Joint FAO/IAEA Programme
Nuclear Techniques in Food and Agriculture

Food Safety and Control Newsletter

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To Our Readers

Food on the global market. Courtesy of the Joint FAO/IAEA Centre.

In many regions of the world we seem, at last, to be emerging from the worst phases of the COVID-19 pandemic and for some of us life is returning to something like normality. There have been many lessons learned from the pandemic, not least of which is the need for greater resilience to events affecting food safety and control systems. The past two years have also highlighted the need for more flexible working practices and mechanisms to ensure that productivity and essential services can continue throughout periods of disruption. These lessons have informed our activities.

There have been two major changes in our subprogamme since the January newsletter. Firstly, I would like to welcome and introduce to you our newly appointed Laboratory Head, Christina Vlachou. Christina brings a wealth of experience and knowledge in the fields of risk assessment and food control systems from her career with the Greek authorities and interactions with various bodies on the international scene. We welcome Christina and have no doubt that she will further strengthen the laboratory. We also acknowledge, with great thanks, the role of our colleague, Simon Kelly in capably filling the Laboratory Head role pending Christina's appointment. The second change is in the name of our subprogramme. Henceforth we will be known as the Food Safety and Control (FSC) section and laboratory, rather than Food and Environmental Protection. This name change has been under discussion for some time. The new name more accurately reflects our mandate and activities as they continue to evolve to meet the needs of our colleagues in member countries. Regardless of the name, our activities have continued with enthusiasm and determination to assist and work with our Member States to provide safe and wholesome food, within the framework of the 'One Health' concept, improved market access, resilience to climate change and the ability to respond to crises affecting the food supply.

Recognizing the importance of promoting careers in science, technology, engineering and mathematics to ensure that food safety and quality is ably supported by the next generation of scientists and technical staff, and always with gender balance in mind, we were pleased to participate in Vienna's Daughters Day (Wiener Töchtertag). Staff from the FSC laboratory (FSCL) gave presentations and interactive demonstrations of our work to girls between the ages of 11 and 16 who attended the event in Vienna International Centre. An overview of the FSC sessions is provided in the feature article.

Over the past six months, we have continued to develop knowhow and transfer technology through meetings and events that are either virtual, face-to-face or in some cases a hybrid mixture combining online and in-person participation. These have included a training course held in Kampala, Uganda, on residue analysis in food crops, and a hands-on electron beam technology workshop held by the IAEA Collaborating Centre at the National Centre for Electron Beam Research at Texas A&M University, USA. Virtual training on various aspects of the detection and control of organic residues and contaminants in food was provided from the FSCL and via a regional training course for Africa.

Updates are provided in this issue of the newsletter on technical cooperation project activities to strengthen food safety testing capabilities in Honduras, on strengthening regional collaboration to address emerging food safety challenges in Latin America and the Caribbean and on a new multidisciplinary ARASIA project.

Interactions with the global food control community included presentations on nuclear techniques in food authentication at the 49th Philippine Nuclear Research Institute – Atomic Energy Week, and at the annual DASICON conference held by the Diplomatic Academy of Vienna, this year focusing on 'Sustainability, Security, and Stability: The Future of Food'. The FSC was also represented at the 18th session of the Sub-committee on Fish Trade of the the Food and Agriculture Organization of the United Nations' Committee on Fisheries, the 15th session of the Codex Alimentarius Committee on Contaminants in Food, and the 69th session of the United Nations Scientific Committee on the Effects of Atomic Radiation.

Our coordinated research activities continue, with six projects currently active, and reports of two research coordination meetings presented in this issue.

The FSCL's capabilities have been further strengthened by the acquisition of instrumentation to implement two techniques new to the laboratory: Fourier-transform near-infrared spectroscopy and surface enhanced Raman spectroscopy. Methodology has also been developed for the use of various techniques to discriminate between organic and conventionally produced food products (e.g., orange juice and spices), for geographical origin verification of palm oil, and for the use of stable isotope analysis to check provenance of starch in foods. Work is also ongoing for food contaminant control laboratory methods.

In addition to our new Laboratory Head, the FSCL is currently benefitting from the work of three new interns. Mr Vamsi Golla, from the University of Göttingen, Germany, started his internship in October 2021, and Ms Sofia Bussalino, from the University of Litoral, Neuquen, Argentina, and Mr Evans Rockson Tawiah from the Food and Drugs Authority of Ghana, both started in February 2022. These internships are of mutual benefit, providing experience and education for the interns and contributing to the laboratory's research capabilities.

I hope you enjoy this issue of the newsletter. Look out in future for the new name – Food Safety and Control!

Andrew Cannavan

Section Head,
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Feature Article

Daughters' Day Vienna International Centre Vienna, Austria

Britt Maestroni, Alina Mihailova, Sofia Bussalino

Vienna Daughters' Day is an annual event that gives an opportunity for girls, between 11 and 16 years old, who go to school in Vienna, Lower Austria, or Burgenland, to spend a day at one of about 190 companies and institutes in Vienna and get first-hand impressions of the world of work. Its focus is on technical/digital, skilled manual and scientific professions. The event in 2022 was held on 28 April. The International Atomic Energy Agency (IAEA) collaborated with the United Nations Industrial Development Organization (UNIDO) and the Comprehensive Nuclear-Test-Ban Treaty Organization (CTBTO) to participate in Daughters' Day through a joint programme of interactive sessions for the daughters of the Vienna International Centre staff.

The Food Safety and Control Laboratory (FSCL) was represented by Ms Britt Maestroni, Ms Alina Mihailova and Ms Sofia Bussalino, who delivered two 45-minute interactive sessions, posing the question "Is it really authentic and safe food?".

Presentation about the food integrity concept.

Food is an essential part of our daily lives. We all need our food to be safe and we want to be able to trust that the content of a product matches the information on its label. This is important not only to individual consumers, but for the whole food and drink supply chain. The sessions included live polls and interactive discussions on the meaning of food safety and food authenticity, quizzes on examples of common food contaminants and adulterants, presentation of some of the analytical equipment which is being used at FSCL at Seibersdorf for food safety and authenticity testing, as well as several practical sessions using two portable instruments.

The first practical session was focused on testing the authenticity of olive oil. Extra virgin olive oil (EVOO) is a high added value product that is often subject to economically motivated adulteration with cheaper and lower quality oils. The participants were presented with authentic EVOO samples, EVOO adulterated with sunflower and rapeseed oil, and fake EVOO (a blend of different oils visually resembling EVOO) and were invited to guess which they thought was the authentic sample. The participants were then shown how to test the samples with a portable near-infrared (NIR) spectrometer, connected to a tablet. This pocket size instrument was able to match the spectrum of a measured oil sample with a spectral library of the authentic olive oil and thus detect if a sample was authentic or not.

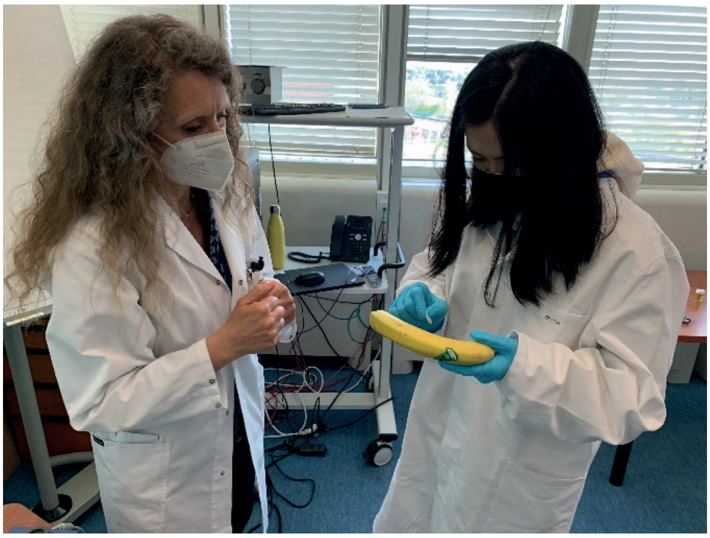
Demonstration of olive oil authenticity testing.

The second practical session focused on the application of surface-enhanced Raman spectroscopy (SERS) for the detection of aspartame in soft drinks as well as residues of the pesticide, thiabendazole, on the surface of bananas. Aspartame is an artificial sweetener used as a sugar alternative in beverage and food products. However, it presents a health hazard to individuals suffering from phenylketonuria as it is converted into the amino acid, phenylalanine, in the body. People with phenylketonuria cannot break down phenylalanine, which then builds up in their blood and brain and can lead to brain damage. Thus, this is an important food safety issue. The participants were presented with 4 different soft drinks, some of which contained aspartame. The girls were given an opportunity to learn how to use laboratory pipettes, prepare the samples for the SERS analysis and perform the measurements. The obtained results were then compared to the spectral library to determine if the soft drink samples that were tested contained aspartame or not.

The second part of the SERS demonstration allowed the girls to test for the presence of thiabendazole on the surface of organic bananas. Thiabendazole is a broad-spectrum pesticide used both as a fungicide in fruits and vegetables and for controlling parasites in animals. For bananas, which are either aerially sprayed or dipped in protectant solutions of thiabendazole, the US FDA has set a maximum residue limit (MRL) of 3 µg/g, and the EU stipulates a MRL of 6 µg/g by weight. In case of organic agriculture, the use of synthetic pesticides is prohibited, and therefore thiabendazole cannot be used to treat the surface of organic bananas. The participants were shown how to take a swab from the surface of bananas, perform the SERS measurement and detect if the bananas were treated with thiabendazole.

One of the participant is learning to use a laboratory pipette to prepare the samples for the SERS analysis.

The girls found the interactive and practical sessions very interesting. The sessions received a lot of positive feedback and helped to develop interest in this field of work, contributing to the concept of gender equality. Participation in the IAEA Daughters’ Day provided an opportunity to raise awareness in the younger generation of the importance of the safety, authenticity, and quality of food, discuss the analytical techniques used to test for food safety and authenticity in the laboratory and showcase the use of portable analytical instruments for food testing.



One of the participants is testing for the presence of thiabendazole on the surface of organic bananas using a SERS strip.

Forthcoming Events

Research Coordination Meetings and Training Courses

First Research Coordination Meeting on Nuclear Techniques to Support Risk Assessment of Biotoxins and Pathogen Detection in Food and Related Matrices (D52044- CR-1), Vienna, Austria, 22–26 August 2022.

Training Course on the Use of Rapid Profiling/Fingerprinting Techniques to Determinate Food Origin and Verify Food Authenticity, Seibersdorf, Austria, 22 August–2 September 2022.

Consultants' meeting on Rapid Screening for Safe Food Vienna, Austria, 26–30 September 2022.

Second Research Coordination Meeting on Innovating Radiation Processing of Food with Low Energy Beams from Machine Sources (D61025-CR-2), Vienna, Austria, 10–21 October 2022.

Third Research Coordination Meeting on Implementation of Nuclear Techniques for Authentication of Foods with High-Value Labelling Claims (INTACT Food) (D52042-CR-2), Vienna, Austria, 31 October–4 November 2022

International Meetings/Conferences

Second International Conference on Applications of Radiation Science and Technology (ICARST-2022), Vienna, Austria, 22–26 August 2022.

The 20th International Meeting on Radiation Processing (IMPRP20), Bangkok, Thailand 7–11 November 2022.

Past Events

18th Session of the COFI Sub-Committee on Fish Trade

Andrew Cannavan

Mr Andrew Cannavan represented IAEA as an observer at the 18th Session of the Sub-Committee on Fish Trade of the Food and Agriculture Organization of the United Nations' Committee on Fisheries (COFI). The written correspondence procedure took place between 8 April and 8 May 2022 and the virtual plenary sessions of the meeting on 7, 8, 9 and 20 June 2022.

The sub-committee covers various important aspects of fish trade, including the nutritional and food loss and waste approach to aquatic food systems, the Code of Conduct for Responsible Fisheries, and market access. Areas of possible future input by the Joint FAO/IAEA Centre, and specifically the Food Safety and Control subprogramme, fall mainly under the market access umbrella, which includes meeting Codex requirements for food safety and quality for fisheries and aquaculture products, the development of guidance for end-to-end traceability along capture fisheries and aquaculture value chains, and small-scale fisheries, trade agreements and international trade. Capacity building projects implemented by the Joint FAO/IAEA Centre through the IAEA Technical Cooperation programme could, for example, contribute to the capabilities of small-scale fisheries to meet Codex requirements for export. Analytical methodology, developed in the Food Safety and Control Laboratory at Seibersdorf and through coordinated research, for the authentication and confirmation of origin of foods, could potentially be integrated into fishery/aquaculture food quality systems and applied to verify traceability systems. The results of the recently completed coordinated research project 'Development and Strengthening of radio-analytical and complementary techniques to control residues of veterinary drugs and related chemicals in aquaculture products' will also be presented to the secretariat for potential inclusion in future sub-committee considerations.

15th Session of the Codex Committee on Contaminants in Foods

Carl Blackburn

The 15th session of the Codex Committee on Contaminants in Foods (CCCF) was held as a virtual meeting from 9 to13 May 2022 with the meeting report being finalized and agreed at a virtual session on 24 May. Mr Blackburn, representing the Joint FAO/IAEA Centre, had provided a written report and in presenting this to the committee, he verbally introduced the item and summarized the information related to nuclear and related techniques for food safety and control plus radioactivity in food.

The Food Safety and Control section is involved with many different Codex committees and at each of these a report is prepared to inform members of the activities and initiatives that will be of interest to Codex members. Representation and reporting to the CCCF is important for many reasons and over the past few years this has not only involved the many different activities that relate to the analysis of contaminants in foods using nuclear and related techniques, but it has also involved providing updates on the international work on harmonizing guidance related to radionuclides in food with pre-existing guidance for radionuclides in drinking water in non-emergency situations (so-called existing exposure situations). Regarding the latter issue, it was reported that technical work is concluding with three documents in preparation.

One document is already published online as a preprint; FAO, IAEA and WHO publication: IAEA Safety Report 114 'Exposure due to Radionuclides in Food Other Than During a Nuclear or Radiological Emergency. Part 1: Technical Material'. This pre-print includes information on the observed distributions of concentrations of key natural radionuclides in various foods, the use of dietary surveys to assess ingestion doses arising from exposure to radionuclides, and it also gives information on radionuclide concentrations in natural mineral waters, in aquaculture and in other foods collected from the wild.

A part 2 document is also in press and should soon be available as an IAEA TECDOC. The TECDOC builds on the technical material in Safety Report 114 by putting forward proposals that competent authorities could use to implement radiation safety standards as they relate to radioactivity in food in existing exposure situations.

