determination and quantification of qualitative parameters of the materials. The high throughput of the method, the capacity to determine in one single analysis a panoply of parameters, the possibility to build a network of spectrometers together with its potential use both on-line and at-line in a production plant made this technique even more attractive. These techniques provide real-time analyses with an increased sample throughput. Moreover, more recent areas as hyperspectral imaging allow collection of spectroscopic images at different levels from single kernel or particle levels to satellite. This is of great interest for laboratories that control feed compound or cereals. Other decisive advantages of spectroscopic methods are the ability to determine simultaneously different parameters and criteria, no use of reagents and reduced sample preparation.

The combination of these techniques with appropriate data treatment or chemometric tools should help to solve the deep and rapid changes that the agro-food sector is facing with increasing consumer concerns about food and feed safety and quality issues. These concerns arise in part from previous safety crises (e.g. dioxin, BSE, melamine) and in part from the health impact of food and feed. The main outcome of these consumer demands is an increased need for appropriate techniques and methods to help producers, retailers and processors to control and to track their products. Infrared and Raman spectroscopy combined with chemometric should allow to build strategies that can be applied to check (on-line, at-line and at the laboratory level) the quality of food and feed materials, to detect non conformity and subsequently to identify targeted or untargeted adulterants and contaminants among others.

The practical aspects of this workshop are oriented to the use of miniaturized NIR spectrometers. The number of portable NIR devices increases day after day at the same level as their quality and applications making them an indispensable tool for food and feed control.

# Speakers

**Dr. Ir. Vincent Baeten** got his Engineer degree in Agronomy (1993) and PhD (1998) in Agricultural Sciences from the Catholic University of Louvain (Belgium). He has been awarded of a *Marie-Curie Fellowship* (1996-1998) at the Instituto de la Grasa of the CSIC (Spain). He is head of the Food and Feed Quality Unit of the Valorisation of Agricultural Products Department of Agricultural products of Walloon Agricultural Research Centre (CRA-W, Gembloux - Belgium). The Food and Feed Quality Unit is involved in the development of rapid, multi-analytes and untarget methods based on electronic and vibrational spectroscopy (Fluorescence, VIS, NIR, NIR imaging, MIR, Raman), optical microscopy and chemometrics. Vincent Baeten is deputy director of the European Union Reference Laboratory for Animal proteins in feedingstuffs (EURL-AP, http://eurlap.craw.eu). Since 2013, he is also invited professor at the Catholic University of Louvain (UCL). He has been awarded of the 2011-Q-Interline Sampling Awards for the outstanding contribution in sampling applied to spectroscopy methods. He has published more than 110 scientific papers and book chapters.



**Dr Juan Antonio FERNANDEZ PIERNA** got his Degree in physical chemistry at the University of Zaragoza, Spain in 1997. Afterwards, in 2003, he obtained his PhD in Pharmaceutical Sciences (Chemometrics) at the Analytical Chemistry department of the Vrije Universiteit Brussel (Professor D. L. Massart) with a thesis entitled "Improvements in the multivariate calibration processes". Since 2003 he works as research assistant at the CRA-W in Belgium where he has been working for the statistical treatment of the data, the application of chemometrics and the validation of methods. From end 2009, he is also responsible of the Hyperspectral Imaging laboratory installed at the Food and Feed Quality Unit. He is author or co-author of 9 book chapters and around 65 scientific papers mainly related to the statistical treatment of spectroscopic data (including homogeneity detection and uncertainty estimation), food and feed authentication and imaging techniques. He is a member of the Belgian Chemometric Society and he was and is still involved at different EU projects: STRATFEED, TYPIC, TRACE, FEED SAFETY, SAFEED-PAP, QSAFFE, FOODINTEGRITY.

**Olivier Minet** is an industrial engineer in agronomy and joined the NIR team of the CRA-W in 2010. His expertise is mainly focused in spectroscopy and chemometrics. A part of his work consists to transfer databases from one instrument to another one and to build calibrations for any types of NIR devices. He works regularly with the main software found on the market: Winisi, OPUS, Unscrambler, NIRCal, and UCal. He pilots a network of spectrometers called REQUASUD in Belgium and organizes inter-laboratory studies for different matrices: cereals, forage and food. He is also involved in different research projects and gives lessons to the annual training about NIR spectroscopy organized by the CRA-W.

## Comparison of performances of NIR hand-held devices

The number of brands and types of NIR spectrometers found on the market increases day after day, especially for miniaturized devices.

They all have in common that they measure at different frequencies the NIR absorbances of a sample. However the mode of measurement, the spectral range, the resolution, the reference used, the sample presentation accessories (e.g. probe, type of glass) and the technology (e.g. monochromator, FT, diode array, AOTF, LVF, Fabry-Perot) are making the spectral response different.

But among all these differences, the spectral range is probably the key parameter which influences the most the quality of a calibration.

Unfortunately, due to the limitation of the technology, miniaturized devices do not allow, now, to acquire the absorbances of a sample on the full classical spectral range (1100-2500nm). For the most advanced, the spectral range goes to 1650 nm but for some of them the spectral range is limited to a small area like for instance 1550-1950 nm for a certain type of spectral engine device.

