



7<sup>th</sup> International Symposium on  
**RECENT ADVANCES IN FOOD ANALYSIS**

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**Workshop on**

**Vibrational spectroscopy and  
chemometrics for monitoring of  
food and feed products and  
contaminant detection**

**Tuesday, November 3<sup>rd</sup>, 9h - 13h**

Chaired by

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# Agenda

9:00h – Basics of Vibrational Spectroscopy (Dr Vincent Baeten, CRA-W, Belgium)

10:00 - Basics of Chemometrics (Dr Juan A. Fernández Pierna, CRA-W, Belgium)

11:00h – Coffee Break

11:30h – Application - Differentiation of coccidiostats-containing feed additives by Mid and Near Infra-Red Microscopy (Dr Jone Omar, JRC, Belgium)

12:00h –Application - Detection of plant and animal contaminant: advantages of spectroscopic methods (Dr Pascal Veys, CRA-W, Belgium)

12:30 – Sampling issues and industrial case contamination study – Conclusion of the workshop (Dr Vincent Baeten & Dr Juan A. Fernández Pierna, CRA-W, Belgium)

# 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.

## 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.



**Dr Jone OMAR** is performing her research activities as postdoc researcher in the Joint Research Centre – Institute for Reference Materials and Measurements (JRC-IRMM) since 2013. Her research is dedicated to the analysis of feed additives by spectroscopic techniques and to the characterisation of nanomaterials in feed additives.

#### **Differentiation of coccidiostats-containing feed additives by Mid and Near Infra-Red Microscopy**

Coccidiostats belong to the group of feed additives, which are authorised within the European Union exclusively for specific preparations. These preparations do not only contain one or more coccidiostats as active substance(s) but also various ingredients such as the carrier, which are included in the European legislation authorising the product. In order to allow the full traceability of the use of feed additives and to check for compliance with legal provisions, there is a strong need for analytical methods that enable the rapid characterisation of these products. This paper describes the applicability of non-destructive techniques such as Mid Infra-Red (MIR) and Near Infra-Red (NIR) microscopy supported by multivariate analysis for the characterisation of coccidiostats-containing feed additives. The application of these methods demonstrated that different feed additives could be distinguished from each other even when containing the same active substance. The use of chemometrics turned out to be crucial especially in cases, where the differentiation of spectra by visual inspection was very difficult.



Holder of a PhD degree in cell biology from the Catholic University of Louvain (Belgium), **Dr Pascal VEYS** is specialized in microscopy. During his thesis, he was a teaching assistant at the Laboratory of Plant Biology of the Catholic University of Louvain and serves as a consultant in microscopic techniques. At present, he is head of the microscopy laboratory at the Valorisation of Agricultural Products Department of the Walloon Agricultural Research Centre - CRA-W in Belgium. He belongs to the European Union Reference Laboratory

for Animal Proteins in feedingstuffs – EURL-AP launched in 2006 where he is in charge of the coordination of all EURL-AP activities for the 27 National Reference Laboratories of the EU. His major fields of research concern the detection of processed animal proteins in feed, toxic plants and seeds and food and feed and contaminants at large. He has been vice-chairman of the AOCS Agricultural Microscopy Division, he is also member of the association IAG Section Feedingstuff Microscopy, the European Microscopy Society and the Belgian Microscopy Society.

#### **NIR spectroscopy for disclosure of toxic or prohibited materials in feed production**

Rapid and robust methods for the detection of toxic contaminants or prohibited materials in feed remain a challenge for the industry and control authorities. Classically, such contaminations are disclosed by chemical techniques (as HPLC, GC-MS, etc.) or other methods such as light microscopy or even PCR. However as those methods shows limitations as simply by the fact that all of them are destructive, complementary analytical methods are needed. The lecture presents alternative analytical strategy for the disclosure and identification of toxic or prohibited contaminants. Examples include the detection of noxious Senecio species in hay, allergenic Ambrosia seeds in bird feed and finally the detection of processed animal proteins in compound feed by the use of rapid and non-invasive near-infrared spectroscopic methods. The advantages –and drawbacks– of the spectroscopic approach over other conventional analytical methods will be discussed.