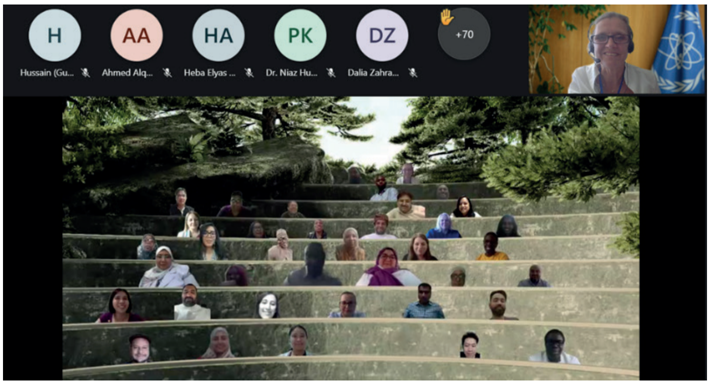
The third document in preparation is the informative document that is intended to be a concise document that might be helpful to food safety authorities. We are hopeful that it could be produced as a Codex information document. A draft is being produced by the FAO, through the Joint FAO/IAEA Centre and in collaboration with colleagues in the IAEA and WHO. This will be circulated by the Codex Secretariat for Codex member comments before a final document on naturally occurring radionuclides in food, feed and water can be presented to the CCCF for its approval at a future meeting.

Virtual Training Course on Analytical Methods to Detect and Control Organic Contaminants in Food

Britt Maestroni, Sofia Bussalino, Shuichi Nakaya,
Marivil Islam, Simon Kelly

Due to continued restrictions imposed by the ongoing COVID-19 pandemic, and to maintain assistance to Member

States, a FAO/IAEA training course on ‘Analytical Methods to Detect and Control Organic Contaminants in Food’ was implemented virtually from 16 to 27 May 2022. The course was attended by 134 participants from Bangladesh, Belarus, Bolivia, Chile, Côte d'Ivoire, Egypt, Kenya, Latvia, Mauritius, Montenegro, Myanmar, Nigeria, Oman, Pakistan, Paraguay, Romania, Saudi Arabia, Senegal, Singapore, Sri lanka, Sudan, Thailand, Tunisia, Turkmenistan, and Uruguay.



Some of the participants of the virtual training course on analytical methods to detect and control organic contaminants in food attending the introduction session on Monday 16 May 2022.

The training was provided under the Peaceful Uses Initiative project, funded by the Japanese Government, “Enhancing Capacity in Member States for Rapid Response to Food Safety Incidents and Emergencies”, as a course on theoretical concepts and laboratory methods and procedures for the detection and control of organic contaminants and residues in food. This training was intended to impart knowledge on the general principles and application of selected analytical techniques and to provide participants with the ability to transfer the techniques and methods to their own environment and infrastructure. Several external experts (Ms N. Besil, Ms S. Lazzaro, Ms V. Cesio, Mr K. Banejeree, Mr Al Gharibeth, Mr M. Zeledon, Mr H. Heinzen, Ms Caroline Tomaszewski and Ms Louise Jank) and FSCL collaborators helped to provide expertise and new training materials for this course in the form of presentations and videos. The methods and laboratory demonstration videos involved the use of equipment and instrumentation from a variety of different manufacturers, and various software applications for instrument operation, data acquisition and processing and statistical analysis. The training focused on an overview of organic contaminants in foods, a detailed description of sample preparation procedure, a thorough description of advanced analytical instrumentation such as liquid and gas chromatography coupled to tandem mass spectrometry and high-resolution mass spectrometry, including options for the analysis of polar analytes, planning of experiments, method development and validation, quality control measures, data analysis and reporting. Finally, examples of fourteen analytical methods or workflows were provided to demonstrate the stable isotope dilution assay and analysis of pesticide and veterinary drug residues, mycotoxins, marine toxins, and polar compounds such as glyphosate and metabolites in various food commodities. The training course material is avaible online at the IAEA Nucleus portal and on the IAEA Share Point Pages.

The participants of the training course had two weeks to access the presentations and video materials, before being invited to participate in live question and answer sessions, to clarify any details in the training materials and receive advice from the experts and collaborators. Although there were limited opportunities for interaction with the FSCL staff or the external experts providing demonstrations, the participants rated the question and answer sessions as very valuable. On Monday 30th of May the participants were invited to provide feedback to the course and to take a short test to demonstrate their basic knowledge in the topics of the training course.

Parallel to the training materials, the IAEA offered daily, recorded webinars, but these were not considered part of the training course. These webinars were meant to broaden the food safety aspects discussed in the training course with specific discussions about food control systems on topics such as legislative aspects, risk analysis and food integrity concepts.

Demonstrating laboratory operations and the application of instrumental methods of analysis via E-learning, without in-person real-time interaction, was a new challenge for the FSCL. The results of the feedback exercise will be presented in the next edition of this newsletter.

The 69th Session of the United Nations Scientific Committee on the Effects of Atomic Radiation

Carl Blackburn

The United Nations Scientific Committee on the Effects of Atomic Radiation (UNSCEAR) met in Vienna from 9 to 13 May. This 69th session was held as a hybrid meeting with participation in-person and via the internet. The Joint FAO/IAEA participated as observers with nominations from both the Food and Agriculture Organization of the United Nations (FAO) and the International Atomic Energy Agency (IAEA).

UNSCEAR was established in 1955 by the General Assembly of the United Nations and given its mandate to assess and report levels and effects of exposure to ionizing radiation. Governments and organizations throughout the world rely on the Committee's estimates as the scientific basis for evaluating radiation risk and for establishing protective measures.

More than 180 scientists from 31 Member States of the UNSCEAR, and representatives from 13 international organizations participated at this session. The Joint FAO/IAEA Centre is very interested in the committee’s work to evaluate public exposure due to ionizing radiation from natural and other sources because this includes an evaluation of exposure to ionising radiation from radionuclides in foods. Other areas of UNSCEAR work include considering medical and occupational exposures, and related technical issues such as epidemiological studies of radiation and cancer. Significant progress has been made by the Committee since the 68th session. This has included the publication of three annexes of the UNSCEAR 2020/2021 Report, and the endorsed new strategy for data collection, analysis, and dissemination to support the Committee's future evaluations on medical, occupational and public exposures. The strategy aims to seek widespread participation in the Committee's surveys to improve regional representation in future reports on medical, occupational, and public exposures to ionizing radiation. More information is available on the new website (www.unscear.com).

Work is continuing on second primary cancer after radiotherapy, epidemiological studies of radiation and cancer, diseases of the circulatory system from radiation exposure and on the evaluation of public exposure to ionizing radiation from natural and other sources. The IAEA Director General, Rafael Grossi, and UNSCEAR, also signed a memorandum of understanding on 10 May 2022 aiming to further strengthen the ongoing collaboration between the two organizations. The Scientific Committee 70th session is scheduled for 19 to 23 June 2023 in Vienna, Austria.

Hands-On Electron Beam Technology Workshop

Carl Blackburn

The National Centre for Electron Beam Research (NCEBR) at Texas A&M University, USA, is a centre of excellence for electron beam irradiation and an IAEA Collaborating Centre. This year it resumed its activities in delivering an annual electron beam workshop, which took place from 25 to 29 April. For the past two years, COVID travel restrictions have meant that the training event was suspended.

The Director of NCEBR, Suresh Pillai, said that "There was an unprecedented level of interest with over 50 attendees representing the medical device industry, the food industry, the pet food industry, technology providers, entrepreneurs, consultants, and the US government. As well as participants from the US, the Centre also welcomed attendees from Argentina, Canada, China, Chile, Colombia, Mexico, Panama, Poland, South Africa, and Uruguay."

Suresh also reported that, "Notable in the workshop agenda this year was a full day of hands-on lab exercises demonstrating dose uniformity ratio, single/double beam and preliminary dose mapping of medical devices, food and pet food and panel discussions."

Workshop participants enjoying one of the hands-on laboratory-based exercises.

The workshop has a broad mixture of lectures and is truly hands-on, with lots of laboratory time set aside to put into practice dose mapping and electron beam irradiation techniques. It includes sessions on food, agriculture, environment, medical device and pharmaceutical sectors. In doing so it provides a deep understanding of high and low energy electron beam and X ray technology applications and principles. It is also a unique opportunity to interact with specialists and leading equipment manufacturers.

Participation in DASICON 'Sustainability, Security, and Stability: The Future of Food'

Andrew Cannavan

DASICON is a student-led, one-day annual conference held by the Diplomatic Academy in Vienna and attended by its affiliates and the general public. The latest conference was held as a virtual event on 25 February 2022 and was titled 'Sustainability, Security, and Stability: The Future of Food', with panels focusing on future developments in the food industry throughout different sectors: technology, security, digitalization, economy, society, and politics. The aim of the conference was to listen to contributions from speakers from diverse backgrounds, engage in critical discussions, and urge participants to consider food sustainability in their daily lives. Mr Andrew Cannavan, Head of the Food Safety and Control section, was invited to contribute to the conference.

The opening panel, on 'The future of food', had keynote presentations by Austrian Federal Minister for Climate Action, Environment, Energy, Mobility, and Innovation and Technology, Leonore Gewessler, and Austrian Federal Minister for Agriculture, Regions and Tourism, Elisabeth Köstinger, who provided a broad overview of the future of food, particularly in the areas of food sustainability and international security. Important issues related to climate change, technological progress in agriculture and supply chain management, were considered.

A second panel discussion focused on ‘Sustainable Development Goal 2: Zero Hunger’, with presentations by Dr Anja Appel, Director of the Coordination Office of the Austrian Bishop’s Conference (KOO) and the Director of the Diplomatische Akademie Wien, Ambassador Dr Emil Brix. Discussion focused on inequalities in access and inadequate management of the Earth’s reserves resulting in millions of people dying of hunger and the need for sustainable agriculture, using contemporary technologies and equitable distribution mechanisms. The disruption to food supply chains by the COVID-19 pandemic was also a discussion point.

The panel discussions were followed by eight breakout sessions on:

- Food security and international relations;
- Digitalization and technology in food production;
- ‘Every step counts’;
- Sustainability challenges in agricultural policy;
- The resilience of food supply chains in times of crisis;
- Farming of tomorrow;
- The economics of food: model and mechanisms;
- Is there a human right to food?

Mr Cannavan was a presenter and panelist in breakout session 2, ‘Digitalization and technology in food production’, with co-panelists Ms Linda Staib, who is responsible for sustainability at the Austrian company ALPLA, one of the world's leading companies in the field of plastic packaging solutions and recycling, and Mr Manfred Borer, co-founder of Koltiva, an agritech firm utilizing proprietary technologies and services to enhance global supply chain traceability, inclusiveness, and sustainability. Mr Cannavan discussed food fraud and the tools and modern controls to combat it, using digitized information such as data generated through the chemical analysis of foods, within digitalized systems and databases. The likelihood of an increasing role for citizen science in the future in combatting and controlling issues such as food fraud was presented. There were several interesting questions from the audience following the presentations which prompted discussion and debate.

Overall, the conference was an interesting and useful event, fostering discussion on various aspects of the future of food.

The 49th Philippine Atomic Energy Week: Presentation on Nuclear Techniques in Food Authenticity

Simon Kelly

The 49th annual Atomic Energy Week (AEW) of the Philippine Nuclear Research Institute (PNRI) was held between 6 and 10 December 2021, with the theme ‘Science and Nuclear Technology: Addressing the Challenges of Today’. Among the events that took place, AEW scheduled virtual technical sessions to overcome the unpredictable nature of travel restrictions that were in place due to the COVID-19 pandemic.

The virtual sessions were aimed at increasing awareness, understanding and interest of students and professionals in nuclear and radiation science and technology and its beneficial applications. The sessions were spread over two days and featured presentations on topics relevant to the safety and application of nuclear technologies.

Mr Simon Kelly was invited by the AEW organisers to give a presentation on ‘Nuclear Techniques in Food Authenticity’ in the special session ‘Nuclear Technology Research and Perspectives from IAEA Experts’. Mr Kelly presented some background information on economically motivated adulteration and food fraud and then went on to address how stable isotope and trace element analysis could be used to answer challenging questions around the authenticity of food origin and production ‘added-value’ labelling claims, with specific examples on beef meat and organic vegetables, respectively. Mr Kelly then answered on-line audience questions posed by the moderator Dr Asuncion-Astronomo.

Mr Simon Kelly participating in the 49th PNRI AEW special session ‘Nuclear Technology Research and Perspectives from IAEA Experts’. IAEA Colleagues Mr Syahril Syahril and Mr Petr Sladek also participated in the session.

The session also featured contributions from IAEA colleagues Mr Syahril Syahril on “IAEA Technical Cooperation in Asia and the Pacific” and Mr Petr Sladek on “Nuclear Instrumentation”. Other technical session topics, over the two days, included “Energy and industry”, “Health and Medicine”, “Environment and Agriculture” and “Nuclear Safety, Security, and Safeguards, Radiation Protection and Radioactive Waste Management”. The conference was well attended, and over 450 on-line participants took part.

Forensic Epidemiology and Impact of Substandard and Falsified Antimicrobials on Public Health (FORESFA) – First Consortium Meeting

Simon Kelly

Substandard and falsified (SF) antimicrobials are a massive but underappreciated global health challenge in need of innovative research to inform interventions. Professor Paul Newton, Head of the University of Oxford's Medicine Quality Research Group, is leading a Wellcome Trust Collaborative Award project to establish an innovative, multidisciplinary research hub that will improve understanding and inform global policy and action on SF medicines. The project consortium is composed of leading specialists investigating illegal wildlife trade, forensic genomics and isotope chemistry, social network analysis and modelling. The collaborators will work together to answer two principal research questions. The first is, how can novel genomic ('pharmabiome'), chemical and isotopic analysis with social network techniques be used to characterise the origins of SF antimicrobials and their constituents to inform action to improve global pharmaceutical supply quality? This will be addressed through high-throughput sequencing and novel chemical and isotopic analysis of falsified and authentic antimicrobials to determine their comparative pharmabiome/isotopic/chemical 'fingerprints'. In addition, social network analysis will be performed to investigate origins and trade routes from accessible reports. The second question to be addressed is, what are the modelled impacts of SF antimicrobials on patient outcome, global public health, especially stimulating antimicrobial resistance and how can these be minimised? Furthermore, using a One Health approach, which pathogen-antimicrobial pairs are at greatest risk of SF antimicrobials?