To evaluate the importance of the spectral range, different tests of calibrations have been carried out on several matrixes (Forage, feed …) using the full classical spectral range and different reduced spectral range.

**Terry McGrath** is passionate about detecting food fraud. He has been working on food safety and security for more than 20 years. Terry has been a Research Fellow at the Institute for Global Food Security since graduating in 2012. He is currently researching and developing methods to allow the food industry to detect food adulteration in a way that allows them to increase efficiency and protect consumers from unsafe food. His efforts focus on detecting economically motivated adulteration using handheld spectroscopic techniques such as Near Infrared and Raman spectroscopy in conjunction with chemometric models.

## Model transferability: An interlaboratory study using SCiO devices to test oregano authenticity

Terry F McGrath[1], Simon Kelly[2], Simon A Haughey[1], Andrew Canavan[2], Christopher T Elliott[1]

The needs to radically improve ingredient supply management in highly complex supply chains, due to accidental contamination or fraud and food terrorism, has never been greater. Food fraud alone costs an estimated $US49b per year to the global food industry. As part of an EIT Food project, 'Food Fortress for raw materials and ingredients in Europe – Gaining Consumer trust through transparency of the supply chain,' chemometric models to detect oregano authenticity, developed using NIR spectra obtained on a single SCiO instrument, were deployed. An interlaboratory study was undertaken as part of this EIT Food project and an IAEA coordinated research project, 'Field-deployable Analytical Methods to Assess the Authenticity, Safety and Quality of Food'. This study, involving more than 30 participants around the world, investigated the ability of the models to correctly identify authenticity status of oregano samples. Instruments, samples and a protocol were distributed to all participants. Data will be presented showing characteristics of instruments and performance of developed models in both SCiO Lab software from Consumer Physics and Simca 15 from Sartorius Stedim Biotech.

**Yannick Weesepoel** received his PhD degree in 2014 from the Food Chemistry group of Wageningen University, where he has specialized in mass spectrometry of phytonutrients. Since 2014 he works as project manager and researcher at the group of Authenticity and Nutrients at Wageningen Food Safety Research. He specializes in miniaturized (vibrational) spectroscopics for fast screening of the authenticity of food products, next to his activities in the statutory tasks of the institute covering a wide variety of food matrices and analytical methods. He is currently participating in the core team of H2020 project 'PhasmaFood', where in collaboration with software and hardware researchers a new type of food scanner is developed with multiple sensors covering a larger variety of applications than the current Near-Infrared scanners (www.phasmafood.eu). For TKI – AgriFood he manages a private-public-project concerning the development of fast portable screening methods for authentication of fats and oils in collaboration with Dutch oil and fat industry (http://www.tki-agrifood.nl/projecten/projecten-vv/16091). He conducted numerous pilots with 'food scanner' hardware and software in collaboration with other Dutch enforcement organisations.

## Miniaturized Near-Infrared devices: application to skimmed milk powder authenticity

Miniaturized Near-Infrared devices are trending in the field of spectroscopics. During the last years many manufacturers have brought their hardware version to the market, in combination with its customized software. Prices range from €200 to over €10000 for a single scanner, as well as the wavelength range. How to make a responsible choice here when purchasing such a device? In this workshop we will use skimmed milk powders as a case for testing different miniaturized near-infrared spectrometers. All steps for evaluating and comparing spectrometers are discussed, including the application of multivariate statistics.

**Dr. Beatriz Carrasco** got her degree in Chemistry in 1994 and her PhD (1998) in Physical Chemistry from the University of Murcia (Spain). She got the Extraordinary PhD Award in Chemistry at the same University (1998). Afterwards, she got a post-doctoral fellowship at the University of Murcia. During this period, she published more than 30 scientific articles. In 2001 she started to work as Quality Consultant in the private sector and in 2004 she joined Blendhub, a multinational company who is deploying the first global network of food production hubs and offers a complete solution for designing, blending and delivering powdered food and nutrition to the industry and final consumers. After being Quality Control Manager for 4 years and Quality Assurance Manager for 7 years, she is currently the Head of the Chemometrics and Analytics unit and has specialized expertise using NIRS for the authentication of raw materials and finished products. She is working in this field since 2008 and has been carrying out some R&D projects about authentication and homogeneity of blends. Also, she has designed and developed Chemometric Brain®, a software focused on qualitative and quantitative analysis of powdered products, pure or blends, besides of homogeneity studies determining the optimal blending time of each specific blend.

## Quality and process control using NIRS in the powdered food industry

NIRS has been used in the food industry for years, but the lack of libraries about powdered ingredients and the complex mathematical methods turns its application difficult for the powdered food industry. Also, the software of most of NIR brands is not focused on qualitative analysis. Blendhub, in order to use NIRS in their factories located in different places around the world, has developed a software that enables an easy, quick and human error-free quality control process, helping their factories to perform at peak and providing a tool to detect food frauds, to authenticate products and to control the blending process. A massive database with all spectra in the cloud is shared between the factories and, in a few seconds, they get the results about the sample analysed, providing the highest quality standards, reducing risks, saving costs and enabling a faster reaction time. NIRS is also used in the process to determine the blending time needed to get the homogeneity.