The IAEA's Food Safety and Control Laboratory (FSCL) is currently negotiating the terms of a collaboration agreement with Oxford University to participate in the FORESFA project. Mr Simon Kelly was invited to present the potential contribution FSCL could make, at the first virtual consortium meeting from 13 to 15 December 2021, based on its expertise in tracing the origin of food and food components when applied to excipient materials in SF medicines (e.g., corn starch, potato starch, lactose, soya lecithin, Xanthan gum, magnesium stearate and cellulose) to uncover trade routes for SF medicines. It is already known that food grade materials are often used in SF medicines instead of pharmacopeial grade products due to their lower cost and wider availability. Mr Kelly presented two novel stable isotope methods to support characterisation of the geographical origin of maize starch excipients through measurement of non-exchangeable hydrogen isotope ratios; 1) trifluoroaceate derivatisation of saccharides measured by compound-specific gas chromatography isotope ratio mass spectrometry (GC-IRMS) analysis and 2) perchlorate-cerate oxidation of sugars to calcium formate, measured by elemental analysis-IRMS. He also presented energy dispersive X ray fluorescence spectroscopy to screen for toxic heavy metals which may occur in the raw materials of active pharmaceutical ingredients (APIs) and/or excipient materials and inferring origin through trace element patterns and chemometrics. Finally, he presented the potential use of headspace ion mobility spectrometry to develop a library of medicine packaging atmospheres and volatile organic carbon signatures in sealed tablet blisters, that may aid in elucidating specific production processes, packaging types and origins. The other project participants involved in laboratory analysis gave presentations on the use carbon, nitrogen and oxygen stable isotope analysis of the APIs and excipients (Fondazione Edmund Mach, IT); and genomics to characterise the fragments of environmental DNA found within SF medicines; and direct analysis in real time – mass spectrometry (DART-MS) to characterise fake pharmaceuticals and give clues as to their origin (University of Edinburgh, UK). Currently, repositories of authentic and seized SF medicines are being compiled, with the help of pharmaceutical companies, medical and enforcement agencies in Southeast Asia and Africa, before characterisation and model building to test the reliability of the analytical procedures. The next in-person meeting of the project consortium is planned for July 2022.

Coordinated Research Projects

CRP Reference Number	Ongoing CRPs	Project Officer
D52040	Field-deployable Analytical Methods to Assess the Authenticity, Safety and Quality of Food	S. Kelly
D52041	Integrated Radiometric and Complementary Techniques for Mixed Contaminants and Residues in Foods	J.J. Sasanya
D52042	Implementation of Nuclear Techniques for Authentication of Foods with High-Value Labelling Claims (INTACT Food)	S. Kelly
D52043	Depletion of Veterinary Pharmaceuticals and Radiometric Analysis of their Residues in Animal Matrices	J.J. Sasanya
D52044	Nuclear Techniques to Support Risk Assessment of Biotoxins and Pathogen Detection in Food and Related Matrices	J.J. Sasanya
D61025	Innovating Radiation Processing of Food with Low Energy Beams from Machine Sources	C.M. Blackburn
D61026	Irradiation Technology for Phytosanitary Treatment of Food Commodities and Promotion of Trade	C.M. Blackburn

First Research Coordination Meeting on Novel Irradiation Technology for Phytosanitary Treatment of Food Commodities and Promotion of Trade (D61026)

Carl Blackburn

This first research coordination meeting (RCM) of the coordinated research project (CRP) D61026 was held from 22 to 29 March 2022 as a virtual online meeting. The RCM was attended by thirty representatives from thirteen countries (Argentina, Australia, Bangladesh, Brazil, China, Egypt, France, Indonesia, Israel, Mexico, Pakistan, USA and Viet Nam) and two international organizations (FAO and IAEA).

The purpose of the virtual meeting was to discuss and agree individual and overall work plans, and to establish outputs and timelines for the project. During the RCM the proposed research of each participant was discussed and reviewed. The meeting helped to facilitate an understanding of the relationship each project has to the overall objectives of the CRP. It also served to promote collaboration between CRP members and others.

This CRP has two overall objectives:

- To validate absorbed radiation doses proposed as generic treatments for nine key groups of pests and develop at least five generic phytosanitary irradiation treatments for adoption in the International Standards for Phytosanitary Measures of the International Plant Protection Convention;
- To innovate new treatments (by investigating factors that might affect the efficacy of irradiation) and stimulate the development of new irradiation techniques.

Since the adoption of the International Plant Protection Convention (IPPC) Guidelines for the Use of Irradiation as a Phytosanitary Measure (International Standard for Phytosanitary Measures No. 18, ISPM 18), a great deal of commercial experience has been gained to confirm the efficacy and utility of ionizing radiation.

There are currently nineteen irradiation treatments that are adopted in International Standards for Phytosanitary Measures (ISPMs) as annexes to the International Standard on Phytosanitary Treatments for Regulated Pests (ISPM 28). However, only two are generic; (ISPM 28 Annex 7) is generic for fruit flies of the family Tephritidae and Annex 39 is generic for fruit flies of the genus *Anastrepha*) but the others relate to specific pest species.

Having more internationally accepted generic treatments, where one radiation dose is broadly applicable to a wide range of different pest species, would support further up-take of the technology and increased trade in irradiated commodities.

As the RCM discussed workplans and collaborations, key collaborative groups were agreed and future CRP research activities were summarized. The ability of the CRP to deliver its stated aims was discussed as the pest species included in research plans were reviewed and the objective of each research group was considered. At the conclusion of the meeting, it was thought highly likely that CRP D61026 would be able to meet its two overall objectives.

As regards the first overall objective of the CRP, research plans indicate that absorbed radiation doses will be validated and proposed as generic treatments for at least five out of the nine key pest groups originally considered. Therefore, at least five generic phytosanitary irradiation treatments could be put forward for adoption in the International Standards for Phytosanitary Measures, as it was considered that the research will contribute significantly to the acceptance treatments as phytosanitary measures against: Leaf miners of the family Agromyzidae or the genus Liriomyza; Mealybugs of the family Pseudococcidae; Eggs and larvae of the order Lepidoptera; Pupae of the order Lepidoptera; All regulated insects except for pupa and adult Lepidoptera.

As regards the second overall objective, investigating factors that might affect the efficacy of irradiation, several groups plan to research the effect of different O_2/CO_2 atmospheres and some research will explore whether dose rate can be used to optimize efficacy of treatment doses. It was proposed that participants would participate in a dosimetry intercomparison exercise provided by Aerial-CRT an IAEA Collaborating Centre and therefore it was tentatively agreed to hold the next RCM at Aerial in France in early July 2023. It was also proposed that the third RCM in 2024 is hosted by the Chinese Academy of Inspection and Quarantine, in Beijing, China.

Second Research Coordination Meeting on Depletion of Veterinary Pharmaceuticals and Radiometric Analysis of their Residues in Animal Matrices (D52043)

James Sasanya

The second research coordination meeting (RCM) for the coordinated research project (CRP), 'Depletion of Veterinary Pharmaceuticals and Radiometric Analysis of their Residues in Animal Matrices' was held virtually from 28 February to 4 March 2022 and attended by 29 participants from Bangladesh, Brazil, Burkina Faso, Canada, Chile, China, Costa Rica, the Islamic Republic of Iran, Morocco, Pakistan, the Republic of Korea, Sudan, Uganda, USA and Uruguay as well as staff of the IAEA.

Some of the participants at the 2nd RCM for D52043.

The purpose of the meeting was to discuss work done since the first RCM; prepare new or updated workplans; and share solutions to challenges encountered.

The meeting noted that eight analytical methods have been developed or validated, with some already in use. Innovative radiolabelling of amoxicillin was performed under the project using Zn-65 for depletion work in fish and relevant data was generated. Imaging was performed using positron emission tomography and scans were taken after administration of the radiopharmaceutical by a dual-head coincidence system. Furthermore, useful data has been obtained indicating the suitability of Zn-65 for depletion studies. Locally synthesized [3H] diaveridine (DVD) was used to study the excretion of DVD in swine and broilers. Similar work was performed in laboratory animals. Non-radiolabelled depletion studies were conducted by three of the CRP research groups, with further studies and other radiolabelled experiments underway.

A publication, 'Development of radioactive tracing coupled with LC/MS-IT-TOF methodology for the discovery and identification of diaveridine metabolites in pigs' was reported. More publications are expected in due course. An opportunity to submit papers to be published as a special issue of a peer-reviewed journal was presented and a number of standard operating procedures shared to support animal experiments. It was also noted that a number of CRP members have received additional support for various procurements to facilitate their research. Nevertheless, there are still challenges with the process of receiving and delivery of radiolabelled material (especially), besides budgetary limitations. given the limited funds provided under research contracts relative to the cost of research inputs and experiments.

Technical Cooperation Projects

Country/Region	Project No.	Title	Technical Officer
Burundi	BDI5003	Strengthening National Capacities for Monitoring and Testing Veterinary Drug Residues in Food	J.J. Sasanya
Burundi	BDI5004	Enhancing Control of Chemical Residues and Related Contaminants in Food	J.J. Sasanya
Benin	BEN5013	Expanding Analytical Capabilities for Systematic Control of Veterinary Drug Residues and Related Contaminants in Foodstuff	J.J. Sasanya
Bangladesh	BGD5032	Building Capacity in Improving Food Safety Using Nuclear and Other Complementary Analytical Techniques	S.D. Kelly
Bangladesh	BGD5034	Enhancing Competence in Nuclear and Complementary Capabilities for Testing/Monitoring Veterinary Drug Residues and Other Contaminants in Foods	J.J. Sasanya A. M.V. M. Rodriguez y Baena
Bahrain	BAH5002	Establishing a National Quality Control Standard for Foodstuffs and Fishery Products	J.J. Sasanya
Bosnia and Herzegovina	BOH5003	Using Nuclear Technology in Enhancing Science Based Safety, Quality and Control Systems in Feed and Food Chains	A. Cannavan
Botswana	BOT5020	Enhancing Capabilities for a Holistic Approach to Testing Food Hazards in Poultry Production and Products	J.J. Sasanya
Botswana	BOT5023	Enhancing Control of Food Hazards in Poultry Production and Products	J.J. Sasanya
Belize	BZE5011	Strengthening Laboratory Capabilities to Monitor Contaminants in Fisheries Products	B.M. Maestroni
Cameroon	CMR5025	Improving Laboratory Testing Capabilities to Enhance the Safety and Competitiveness of Agricultural Products - Phase I	J.J. Sasanya
Chile	CHI0021	Building General Capacity for Nuclear Science and Technology Applications in Key Sectors	S.D. Kelly J.J. Sasanya
Chile	CHI5053	National Reference System for Verification of Authenticity and Determination of Origin of Food using Nuclear Isotopic Techniques	S.D. Kelly
Costa Rica	COS5037	Strengthening Capabilities to Analyse and Monitor Toxic Metals in Animal Products	J.J. Sasanya
Cuba	CUB5022	Promoting Food Safety through the Mitigation of Contaminants in Fruits for Human Consumption	C.M. Blackburn J.J. Sasanya
Djibouti	DJI5001	Developing Nuclear/Isotopic and Complementary Food Safety Testing Capabilities	J.J. Sasanya

Country/Region	Project No.	Title	Technical Officer
Dominica	DMI5002	Enhancing Capacity to Monitor Agrochemical Residues in Foods and Related Matrices	J.J. Sasanya
Dominica	DMI5003	Strengthening a Nuclear Isotopic Laboratory and Complimentary Field Food Safety Surveillance Capabilities	J.J. Sasanya
Dominican Republic	DOM5005	Strengthening National Capabilities to Ensure Food Authenticity	S.D. Kelly
Ecuador	ECU5030	Reducing Post-Harvest Losses of Native Potatoes and other Fresh Foods by Irradiation	C.M. Blackburn
Ecuador	ECU5033	Strengthening Laboratory Capacities for Monitoring Residues of Neonicotinoid Pesticides in Honey Bees and Honey	B.M. Maestroni
Eritrea	ERI5012	Developing Analytical Capabilities for Food Safety	J.J. Sasanya
Eritrea	ERI5014	Enhancing Food Safety Analytical and Monitoring Capabilities	J.J. Sasanya
Fiji	FIJ5002	Increasing Trade and Export Capacities of Selected Value Chains within the Agro-Food Sector through the Adoption of an Appropriate Quality Infrastructure	C.M. Blackburn
Fiji	FIJ5005	Establishing a Food Safety Laboratory for Analysis of Pesticide Residues in Fresh Fruits, Vegetables and Root Crops — Phase II	B.M. Maestroni J.J. Sasanya
Georgia	GEO5001	Enhancing National Programmes for Testing and Monitoring Food Contaminants and Residues	J.J. Sasanya
Haiti	HAI5009	Strengthening Laboratory Capacity to Test and Monitor Food Contaminants	J.J. Sasanya
Honduras	HON0003	Improving National Capabilities in the Use of Nuclear Technologies for the Promotion of Sustainable Development Goals	Mr P.D.M. Brisset Ms T. Jevremovic Ms I.T.Bertral J.J. Sasanya
Indonesia	INS5045	Strengthening Food Security Through Improvement of Food Safety for Exports Using Gamma Irradiators and Electron Beams	C.M. Blackburn B. S. Han
Iran, Islamic Republic of	IRA1011	Building Capacity for the Development of Stable Isotope Techniques in Medicine, the Environment, Agriculture, and Sciences	S. D. Kelly U.D. Sarvana Kumar O. Kracht J.A. Miller
Cote d'Ivoire	IVC5042	Improving Testing and Monitoring of Food Hazards Using Nuclear and Isotopic Techniques	J.J. Sasanya
Cambodia	KAM5004	Strengthening National Capability for Food and Feed Safety	J.J. Sasanya
Kazakhstan	KAZ5005	Building Capacities in Effectively Irradiating Food	C.M. Blackburn

Country/Region	Project No.	Title	Technical Officer
Kenya	KEN9007	Establishing a National Standard Laboratory for Individual Monitoring, Radioanalysis, and Calibration of Neutron and Surface Contamination Services	C.M. Blackburn J.J. Sasanya
Kyrgyzstan	KIG5001	Establishing Effective Testing and Systematic Monitoring of Residues and Food Contaminants and of Transboundary Animal Diseases	J.J. Sasanya I. Naletoski
Lebanon	LEB1010	Establishing an Isotopic Ratio Mass Spectrometry Laboratory Dedicated to Authentication and Provenance for Supporting the National Fraud Repression Scheme	S. D. Kelly M. Groening
Lebanon	LEB5016	Strengthening Capacity for Exposure Assessment of Residues and Contaminants in the National Diet	J.J. Sasanya
Lebanon	LEB5017	Strengthening Technical Capabilities by Introducing Metal Speciation Techniques to Support Health and Environmental Safety	S. D. Kelly
Lesotho	LES5011	Strengthening Nuclear and Related Food Safety Laboratory Capabilities to Control Veterinary Drug Residues and Related Contaminants	J.J. Sasanya
Madagascar	MAG5028	Developing Food Safety Laboratory Capabilities	J.J. Sasanya
North Macedonia	MAK5009	Enhancing National Capacities to Standardize Nuclear Based and Related Techniques for Food Safety and Detection of Irradiated Food	A. Cannavan B. S. Han A. Mihailova C.I. Horak
Malaysia	MAL5032	Strengthening National Capacity in Improving the Production of Rice and Fodder Crops and Authenticity of Local Honey Using Nuclear and Related Technologies	Mihailova S.D. Kelly J. J. Adu-Gyamfi E. Fulajtar C. Zorrilla
Mauritius	MAR5027	Building Capacity to Analyse Veterinary Drug Residues and Related Chemical Contaminants in Animal Products	J.J. Sasanya
Mauritania	MAU5008	Strengthening Laboratory Capacity to Analyse and Monitor Residues and Contaminants in Foods	J.J. Sasanya
Marshall Islands	MHL5002	Building Core Capacities to Control Contaminants and Other Residues in Food — Phase I	J.J. Sasanya
Mali	MLI5032	Improving Laboratory and Monitoring Capabilities for Contaminants in Cereals and Nuts	J.J. Sasanya
Mongolia	MON5024	Enhancing Food Safety Analytical Capabilities for Veterinary Drug Residues and Related Contaminants Using Isotopic Techniques	J.J. Sasanya

Country/Region	Project No.	Title	Technical Officer
Mozambique	MOZ5010	Strengthening Confirmatory Analytical Capabilities for Veterinary Drug Residues and Related Contaminants in Animal Products	J.J. Sasanya
Mozambique	MOZ5012	Enhancing Food Safety Testing and Monitoring of Hazards Using Nuclear and Related Techniques	J.J. Sasanya
Namibia	NAM5018	Strengthening Animal Health and Food Safety Control Systems	J.J. Sasanya
Namibia	NAM5019	Enhancing National Capacity for Contaminant and Adulteration Monitoring of Marine and Other Food Products for Consumer Protection	J.J. Sasanya A. Mihailova M. H. T. Metian
Nepal	NEP5007	Supporting Analysis of Pesticide Residues in Agricultural Products	B.M. Maestroni
Niger	NER5023	Strengthening Capacity of the Public Health Laboratory to Monitor Food Contaminants	J.J. Sasanya
Niger	NER5025	Improving Food and Biological Hazard Detection, Food Preservation and Mutation Breeding	J.J. Sasanya C.M. Blackburn S. Sivasankar
Vanuatu	NHE5004	Strengthening Agro-Food Laboratory Quality Infrastructure — Phase II	J.J. Sasanya
Nicaragua	NIC5012	Strengthening the Monitoring and Control System for Food Contaminants	J.J. Sasanya
Oman	OMA5008	Enhancing National Capabilities in Food Safety and Traceability	S.D. Kelly
T.T.U.T.J. of T. Palestinian A.	PAL5010	Strengthening Capability to Monitor Contaminants in Food and Related Matrices through Nuclear and Complementary Analytical Techniques	J.J. Sasanya
Panama	PAN5027	Strengthening Analytical Capabilities for Risk-based Monitoring of Agricultural Products for Internal Consumption	J.J. Sasanya
Panama	PAN5030	Strengthening Laboratory Capacity in Monitoring Veterinary Drug Residues and Contaminants in Milk and Honey Using Nuclear/Isotopic Techniques	J.J. Sasanya
Philippines	PHI5035	Advancing Laboratory Capabilities to Monitor Veterinary Drug Residues and Related Contaminants in Foods	J.J. Sasanya
Qatar	QAT5009	Enhancing National Food Safety Capacity to Test and Monitor Residues/Contaminants Using Nuclear and Related Isotopic Techniques	J.J. Sasanya C.M. Blackburn
Romania	ROM5010	Enhancing Food Safety and Quality of Consumer Protection	C.M. Blackburn J.J. Sasanya
Rwanda	RWA5003	Strengthening Laboratory Capacity of the Standards Board to Analyse and Monitor Chemicals (Veterinary Drug Residues and Related Contaminants) in Foods — Phase II	J.J. Sasanya A. M.V. M. Rodriguez y Baena

Country/Region	Project No.	Title	Technical Officer
South Africa	SAF5018	Establishing National Capacities for Monitoring and Control of Pesticide Residues in Agricultural Produce	B.M. Maestroni
Seychelles	SEY5014	Developing Toxicological Analytical Capability for Monitoring and Biomonitoring Exposure to Toxic Agents in Biological and Environmental, as well as Food and Water Matrices	J.J. Sasanya
Singapore	SIN5001	Enhancing Food Safety	C.M. Blackburn J.J. Sasanya
Sri Lanka	SRL5048	Strengthening National Capability for Food and Feed Safety	A. Cannavan A. Mihailova
Sudan	SUD5040	Strengthening the Evaluation of Quality, Monitoring and Control Programmes for Food Contaminants	J.J. Sasanya
Uganda	UGA5042	Strengthening Capabilities of Two Central Food Safety Laboratories and Selected Regional Veterinary Centres of Public Health	J.J. Sasanya
Democratic Rep. of the Congo	ZAI5028	Controlling Food and Feed Contaminants in Fish Production	J.J. Sasanya
Zambia	ZAM5032	Strengthening and Expanding Analytical Capacity to Monitor Food Contaminants using Nuclear/Isotopic and Complementary Tools	J.J. Sasanya
Africa	RAF0050	Promoting Institutional Capacity Building Through Triangular Partnerships (AFRA)	V. Gershan J.J. Sasanya
Africa	RAF5084	Strengthening Food Contaminant Monitoring and Control Systems and Enhancing Competitiveness of Agricultural Exports using Nuclear and Isotopic Techniques (AFRA)	J.J. Sasanya
Africa	RAF5088	Building Capacity for Food Irradiation by Facilitating the Commercial Application of Irradiation Technologies — Phase II (AFRA)	C.M.Blackburn B. S. Han C.I. Horak
Asia/ Pacific	RAS5081	Enhancing Food Safety and Supporting Regional Authentication of Foodstuffs through Implementation of Nuclear Techniques (RCA)	S.D. Kelly
Asia/ Pacific	RAS5087	Promoting Food Irradiation by Electron Beam and X Ray Technology to Enhance Food Safety, Security and Trade (RCA)	C.M. Blackburn
Asia/ Pacific	RAS5096	Strengthening Multi-Stakeholder Food Safety Monitoring Programmes for Chemical Contaminants and Residues in Plant and Animal Products Using Nuclear/Isotopic Techniques	J.J. Sasanya

Country/Region	Project No.	Title	Technical Officer
Asia/ Pacific	RAS5099	Developing Climate Smart Crop Production including Improvement and Enhancement of Crop Productivity, Soil and Irrigation Management, and Food Safety Using Nuclear Techniques (ARASIA)	J.J. Sasanya M. Zaman
Latin America/ Caribbean	RLA5069	Improving Pollution Management by Persistent Organic Pollutants to Reduce Impact on People and the Environment (ARCAL CXLII)	B.M. Maestroni
Latin America/ Caribbean	RLA5079	Applying Radio-Analytical and Complementary Techniques to Monitor Contaminants in Aquaculture (ARCAL CLXXI)	J.J. Sasanya
Latin America/ Caribbean	RLA5080	Strengthening the Regional Collaboration of Official Laboratories to Address Emerging Challenges for Food Safety (ARCAL CLXV)	B.M. Maestroni A. Cannavan
Latin America/ Caribbean	RLA5081	Improving Regional Testing Capabilities and Monitoring Programmes for Residues/Contaminants in Foods Using Nuclear/Isotopic and Complementary Techniques (ARCAL CLXX)	J.J. Sasanya
Latin America/ Caribbean	RLA5084	Developing Human Resources and Building Capacity of Member States in the Application of Nuclear Technology to Agriculture	J. J. Adu-Gyamfi I. Naletoski W.R.E. Hoeflich C. Zorilla J.J. Sasanya

Updates on RLA5080 ‘Strengthening the Regional Collaboration of Official Laboratories to Address Emerging Challenges for Food Safety’

Britt Maestroni, Nicola Schloegl

The UN 2030 Agenda for Sustainable Development identifies the need for sustainable food production systems and resilient agricultural practices that provide nutritious, safe, and affordable food to all consumers. To this end, tools like databases and risk assessment exercises are essential to identify and navigate the major global drivers, related trends and other issues that may emerge in the future, bringing varying impacts on food production systems, including food safety. National food safety authorities continue to develop and ensure compliance with standards, guidelines and policies to keep food supply chains safe. In this context, the access to scientific evidence is crucial to stay abreast of present and future challenges related to food safety.

Under the framework of project RLA5080, ‘Strengthening the Regional Collaboration of Official Laboratories to Address Emerging Challenges for Food Safety’ (ARCAL CLXV), national food safety authorities in the region of Latin America and the Caribbean are in the process of formalizing a Data Sharing Committee (DSC) under the Latin American Network of Analytical Laboratories (RALACA). RALACA-DSC currently comprises officially designated representatives from thirteen countries in the region.

Considering the need for reliable data, project RLA5080 was forefront in terms of establishing an official regional framework document that includes legal provisions for data exchange and data collection, including the definition of roles, responsibilities, processes for data validation and use, as well as a standardized data record sheet and information categories. Under this framework each RALACA-DSC representative has the task of coordinating the provision of national analytical data on chemical contaminants in food to a regional data repository.

In parallel to the establishment regional data-sharing framework, a data collection repository hosted by the IAEA has been identified and will serve as the RALACA-DSC database. The RALACA-DSC database will be hosted in the International Research Integration System (IRIS) which is currently being customized to meet RALACA-DSC user requirements. IRIS has a specific personal data and privacy notice that is in full compliance with the IAEA's data security requirements. IRIS also offers an intuitive user interface with a wide range of functional features including data collection in the form of Excel sheets, providing the possibility to create graphs and export data for further analyses using the Application Programming Interface (API). The latter is a software intermediary that allows connection between applications, such as a risk assessment software application or advanced statistical software. The sensitive data is encoded, to ensure data anonymity throughout the sharing process. The final RALACA-DSC database is expected to be ready for use in the second semester of 2022.

The database will be used to carry out ad hoc risk-based decision-making and policy-formulation in food safety in the LAC region. The database approach help providing a means of looking at food safety issues holistically, from a multisectoral point of view, which is inherent in a resilient food control system**,** ensuring that food is safe, wholesome, and fit for human consumption.

Virtual Training on Veterinary Drug Residue Monitoring Programmes

James Sasanya

About 100 food safety scientists and managers from 24 African countries participated in a virtual training course, held from 26 February to 6 April 2022, on the establishment and implementation of national residue monitoring programmes for veterinary drug residues in animal products. The countries included Angola, Benin, Botswana, Burundi, Cameroon, Egypt, Eritrea, Ethiopia, Ghana, Côte d'Ivoire, Kenya, Madagascar, Malawi, Mauritius, Mozambique, Namibia, Nigeria, Rwanda, Seychelles, South Africa, Sudan, Uganda, Zambia and Zimbabwe. Relevant legislation encompassing all measures needed to run a surveillance programme was covered. This aspect addressed the competent authority and service partners working as a team to design and implement a plan based on risk analysis and investigating non-compliant results. This is based on full traceability of samples from collection to reporting results, as well as the use of robust and valid analytical methods. Legislation coverage also addressed aspects of authorising sampling/inspection officials, taking samples and giving legal backing to the analytical services, giving officials powers to detain animals when further investigations are necessary and describing offences and penalties for non-compliance.

Participants also learnt about developing residue plans, including describing the criteria considered in preparing a robust surveillance plan and using information/expertise from all relevant agencies involved in coordinating and implementing the plan. Analytical requirements including a range of screening and confirmatory techniques were also covered. In this regard, recent changes in EU guidelines for the use of such methods in residue monitoring were highlighted and relevant literature provided. Given the importance of risk-based food safety management, the participants were also informed about risk sampling and matrix ranking of pharmacologically active veterinary substances (and related hazards). This requires a system set up by a team of independent experts from all aspects of the "farm to fork" chain to assess authorised and prohibited/unauthorised substances, and a range of criteria to rank them in terms of national risk. Case studies were also presented on the investigation of non-compliant test results for both authorised and unauthorised substances, and the remedial/corrective actions taken. Finally, discussions were held, and experiences shared among the participants from the 24 countries. A range of challenges was also presented, and solutions proposed.

Some participants at the training on residue monitoring.

Regional (Africa) Training on Residue Analysis for Food Crops from Supervised Field Trials

James Sasanya

Thirty scientists from Benin, Burkina Faso, Burundi, Cameroon, Egypt, Kenya, Malawi, Mali, Mauritius, Morocco, Rwanda, Senegal, South Africa, Tunisia, Uganda and Zimbabwe participated in a regional training course in Uganda, from 28 March to 1 April 2022, on analysing pesticide residues in food crops (using okra as an example) from supervised field trials. The training was hosted by the Directorate of the Government Analytical Laboratory and the Uganda National Bureau of Standards.

Prior to the training, the hosts prepared a garden and cultivated okra. Pesticide (trifloxystrobin and tabuconazole) formulations were applied, and samples collected and stored ready for analysis by the participants.

The training included: (a) multi-location supervised field trials following good agricultural practices (GAP) to support the establishment of maximum residues levels (MRLs); (b) laboratory practicals including use of liquid- and gas-chromatography-mass spectrometry to analyse pesticide residues from the trial, including reliable sample preparation; (c) a laboratory quality management system to support such studies; and (d) monitoring of pesticide residues through a systematic approach, using examples from India. Other topics covered were: (e) purity and stability of certified reference materials for pesticides (a pressing matter among several developing countries and a topic of discussion during the past two sessions for the codex committee on pesticide residues (CCPR)); (f) residue data analysis from decline studies, and how this information is reported to stakeholders, including risk managers for the purposes of establishing MRLs or taking other management decisions; (g) the process of submitting GAP data for Codex maximum residue limit (MRL) setting - this was addressed in detail, including India's experience; and finally the group also learnt about (h) bio-efficacy studies, including identification of pest/disease, selection of associated pesticides, their efficacy, cost and availability of a national or Codex MRL. The participants also undertook field trips including a visit to the okra field where the supervised field trials were performed.

Preparation of okra samples for pesticide residue analysis.

Supporting Honduras' Endeavours to Strengthen Food Safety Testing

James Sasanya

Honduras has set food safety as a top priority in an effort to esure consumer protection and enhance food exports. Relevant initiatives are ongoing and central to this is the need to strengthen laboratory capabilities.

One institution at the forefront of food safety testing, is the Laboratorio Nacional de Analisis de Residuos (LANAR) in Honduras under the administration of the International Regional Organization for Agricultural Health (OIRSA). While LANAR has a range of capabilities such as LC-MS/MS, GC-MS, GC-ECD, HPLC, and ICP-MS and conducts various tests, including for pesticide residue and toxic metals as well as microbiological tests, areas of need have been identified in analytical instrumentation, methods of analysis and human resource development. Thus, through TCPs HON003 and RLA5081, support is being provided to address some of these challenges. Upon LANAR's request, a radio receptor assay instrument and associated kits has been procured (pending delivery) to support the screening of a range of veterinary drug residues, mycotoxins and some pesticides. This is expected to ease the pressure on the confirmatory analytical instrumentation and will improve analytical turn-around time. A number of consumables and minor items of equipment have also been procured, some already delivered and put to good use.

During a recent visit to the country from 26 April to 1 May 2022 Mr Sasanya provided technical support/guidance to LANAR staff in their effort to establish four analytical methods for a range of veterinary drugs and pesticide residues. A manual of standard operating procedures and videos to demonstrate certain analytical procedures were also presented. He also assessed needs and provided recommendations with follow-up action now ongoing.

Sample management for food safety testing at LANAR.

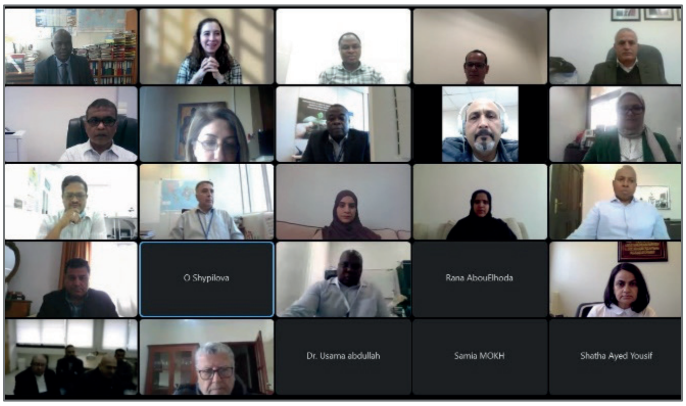
LANAR and relevant authorities in the country, including the Ministry of Agriculture, are keen on expanding the laboratory's analytical scope and instrumentation for chemical hazard analysis to cover topics such as inorganic chemistry, including toxic metal speciation, analyzing food additives, and enhancing microbiological hazard testing etc. Plans are also underway to expand the scope and number of methods accredited to ISO/IEC 17025.

Consequently, LANAR and Servicio Nacional de Sanidad Agropecuaria (SENASA) will continue to collaborate with the IAEA including initiation of a new national TCP for the 2024–2025 cycle. During Mr Sasanya's visit, he had meetings with various high level officials. There is a high level of political commitment to supporting food safety in the country, according to the Deputy Minister of Agriculture, the Honourable Jose Angel Acosta and Dr Emilio Aguilar, Director General of SENASA, among other managers.

Virtual Coordination Meeting for a New ARASIA Multidisciplinary Project (RAS5099) Encompassing Food Safety

James Sasanya

A new regional project for ARASIA, RAS5099 “Developing Climate Smart Crop Production including Improvement and Enhancement of Crop Productivity, Soil and Irrigation Management, and Food Safety Using Nuclear Techniques” involving institutions from Iraq, Jordan, Kuwait, Lebanon, Oman, Qatar, Saudi Arabia, Syrian Arab Republic, United Arab Emirates and Yemen was recently launched, with a projected duration of 4 years (2022–2025).



A session at the RAS5099 virtual coordination meeting.

ARASIA is the Cooperative Agreement for Arab States in Asia for Research, Development and Training related to Nuclear Science and Technology. ARASIA’s Programme Committee, in 2017, recommended that one of ARASIA’s main strategic cooperative thrusts would be supporting enhanced food safety and increased agricultural productivity. Food safety remains a major concern to Arab countries from public health and trade perspectives.

Under this framework, it is important to improve food safety through regional networking that produces reliable scientific data using nuclear isotopic techniques, enabling institutions to work together andconduct joint training on targeted topics; toshare experiences and benchmark good practices; and to generate data that can be used to set or influence international (Codex) trade standards to promote fair trade and safeguard consumers.

Consequently, under RAS5099, one of the outputs is that ARASIA capabilities for monitoring and testing of veterinary drug and pesticide residues in food would be strengthened. In the interest of promoting the farm-to-fork continuum, the project would explore mechanisms to improve interactions between food safety laboratories and with other sectors of agriculture such as crop production and soil and water management, as well as farmer knowledge and practices in relation to food safety and trade.

To launch this multidisciplinary project, a virtual coordination meeting was held on 24 March 2022. The meeting was attended by 25 participants, from Iraq, Jordan, Kuwait, Lebanon, Oman, Syria, Qatar and United Arab Emirates. The workplan was fine-tuned and implementation has been initiated. Prior to the meeting, a survey was conducted to gather information about laboratory and institutional capabilities and the national targets, among other topics. Findings of the survey were discussed, and this partly informed the updated workplan. The project has a number of unfunded activities and extrabudgetary donations would be welcome.

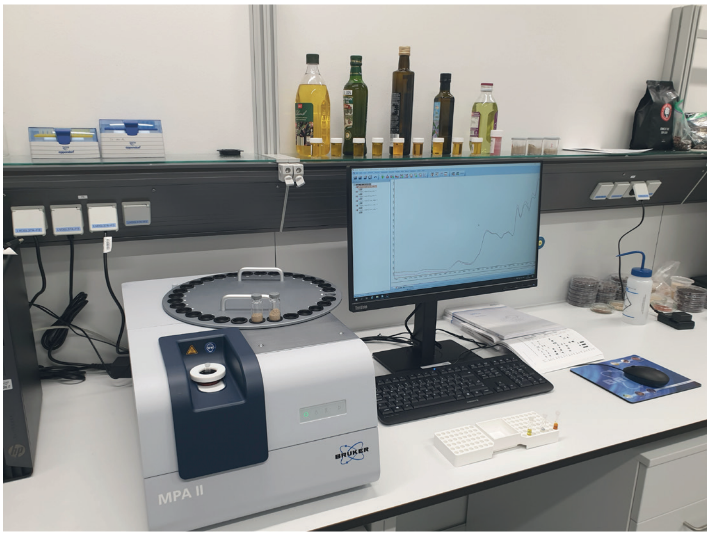
Developments at the Food Safety and Control Laboratory

Fourier-Transform Near-Infrared Spectroscopy – a New Technique for Food Safety and Authenticity Screening at FSCL

Alina Mihailova, Marivil Islam, Britt Maestroni,
Simon Kelly

A new benchtop Fourier-transform near-infrared (FT-NIR) spectrometer was commissioned at the Food Safety and Control Laboratory (FSCL) in April 2022.

FT-NIR spectroscopy is a non-destructive analytical technique which measures the absorption of light from the sample in the near-infrared region of the electromagnetic spectrum from 4000 to 12500 cm^{-1} (2500 nm to 800 nm; see Figure 1 for more detail). The recorded NIR spectrum consists of overtones and combinations of fundamental vibrations of molecules that contain C–H, O–H, and N–H bonds, which provide information about the molecular and physical structure of the sample. This makes FT-NIR spectroscopy suitable for the identification of raw materials, composition analysis, conformity testing and process monitoring.



MPA II FT-NIR spectrometer at FSCL.

The benchtop FT-NIR spectrometer installed in FSCL, A Bruker MPA II instrument, allows analysis using three types of optical measuring modes: transmission, reflection, and transflection. When measuring in transmission mode, the light is directed at a sample with a focused or parallel beam; some of this light is absorbed, but the remaining part is transmitted to the detector. This type of measurement is typically used for the analysis of clear liquids (direct transmission), however diffusely reflecting or slightly scattering samples can also be analysed in this way (diffuse transmission). Reflection mode is used for the analysis of powders, pellets or granulates. In this mode, the light is directed in a broad nearly parallel beam onto a sample and reflected from its surface.

Transflection is a combination of transmission and reflection modes. When a mirror is placed behind the sample, the light transmitted through the sample is reflected through the sample and onto the diffuse reflectance probe or integrating sphere. Transflection mode is useful for the analysis of emulsions, gels, and turbid liquids.

Training on the use of the MPA II FT-NIR spectrometer at FSCL.

FT-NIR spectroscopy has been used for a wide range of applications in the areas of chemistry, petrochemistry, pharma, food and feed, agriculture, materials science and medicine. In the area of food safety and authenticity, FT-NIR has been applied for the quality control, authentication and traceability of various commodities, e.g. edible oils, spices, milk, meat, fish, wine etc. Some examples of the food safety and authenticity applications using benchtop FT-NIR systems include the species authenticity and adulteration in crabmeat, the detection of melamine adulteration in milk powder and soya bean meal, detection of pulp wash in orange juice, the adulteration of strawberry and raspberry purees with apple, and differentiation of white wines according to grape variety.

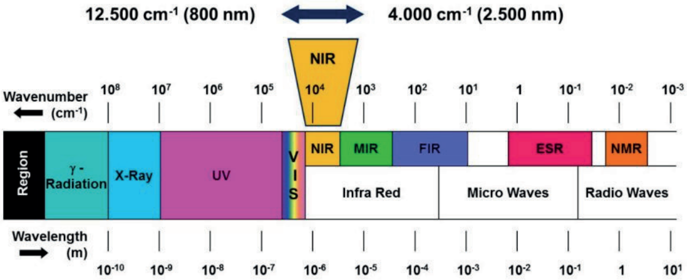


FIG. 1. The electromagnetic spectrum highlighting the NIR range (Image source: Bruker Optics GmbH).

FT-NIR spectroscopy offers multi-analyte screening capability as well as low operational costs. It requires little or no sample preparation, does not involve the use of chemicals or specialized laboratory facilities, and allows rapid analysis (10 to 60 seconds analysis time) and high sample throughput. This makes FT-NIR spectroscopy a suitable and cost-effective tier 1 analytical tool that can be used for sample screening before committing to more sophisticated and time-consuming tier 2 techniques (e.g. stable isotope analysis, high resolution mass spectrometry) for confirmatory or orthogonal analysis. The use of FT-NIR spectroscopy will complement the other analytical approaches that are being developed and used at FSCL under CRP D52042 “Implementation of Nuclear Techniques for Authentication of Foods with High-Value Labelling Claims” and transferred to the Member State laboratories.

FSCL is currently in the process of using the MPA II FT-NIR system to develop methods for the geographical discrimination of green coffee from Costa Rica and of crude palm oil from Malaysia, and the verification of authenticity of organic strawberries. Future applications may include the authenticity and safety screening of spices, edible oils, and other high added-value commodities. Some of this research work will be presented in future editions of the FSC newsletter. Visiting scientists, fellows and interns will have the opportunity to be trained in the operation of the MPA II benchtop FT-NIR system, and to learn the analytical methods that can be applied, thereby supporting Member State efforts to improve their food safety and authenticity control systems and raising awareness of this highly accessible and novel rapid screening technology.

SERS: a New Technique for Food Safety Screening at FSCL

Britt Maestroni, Sofia Bussalino, Simon Kelly

A new surface enhanced raman spectroscopy (SERS) spectrometer from Metrohm, known as the Metrohm Instant SERS Analyzer (MISA) was commissioned at the Food Safety and Control Laboratory (FSCL) in May 2022.

Raman spectroscopy is a non-destructive chemical analysis technique which provides detailed information about chemical structure and molecular interactions. It is based upon the interaction of light with the chemical bonds within a specific matrix.

Raman is a light scattering technique, by which molecules scatter incident light from a high intensity laser light source. Most of the scattered light is at the same wavelength (or colour) as the laser source and does not provide useful information – this is called the Rayleigh scattering. However, a small amount of light (typically 0.0000001%) is scattered at different wavelengths, which depend on the chemical structure of the molecules (analytes) – this is called Raman scattering. This is a chemical fingerprint for a particular molecule or matrix, and can be used to very quickly identify the matrix, or distinguish it from others. A Raman spectrum enables the identification of molecules and their functional groups, similar to infrared spectroscopy, but within the visible light range (Figure 1). Not every molecule or functional group exhibits Raman scattering, so this technique is specific to some molecules, and extensive reports exist in the literature on which compounds can be analysed with this technique. One drawback of Raman scattering is the inherent insensitivity of Raman, as only ~1 in 10^6 incident photons are Raman scattered and the typical limits of detection for normal Raman scattering can range from 1 to 10% in concentration. Therefore, surface enhancement is required to improve the detection levels, typically to parts per million (ppm) or parts per billion (ppb) levels.

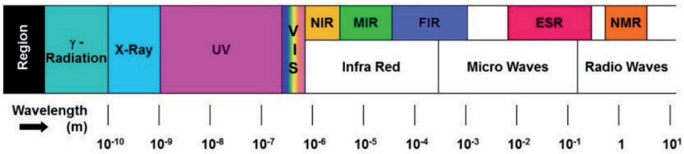


FIG. 1. The electromagnetic spectrum highlighting the VIS range (Image source: Bruker Optics GmbH).

The development of the SERS enhancement technology is being driven by the recent availability of colloidal solutions of silver or gold nanoparticles, or by highly controlled nanostructures that can be deposited on a strip substrate using metals such as gold or silver (Figure 2). In this case the signal is enhanced by approximately one trillion times, provided the correct laser wavelength is selected for the best interaction possible with the metals. In the case of the strips, a few microliters of a sample extract are deposited on the strip surface and introduced into the dedicated strip holder of the SERS instrument.

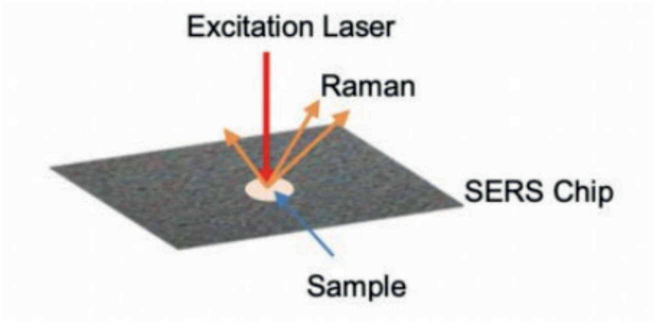


FIG. 2. The principle of the SERS-strips (Image source: Metrohm).

SERS spectroscopy offers a possibility for screening samples for residues and contaminants, and the FSCL is interested in developing new applications to detect Raman scattering analytes in various matrices, thereby supporting Member States with novel and highly accessible rapid screening technology.

Upon commissioning of the screening device, the FSCL worked on an existing application for detection of Sudan dyes. Sudan dyes (Sudan I, II, III, IV) are oil-soluble azo dyes banned for use as food colorants. However, they have been illegally used to intentionally adulterate a series of food products, including spices, and palm oil, among others, to enhance the perceived quality of the products. It is important that laboratories worldwide are able to detect the presence of such banned substances by applying rapid screening technologies.

Figure 3 shows a comparison made at the FSCL between strips and colloidal solutions of metals for SERS enhancement of Sudan IV at a concentration of 1 ppm in organic solvent.

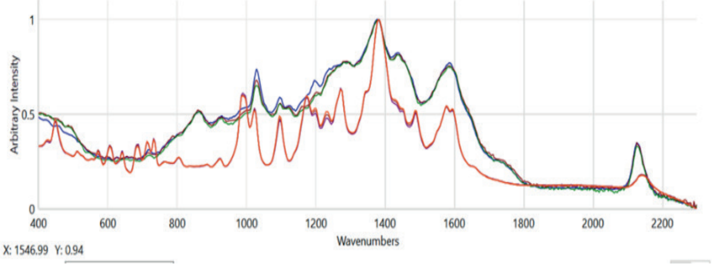


FIG. 3. Spectra obtained from the analysis of Sudan IV dye at 1 ppm using strips (red spectra) and colloidal solutions (bue/green spectra).

The colloidal solutions approach provides much more informative spectral data in specific regions for Sudan IV dye in organic solvent. Future development work will include the detection of Sudan IV dye in food matrices, such as palm oils, to enable laboratories to quickly and confidently apply a screening technology to test food products for contaminants and adulterants such as toxic dyes.

Another application of the MISA, successfully implemented at FSCL and demonstrated at the IAEA event contributing to Vienna Daughter's Day, was the detection of the fungicide, thiabendazole, on the surface of bananas, using the strip application. Swabbing the surface of the bananas with silver strips, previously wetted with methanol, allowed the identification of samples with thiabendazole residues at a concentration of 1 mg/Kg. Further studies will be implemented to decrease the quantitation limits, which are currently above the maximum residue limits set for thiabendazole in food commodities other than bananas, and expand the scope of the swabbing test applications to other relevant commodities.

SERS has the potential to be a suitable and cost-effective tier 1 analytical tool that can be used for sample screening, as the operational costs are low, the sample throughput is high, the analysis time is extremely short, and the testing can be implemented in the field with no need of a specialized laboratory facility.

Future applications may include the authenticity of spices, and other high added-value commodities. Visiting scientists, fellows and interns will have the opportunity to be trained in the operation of the MISA, and to identify new applications for ensuring food safety.

Discrimination of Organic and Conventional Orange Juice Using Untargeted HRMS-Metabolomics

Alina Mihailova, Marivil Islam

Currently no routine authenticity testing is carried out on organic products before they reach the consumer. The verification of organic production relies predominantly on certification processes and regular farm inspections. Significant cases of fraud, where conventional produce has been mislabelled and passed off as organic, on a large scale, have been reported over the past several years in Europe and worldwide. For consumer confidence and the integrity of the whole organic food and drink supply chain it is therefore highly important that, in addition to certification, the claimed specifications of foods can be analytically verified in an objective and independent way.

Various analytical techniques have been applied over the past decade for the authentication of organic products. Stable isotope and elemental analysis have been the most widely tested approaches; however, successful verification of organic production often cannot be achieved based solely on these techniques. Current thinking is that the authentication of organic food products is unlikely to be achieved by the measurement of a single or only a few selected markers.

Metabolomics allows the study of small molecules (metabolites) within cells, biofluids, tissues or organisms. The growing number of studies on the use of untargeted high-resolution mass spectrometry (HRMS)-based metabolomics report that this analytical approach has potential for differentiation between organically and conventionally grown crops. A wide range of primary and secondary metabolites, including phenolic compounds, organic acids and amino acids, have been reported to differ significantly between organic and conventional crops and could serve as potential discriminant markers. Therefore, more, well-designed studies are required to assess the applicability of the untargeted metabolomics approaches for organic food authentication. In addition, the combination of untargeted metabolomics and stable isotope analysis, has been shown to produce robust classification models and would warrant further investigation in future.

FSCL has investigated untargeted metabolomics analysis for the authentication of organic orange juices from Mexico. This work supports CRP D52042 "Implementation of nuclear techniques for authentication of foods with high-value labelling claims".

A total of 40 organic and 16 conventional orange juice samples from Mexico were used for this study. Samples were extracted in methanol and analysed using ultra-high performance liquid chromatography-quadrupole time-of-flight mass spectrometry. Mass spectral data were acquired using electrosparay ionisation in both positive and negative ion modes over the range m/z 50 to 1200, in continuum format. Data processing (alignment, normalization, peak detection, data mining) was performed before chemometrics analysis.

Principal component analysis (PCA) was used to assess the data quality and evaluate the initial groupings of the organic, conventional, and quality control (pooled) samples. PCA showed a clear separation of organic and conventional samples in both positive and negative ion modes (Figure 1). The goodness of fit (R2(cum)) and predictability (Q2(cum)) values of the PCA model in positive ion mode (Figure 1A) were 0.876 and 0.741, respectively. The goodness of fit and predictability values of the PCA model in negative ion mode (Figure 1B) were 0.785 and 0.651, respectively.

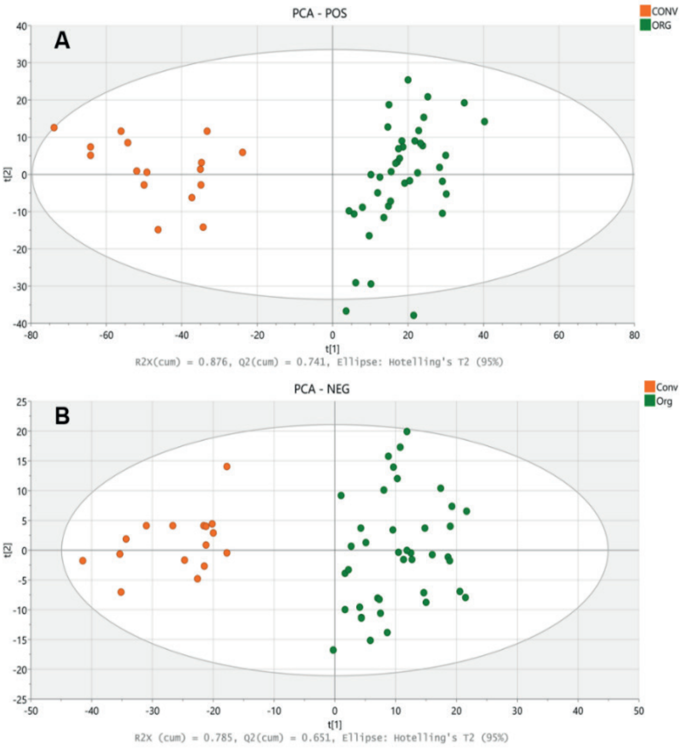


FIG. 1. PCA score plot of organic (green dots) and conventional orange (orange dots) juice samples in positive (A) and negative (B) ion mode.

A supervised chemometric approach, orthogonal projections to latent structures discriminant analysis (OPLS-DA), was applied to discriminate organic and conventional juice samples. The OPLS-DA model allowed good discrimination of organic and conventional samples (Figure 2). The goodness of fit (R2X(cum), R2Y(cum)) and the predictive ability values of the 7-fold cross-validated OPLS-DA model in positive ion mode (A) were 0.568, 0.985 and 0.966, respectively. The goodness of fit (R2X(cum), R2Y(cum)) and the predictive ability (Q2(cum)) values of the 7-fold cross-validated OPLS-DA model in negative ion mode (B) were 0.602, 0.989 and 0.959, respectively.

Further, the selection of features that were significant for the discrimination between organic and conventional orange juices, and the identification of chemical compounds was carried out. A total of 2893 and 939 features in positive and negative ion mode, respectively, were aligned. The filtered features weresubjected to further chemometric analysis using OPLS-DA. Variable importance in projection (VIP) scores and the S-plot from the generated OPLS-DA model were used to select the most significant markers for the discrimination. This step resulted in 64 and 65 features in positive and negative ion mode, respectively.

The molecular formula of each marker was calculated using accurate mass information. Initial metabolite identification was performed based on their exact mass (mass error < 2 mDa), molecular formula and isotopic pattern using chemical databases. The experimental MS/MS spectra were compared to reference spectra for further confirmation.

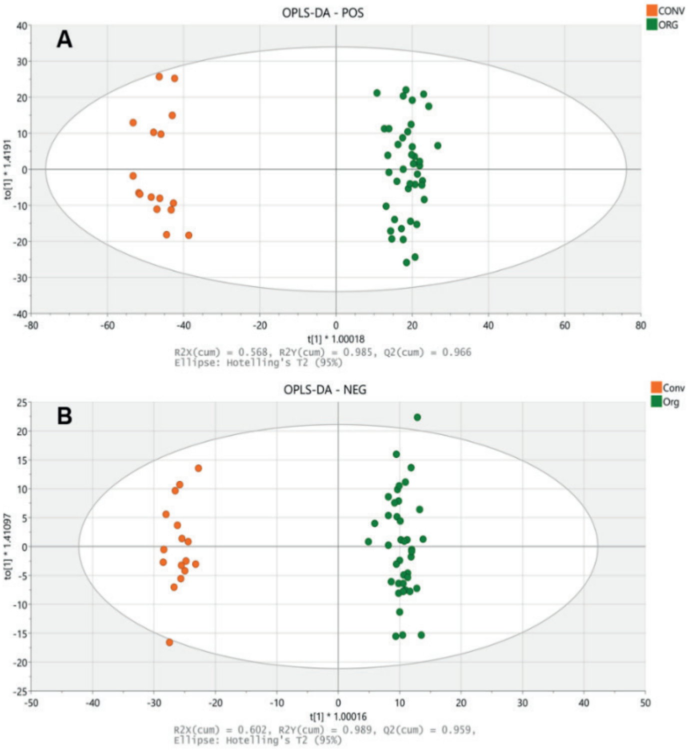


FIG. 2. OPLS-DA score plot of organic (green dots) and conventional (orange dots) orange juice samples in positive (A) and negative (B) ion mode.

Table 1 shows the putatively identified markers that were significant for the discrimination of organic and conventional orange juices. The most significant markers responsible for the differentiation were found to be flavonoids (e.g. naringenin, rutin, hesperitin, hesperidin, isosakuranetin, tangeretin), organic acids (citric acid, citraconic acid) and sugars (sucrose).

Further work on the differentiation of organic and conventional fruit juices will continue at FSCL upon receipt of more authentic juice samples.

TABLE 1. SUMMARY OF TENTATIVELY IDENTIFIED MARKER COMPOUNDS RESPONSIBLE FOR THE DISCRIMINATION OF ORGANIC AND CONVENTIONAL ORANGE JUICES

Putative identification[1]	Elemental composition	RT[2] (min)	m/z (Precursor ion)	Ion	m/z (Fragment ion)	Mass error (mDa)	ANOVA p-value
Citraconic acid	$C_5H_6O_4$	0.80	129.0181	$[M-H]^-$	85.0262; 129.0158	-0.7	0.0000
Sucrose	$C_{12}H_{22}O_{11}$	1.01	341.1083	$[M-H]^-$	89.0224; 179.0531	-0.1	0.0000
Citric acid	$C_6H_8O_7$	1.56	191.0187	$[M-H]^-$	87.0071; 111.0071	-0.5	0.0000
Quercetin 3-rutinoside 7-galactoside	$C_{33}H_{40}O_{21}$	8.05	771.1988	$[M-H]^-$	301.0301; 300.0254	0.4	0.0234
Vicenin 2	$C_{27}H_{30}O_{15}$	9.10	593.1509	$[M-H]^-$	353.0665; 473.1083	0.3	0.0000
Isorhamnetin-3-Galactoside-6"-Rhamnoside	$C_{28}H_{32}O_{16}$	9.14	623.1619	$[M-H]^-$	315.0502; 300.0219	0.7	0.0000
Naringenin	$C_{15}H_{12}O_5$	9.56	271.0603	$[M-H]^-$	151.0033; 119.0489	-0.3	0.1710
aringenin-7-O-glucoside	$C_{21}H_{22}O_{10}$	9.56	433.1142	$[M-H]^-$	271.0603; 272.0659	0.7	0.0500
Xanthorhamnin	$C_{34}H_{42}O_{20}$	10.91	769.2187	$[M-H]^-$	314.0441; 315.0502	-0.4	0.0001
Rutin	$C_{27}H_{30}O_{16}$	11.20	609.1451	$[M-H]^-$	300.0290; 301.0337	-0.5	0.0155
Naringenin chalcone	$C_{15}H_{12}O_5$	12.29	271.0603	$[M-H]^-$	151.0008; 119.0489	-0.3	0.0000
Isorhamnetin	$C_{16}H_{12}O_7$	12.72	315.0502	$[M-H]^-$	300.0254; 151.0008	-0.3	0.0000
Hesperitin	$C_{16}H_{14}O_6$	13.13	301.0714	$[M-H]^-$	164.0103; 136.0143	0.2	0.0000
Hesperidin	$C_{28}H_{34}O_{15}$	13.14	609.1823	$[M-H]^-$	301.0727; 286.0497	0.4	0.0000
Isosakuranetin	$C_{16}H_{14}O_5$	15.96	285.0764	$[M-H]^-$	164.0103; 136.0143	0.1	0.0000
Naringenin	$C_{15}H_{12}O_5$	9.56	273.0756	$[M+H]^+$	153.0175; 147.0444	-0.7	0.0000
Apigenin-6-C-glucoside	$C_{21}H_{20}O_{10}$	10.94	433.1149	$[M+H]^+$	286.0612; 313.0715	1.4	0.0000
Isorhamnetin	$C_{16}H_{12}O_7$	12.72	317.0653	$[M+H]^+$	302.0420; 153.0201	-0.8	0.0000
Hesperitin	$C_{16}H_{14}O_6$	13.13	303.0854	$[M+H]^+$	177.0542; 153.0175	-1.5	0.0001
Isosakuranetin	$C_{16}H_{14}O_5$	15.96	287.0918	$[M+H]^+$	153.0175; 161.0601	-0.1	0.0000
Tangeretin	$C_{20}H_{20}O_7$	21.44	373.1290	$[M+H]^+$	343.0825; 358.1050	0.3	0.0000

1 - Compound identification was performed in accordance with the Metabolomics Standards Initiative (Sumner et al., 2007) at the identification level 2.
2 - Retention time.

Characterization of Commercial Spices by FTIR, ED-XRF, LC-MS/MS and Multispectral Imaging

Britt Maestroni, Sofia Bussalino, Shuichi Nakaya,
Alina Mihailova, Marivil Islam, Simon Kelly

There is an increasing interest in natural food products rich in bioactive phytochemicals with health properties, and turmeric (Curcuma longa), is among those natural food products. As a medicine, turmeric is used for the prevention and therapy of diseases, as a food it is used as a spice and a colorant, and as a food supplement it is adopted for its antioxidant, anti-inflammatory, antimicrobial, and antimutagenic properties.

Given the economic importance of spices and medicinal plants that grow primarily in tropical regions, including India, Thailand, Pakistan, China, Sri Lanka, Peru, Haiti, Madagascar and Jamaica, and the fact that many Asian countries consume a large amount of the worldwide turmeric production, the FSCL published a method for the analysis of multiple residues and contaminants in turmeric powders to help ensure their safety and quality.

However, it is not only residues and contaminants that are of concern in spices such as turmeric. Economically motivated food fraud is an important aspect affecting the commercialization of natural food products. It is well known that economically motivated adulteration, involving mixtures and dilutions of authentic spices with low-cost adulterants, such as toxic dyes or plant materials with no nutritional value, to increase profit margins, is frequent. One aspect of economically motivated adulteration is the deception of consumers when conventional crops are sold as organic, for which a premium price must be paid.

From an analytical point of view, the biggest challenge for organic certification is the lack of officially recognized end-product tests to verify the many aspects of organic production requirements. This results in food fraud incidents, which are very costly not only in terms of branding reputation and loss of consumer trust in the national food control systems, but potentially also to consumers’ safety if harmful adulterants are used.

As part of the continuing effort by the FSCL to offer analytical methods to Member States to verify the integrity of food products, which includes not only food safety but also food authenticity and food quality aspects, the FSCL has continued in its efforts to identify analytical techniques that could effectively and rapidly characterize commercial samples of turmeric, with one of the goals being to be able to discriminate between organic and conventional turmeric samples.

It was previously shown by FSCL that the analysis by headspace-gas chromatography-ion mobility spectroscopy of the volatile fraction from turmeric teas, is a potentially useful technique to distinguish between organic and non-organic (from conventional agriculture) turmeric teas.

In this second study a set of analytical techniques were applied to 51 samples of turmeric sold as either organic or conventional. Eight samples were obtained by cryogenic milling of rhizomes (3 organic and 4 conventional). The commercial powders of turmeric were purchased from the Austrian, Italian, Spanish and UK markets. One turmeric sample was purchased from NIST. Most of the labels on the commercial products provided an indication of the provenance of the turmeric powders. Some labels did not provide this information. It was recognized that the samples sold on the market as organic may, in fact, have been fraudulent and could not be regarded as authentic organic samples. However, the focus of this study was to investigate the characterization of the turmeric samples by different analytical techniques to evaluate the applicability of each techniques and the information provided.

A set of different analytical techniques were individually applied to all the samples to provide different types of data. These techniques included rapid screening techniques that required minimal sample preparation and a confirmatory technique, using liquid chromatography-tandem mass spectrometry, with a simplified sample preparation procedure.

Fourier-transform infrared spectroscopy with attenuated total reflection (FTIR-ATR) was used to acquire spectra in the range 450 to 4000 cm^{-1} (Figure 1). For this technique, turmeric samples were homogenized using a ball mill, freeze dried and then well mixed again in a mortar and pestle. A few milligrams of sample were utilized to acquire 10 genuine replicates. The data from replicate scans were averaged, pre-processed using the multiplicative scatter correction function and subjected to principal component analysis (PCA).

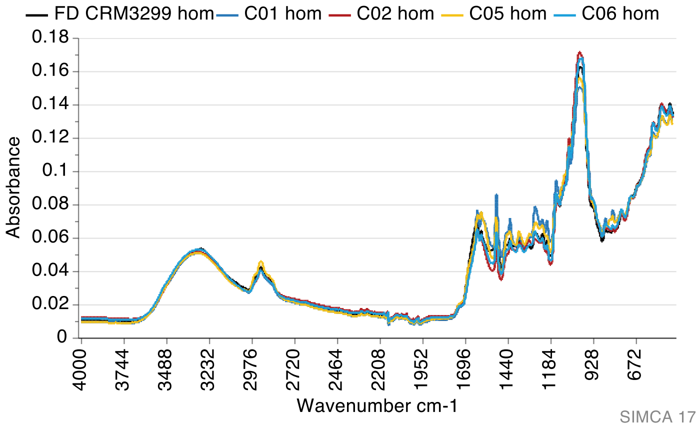


FIG. 1. Mean raw FTIR-ATR spectra of selected authentic samples of turmeric between 4000 and 450 cm^{-1}.

Multispectral imaging (MSI) is an innovative and non-destructive technique that combines imaging and spectral technologies with advanced digital image analysis and machine learning. This technique was applied to acquire, in less than 10 seconds, spectra in the range 365 nm (UV-A region) to 970 nm (near-infrared region). The high-resolution spectral image acquired permits fast and accurate characterization of turmeric samples in terms of colour, surface chemistry, texture, shape, and size. For this technique, the homogenized samples from the FTIR study were further combined with a wax binder and pressed into a 32 mm circular pellet using a pellet press. The pellets were then subjected to MSI analysis. First, a region of interest of the same size was selected for each turmeric sample, and the spectra (365–970 nm) were obtained for each region. The software was used to obtain the morphological and colour features of each sample. The spectral, morphological and colour features of each sample were scaled and subjected to chemometrics analysis. PCA was used to assess the data quality and evaluate the initial turmeric sample groupings.

Energy Dispersive X ray Fluorescence (EDXRF) spectrometry was applied to conduct rapid elemental profiling of turmeric samples, to screen for excessive concentrations of some elements that may pose a threat to food safety. A qualitative-quantitative method was initially applied to all turmeric samples to identify possible elements of interest in the turmeric samples. For selected elements, a calibration using a series of NIST reference standards at different element concentration was used (Figure 2). Although the NIST reference standards used in this study, except for one turmeric matrix, were different in terms of the matrix analysed, calibration functions with regression coefficients $R^2 > 0.99$ and quantitative information could be obtained for a few elements. Data were normalized using the standard normal variate transformation algorithm and subjected to PCA.

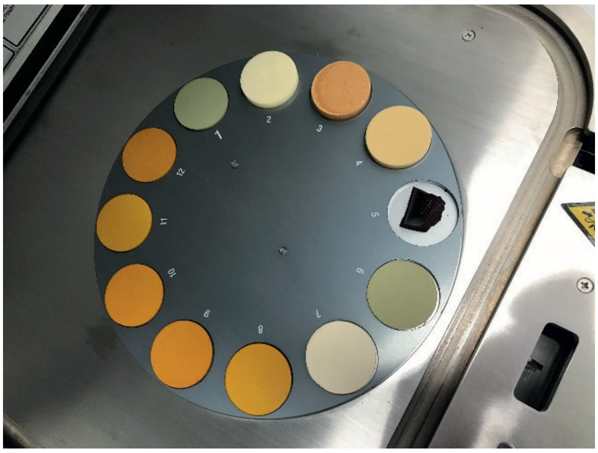


FIG. 2. Turmeric samples are analysed together with NIST reference materials in the ED-XRF.

Ultra-high-performance liquid chromatography coupled to tandem mass spectrometry (UHPLC-MS/MS) was applied to characterize the turmeric samples in terms of their curcuminoids content, the most valuable and active ingredients present in turmeric. The curcuminoids are a mixture of curcumin, and the two derivatives, demethoxy curcumin and bis-demethoxy curcumin. The trade price of turmeric also depends on its curcuminoid content; therefore, it is important to be able to determine their quantity and quality (organic vs synthetic). A sample preparation method was developed for the analysis of curcuminoid content in turmeric. Essentially two strategies were adopted to extract the curcuminoids: a sonication in the presence of methanol and a microwave extraction using methanol, followed by filtration. A 'dilute and shoot' strategy was employed to inject the extracts into the UHPLC-MS/MS. The chromatographic separation took place in a Raptor Biphenyl column, and the MS/MS detection of the curcuminoids was performed in the multiple reaction monitoring mode using transitions and collision energies previously selected for each compound. Data were normalized using the standard normal variate transformation algorithm and described with PCA.

Data generated through the different techniques helped to characterize the commercial samples of turmeric. Regarding the discrimination of organic and conventionally produced turmeric, the PCAs obtained by the individual techniques, did not indicate any strong tendency to separate the samples into two well defined groupings. However, as mentioned previously, the samples tested cannot be considered authentic and these individual results are still preliminary. Nevertheless, the results will provide useful information to the analytical chemist who may be challenged by the analysis of commercial samples of turmeric powders, in terms of the sensitivity and application of the various techniques used and the information that can be extracted through their application. Additional analyses of organic residues and contaminants in the samples of turmeric are currently being carried out. The additional analytical data generated will be combined with existing information using data fusion approaches. It is important to clarify if data fusion and chemometrics approaches will be able to identify trends from the analysis of commercial powders; there is little information available on this. It is hoped that the FSCL collection of authentic samples of turmeric can be increased in the near future by Member States contributions.

TABLE 1. THE MS/MS CONDITIONS TO DETECT CURCUMINOIDS IN TURMERIC

Target	R.T.[1] (min)	Polarity	Transition 1	CE[2]	Transition 2	CE	Transition 3	CE
Curcumin	2.283	-	367.10>134.25	30	367.10>217.25	20	367.10>149.15	26
Demethoxy curcumin	2.158	-	337.10>119.15	10	337.10>217.20	20	337.10>173.20	16
Bis-demethoxy curcumin	2.05	-	307.10>119.10	18	307.10>187.15	16	307.10>143.25	50

1 - Retention time.
2 - Collision energy.

Development of a Rapid Low-Cost Compound-Specific Dual Stable Isotope Analysis of Nitrate (δ15N, δ18O) in Fruit Extracts to Differentiate Organic and Conventional Production

Simon Kelly, Marivil Islam, Aiman Abrahim,
Florence Maxwell, Cedric Douence, Len Wassenaar

Organic crops continue to be an important and growing export market for many low and middle income countries. Furthermore, the global organic food and drink market reached a value approaching 100 billion US dollars in 2017 and is predicted to triple by 2025. Organic produce is usually sold at a premium price compared to its conventional counterparts because of additional labour costs, less intensive production methods and generally lower yields and this can result in price differences of a few to hundreds of percent. Although supply chain inspections should verify the traceability of organic foods with respect to permissible inputs on the farm and at all stages of production and distribution, the length and complexity of global trade routes means this a formidable task, and inevitably fraudulent misdescription of conventionally produced foods as organic does occur. As with conventionally cultivated crops, organic crops are usually subject to routine safety monitoring, such as pesticide residue analysis, as part of member state surveillance programmes. However, there are no officially recognized end-product tests to verify the many aspects of organic production requirements, such as the non-permitted use of synthetic nitrate fertilizers. This is a major gap in the quality control of organic produce since significant economic incentives, to fraudulently mislabel conventionally cultivated crops as organic, remain high. This has led to many recorded incidents of fraud in recent years, at a cost of millions of dollars, and it therefore seems quite probable that many cases of organic food fraud will have gone undetected due to the absence of end-product tests. Ultimately, this can lead to reputational damage for an industry that promotes ethical production and relies heavily on consumer trust. The potential for fraud effects all retail outlets, from small local farmers' markets to supermarket chains and global retailers. To underpin the integrity of the organic food and drink sector, it is extremely beneficial, in addition to certification systems, when the authenticity of organic labelling claims can be independently verified using analytical methods that can assess specific aspects of organic production.

Organic food systems do not use synthetic nitrate (NO_3^-) fertilizers that possess characteristically high stable oxygen-18 isotope ($\delta^{18}O$) values and relatively low stable nitrogen-15 isotope ($\delta^{15}N$) values, compared to nitrate produced naturally by nitrifying bacteria in soil. This rationale may be used as the basis to verify explicit fertilization practices that are inherent in organic production labelling claims. Compound-specific dual isotope analysis of nitrate ($\delta^{15}N$, $\delta^{18}O$) in fruits and vegetables has been used previously to successfully differentiate organic from conventional food production where synthetic nitrate has been used. However, one major barrier to its routine implementation for analyses of nitrate from plant or food extracts is the lengthy and costly techniques required to convert extracted nitrate into nitrous oxide gas (N_2O), which is used for measurement of the isotope $\delta^{15}N$ and $\delta^{18}O$ values. Currently, methods such as bacterial denitrification or cadmium reduction have been used that require specialised culturing of denitrifying microbes or chemicals that are toxic, and difficult and costly to dispose of, respectively. Consequently, rapid, low-cost methods are needed to facilitate the isotopic analyses of nitrate in food products to support this aspect of organic certification and to help confirm the veracity of production claims. FSCL, in collaboration with the IAEA's Isotope Hydrology Laboratory, developed a new titanium (III) reduction technique that provides a low-cost and rapid measurement of the compound-specific $\delta^{15}N$ and $\delta^{18}O$ isotope values of nitrate in fruit and vegetable hot-water extracts. The feasibility of this new method was successfully demonstrated by differentiating between organic and conventionally cultivated strawberries of known provenance. Authentic commercial samples of organic and conventionally cultivated strawberry fruits were obtained via a collaborating body, the SGF (Sure, Global, Fair, previously known as Schutzgemeinschaft der Fruchtsaft- Industrie e.V) a non-profit industrial association financed by more than 650 fruit juice companies from nearly 60 countries worldwide. Forty authentic production samples of organic (n=20) and conventionally produced strawberries (n=20) were obtained from Spain. One-kilogram samples were supplied as frozen whole strawberries, with each sample coded to the location of the grower. The organic and conventional samples were from the Southern part of Province Huelva, Spain. Both types of strawberries were cultivated in polythene tunnels, with fertigation from water that was derived from surface or well water.

The strawberry samples were homogenized and a 0.5g subsample taken for analysis. The nitrate was extracted using hot-water according to the European Standard method (EN 12014-2:2017). The nitrate extracts were analysed by ion chromatography to determine the nitrate concentration and a range of other cations and anions (K^+, Mg^{2+}, Ca^{2+} NH_4^+, F^-, Cl^-, PO_4^{3-} and SO_4^{2-}). The extracts were then treated with sulfamic acid to remove possible interference from any nitrite (NO_2^-) present and diluted in a headspace vial to achieve a nominal nitrate concentration of 0.1 ppm. Each sample was then acidified with hydrochloric acid and the reduction of nitrate to N_2O gas was completed by reaction with an acidic solution of Titanium (III) Chloride. Samples were left standing overnight at room temperature in a fume hood to permit complete conversion of NO_3^- to N_2O gas. The mass distribution of the N_2O gas in the headspace was then determined, using continuous-flow isotope-ratio mass spectrometry, against nitrate stable isotope reference materials permitting the $^{15}N/^{14}N$ and $^{18}O/^{16}O$ ratios and $\delta^{15}N$ and $\delta^{18}O$ values of the strawberry nitrate extracts to be determined.

The nitrogen and oxygen stable isotope analyses of nitrate from strawberry extracts revealed a clear differentiation between the organic and conventional production systems for the samples used in this study, with mean $\delta^{18}O$ and $\delta^{15}N$ values of +18.3 ±1.2 mUr and +17.6±1.2 mUr versus +28.2 ± 4.5 mUr and +14.9 ±2.9 mUr, respectively (Figure 1). (note: the urey (Ur) is now the preferred supplementary SI unit, previously reported as per mil; 1 mUr = 1 ‰).

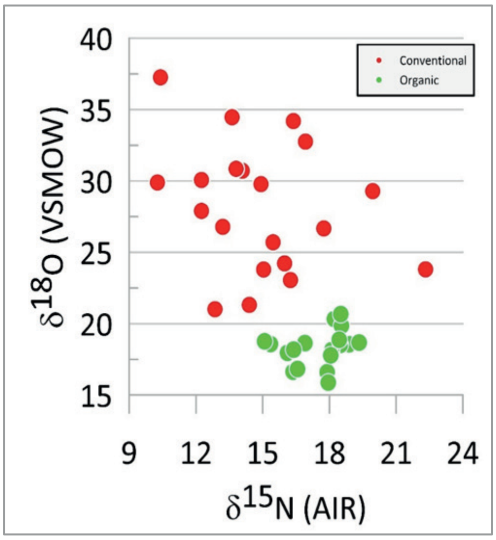


FIG. 1. Oxygen and nitrogen stable isotope composition of nitrate ($\delta^{18}O$ and $\delta^{15}N$ measured as N_2O) in fresh strawberry extracts from conventional and certified organic fruit grower operations in Andalucía, Spain.

A students t-test revealed that the mean $\delta^{18}O$ and $\delta^{15}N$ values for organic and conventional nitrate samples differed significantly from each other (p=0.05). The larger $\delta^{18}O$ difference in the nitrate between organic and conventional production of approximately 10 mUr provided an unequivocal diagnostic differentiation between synthetic nitrate and organic fertilization practices. Notably, there was a smaller isotopic range and variation in the $\delta^{18}O$ values of nitrate extracted from the certified organic strawberry cultivated samples compared to the conventionally cultivated samples (Figure 1). This implies that certified organic production practices may be more consistent due to their biological-based fertilization practices. Because the δ^{18}O in nitrate derived from nitrification of organic fertilization is largely controlled by the oxygen isotopic composition of the (local) water used in the irrigation/fertigation system. Consequently, there may be geographic differences in nitrate δ^{18}O amongst organic production operations over greater geographical regions, which could, with further research, potentially be linked to known and predictable spatial oxygen isotope patterns. This could potentially be used to further evaluate the authenticity amongst organic growers at larger spatial scales. However, across the strawberry cultivation area in Andalucía studied here, the range for annual weighted-mean oxygen isotope values in precipitation, that should be similar to the irrigation water, was only 1.5 mUr, and therefore too small to solely explain the larger δ^{18}O differences observed for nitrate in organic strawberries. However, the conventional strawberry cultivation, utilizes a much wider range of potential inputs and fertilizers, which was reflected in the wider isotopic range and scatter for δ^{18}O and δ^{15}N. Overall, the results show the Ti(III) reduction method provides a new low-cost and rapid analytical method to reliably measure nitrogen and oxygen stable isotope ratios in extracted nitrate from fruits and vegetables, for the purposes of assessing nitrate fertilization practices of organic versus conventional production claims and to support authenticity investigations. The ability to prepare and analyse 70 nitrate reference controls, and unknown samples (limited only by autosampler tray size) over 48 hours at low cost makes this method attractive for adoption by Member States. The total time to prepare 70 samples including the 12 calibration standards and 4 control standards was approximately 2 hours. The total cost of the reagents and vials was less than €10 per sample. Furthermore, potential exists to assess the geographic authenticity of certified organic fruits and vegetables by exploiting the highly predictable spatial patterns in δ^{18}O of irrigation waters i.e. fruit or vegetable nitrate oxygen isotope maps (isoscapes).

Development of a New Method to Determine the Hydrogen Isotope (δ2H) Value of Starch in Foods to Determine its Provenance

Aiman Abrahim, Vamsi Golla, Simon Kelly

The measurement of the stable hydrogen isotope (δ^{2}H) value of mono-, di- and polysaccharides in foods has great utility to determine their botanical and geographical origin in food authenticity studies, where there is suspicion that their claimed provenance does not match their true identity. However, the hydrogen isotopic information from saccharides present in food must be corrected for extraneous water because the hydrogen atoms in hydroxyl groups (OH) in saccharides can freely exchange with their surroundings. Consequently, physically isolated saccharides, which are relatively hygroscopic can be affected by moisture that has become ‘attached’ by absorption, adsorption and/or chemical exchange with OH from the food matrix and atmospheric water vapour. The net effect is that the direct measurement of the δ^{2}H value of a saccharide measured in one geographical location can vary significantly from the δ^{2}H value of the same sample measured at different location, due to variations in the global distribution of stable hydrogen isotopes in water (and vapour). The deleterious effects of this extraneous water on the δ^{2}H measurement of saccharides can be removed by appropriate methods, and currently one of the most popular approaches is the dual water equilibration technique, that can ‘separate’ the extrinsic hydrogen contribution from the intrinsic hydrogen contribution, or carbon-bound non-exchangeable (CBNE) hydrogen. The dual water equilibration technique requires saccharide samples to undergo equilibration with the vapour from two isotopically distinct ‘standard’ waters in a sealed and previously evacuated chamber. After a given period of time it is assumed that the extrinsic water in the sample will have completely exchanged with the vapour in the chamber. A further confounding factor (α) is introduced at this stage, as the vapour of the two standards will not have the same δ^{2}H value as the water. The factor ‘α’ is the ratio of the hydrogen isotopic composition of the vapour to the water and varies with temperature. After equilibration and drying of the saccharide samples under carefully controlled conditions, and measurement of the δ^{2}H value by isotope ratio mass spectrometry (IRMS), it is possible to calculate the intrinsic or CBNE hydrogen isotope value of the (poly)saccharide. This process is time consuming because equilibration may take up to 3-days and samples must be prepared in duplicate, for each of the isotopically distinct water equilibration chambers, and this is before any replicate measurements of the duplicate preparations are considered.

The Food Safety and Control Laboratory (FSCL) previously reported the development and application of a novel procedure for the direct and rapid isotope analysis of the CBNE hydrogen in mono- and disaccharides by gas chromatography (GC) coupled to IRMS via a modified chromium/silver (CrAg) -reduction reactor (FEP Newsletter, Vol. 24, No. 1, January 2021 pp 30–32). This was used successfully to demonstrate the feasibility of detecting undeclared addition of exogenous sugar products to foods and beverages susceptible to economically motivated adulteration e.g., sucrose addition to pineapple juice. The procedure utilizes a simple one-step reaction, with the derivatising agent N-methyl-bis-trifluoroacetamide (MBTFA), to substitute the exchangeable hydroxyl-hydrogens in mono- and disaccharides with trifluoroacetate derivatives that are sufficiently volatile to be separated and measured by GC-IRMS. The modified CrAg-reduction reactor retains carbon, oxygen and fluorine whilst releasing hydrogen gas for stable isotope measurement. The new procedure is easy to use, has fast analysis times and provides compound-specific information. However, it cannot be applied to polysaccharides such as starch due to their high molecular mass and non-volatility.

Consequently, a new approach has been developed in FSCL to permit the δ^2H value of the polysaccharide starch to be determined as glucose. Figure 1 shows a schematic of the method protocol involving microwave assisted hydrolysis (MAH) followed by freeze-drying. The resulting glucose is derivatised in the usual way using MBTFA and the δ^2H value determined by GC-IRMS with the modified CrAg-reduction reactor (GC-CrAg-IRMS).

It was very important to demonstrate that the methodology does not introduce any uncontrolled isotopic fractionation of the hydrogen isotopes during the MAH of starch, freeze-drying and/or MBTFA derivatisation of glucose. To test the repeatability of the entire procedure a number of replicate experiments were made on different days to ensure that no significant fractionation was observed when compared to the previous and expected δ^2H measurement performance for mono- and disaccharides. Table 1 shows the results of δ^2H measurements, over four days, of glucose derived from MAH of rice starch.

TABLE 1. MEASURED δ^2H VALUES OF TFA DERIVATIVES OF GLUCOSE OBTAINED AFTER MICROWAVE ASSISTED HYDROLYSIS OF RICE STARCH

Preparation day	1 (n = 3)	2 (n = 3)	3 (n = 3)	4 (n = 3)	overall (n = 12)
Mean $\delta^2H_{VSMOW-SLAP}$ [mUr]	-114.2	-114.7	-113.9	-113.2	-114.0
SD ($\sigma n - 1$) [mUr]	1.9	1.9	1.7	1.6	1.6

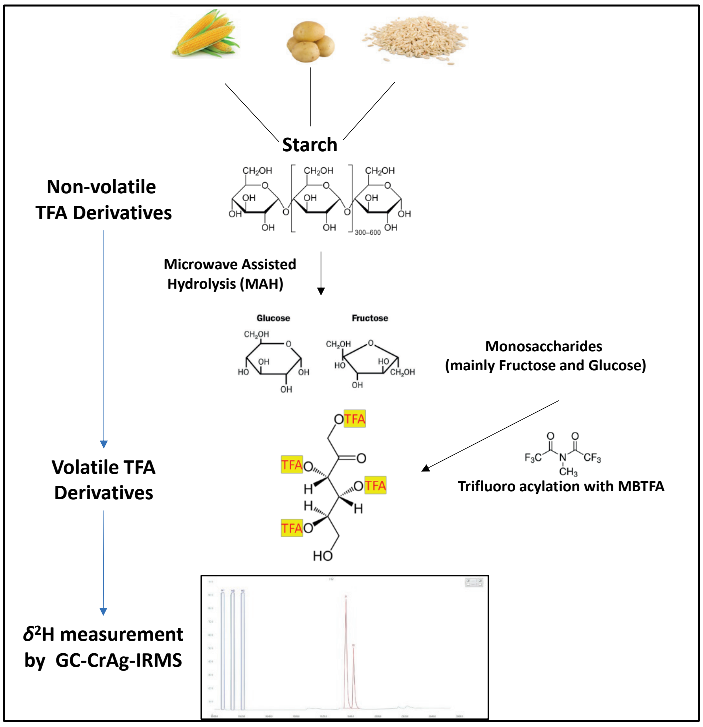


FIG. 1. Schematic of the methodology for determination of the δ^2H value from the non-exchangeable hydrogen in starch, after microwave assisted hydrolysis to glucose and measurement by GC-CrAg-IRMS.

In all cases the precision achieved on the four days, for three replicate injections of the glucose-trifluoracetate (TFA) derivative, was ≤ 1.9 mUr. This agrees well with the range of SDs reported previously for the replicate δ^2H measurements of fluorine bearing organic compounds fluorobenzene and 1,3,4 trifluorobenzene The overall SD determined on the measurements across all four days for the TFA derivative of glucose was 1.6 mUr (n =12). Once again this compares favourably with analysis of TFA derivatives of high purity reagent-grade mono- and disaccharide

materials and generally with our expectations for compound-specific hydrogen stable isotope measurements reported elsewhere. These results demonstrate that acceptable precision can be achieved under within-laboratory repeatability conditions for the preparation and δ^2H analysis of the CBNE hydrogen present in starch, as glucose after MAH, freeze-drying and derivatisation with MBTFA. This methodology will also be tested on, and applied to, other important sources of starch from wheat, maize, potato and cassava and other polysaccharides such as cellulose. Furthermore, direct comparison of δ^2H values obtained with the dual-water equilibration technique will be made in the future when FSCL receives a new automated and combined equilibration-autosampler device from Eurovector (Uniprep system).

Geographical Discrimination of Palm Oil Using Ion Mobility Spectrometry

Marivil Islam, Alina Mihailova, Florence Maxwell, Simon Kelly

Palm oil is one of the most economically important products in Malaysia. Malaysia and Indonesia are responsible for 85% of global palm oil production. The global palm oil industry is predicted to expand significantly in the coming decades due to an increasing global population and greater demand for palm oil from the food, oleochemical and energy industries. Palm oil is commonly traded as a global commodity with batches from different sources often being mixed at multiple stages during processing, shipment, refining, storage, and delivery to end users. The traceability of palm oil within the supply chain is, therefore, a complex and challenging issue. Several certification schemes, such as Malaysian Sustainable Palm Oil (MSPO) and the Roundtable on Sustainable Palm Oil (RSPO), have been set up to assure the sustainability of palm oil production and its traceability across the supply chain. The current measures in place for the traceability certification of palm oil production are based largely on paper trails and audits. Although the certification of palm oil production should ensure complete traceability of oil at all stages of production, including processing and marketing, such “paper-trail” based systems are prone to falsification. For consumer confidence and to support the integrity of the whole palm oil supply chain it is, therefore, highly important that, in addition to traceability and certification schemes, the claimed geographical origin can be analytically verified in an objective and independent way.

Various analytical techniques have been applied over the past decade for the traceability and authentication of palm oil. Geographical authentication of palm oil has been and continues to be a highly active area of research. Such authentication requires a given sample to carry characteristic chemical fingerprints that reflect its place of origin. In the case of palm oil, this can be best achieved via the analysis of major compounds (fatty acids and triacylglycerols), minor components (e.g. carotenes, tocopherols, tocotrienols and sterols), stable isotopes and volatile organic compounds. Such analytical approaches are usually assessed against several important criteria including cost and analytical speed, instrument sensitivity and selectivity, accessibility for implementation in the field, and sample pre-treatment requirements.

In this study gas chromatography ion mobility spectrometry (GC-IMS), coupled with principal component analysis (PCA) and orthogonal projections to latent structures discriminant analysis (OPLS-DA), was applied for the geographical discrimination of crude palm oils from Malaysia. This work contributes to the development of analytical approaches under CRP D52040, “Field-deployable Analytical Methods to Assess the Authenticity, Safety and Quality of Food”.

Crude palm oil samples were collected over 6 months (February–July 2019) at 4 different locations: Peninsular Malaysia (East, South and Central) and East Malaysia. The analysis was performed using a FlavourSpec GC-IMS system (G.A.S. Dortmund, Germany). Crude oil samples were incubated at 70°C and the volatile fraction was analysed. Data were normalized and pre-processed using Pareto scaling function.

Initial chemometrics analysis revealed high variability between the collection months. Discriminative models were, therefore, generated for each month separately. A supervised chemometric approach, OPLS-DA, was able to discriminate East Malaysia from the Peninsular Malaysia in most of the months. An example of the OPLS-DA score plot of the oil samples, collected in July, is shown in Figure 1. The goodness of fit (R2X(cum), R2Y(cum)) and the predictive ability (Q2(cum)) values of the 7-fold cross-validated OPLS-DA model were 0.898, 0.899 and 0.745, respectively.

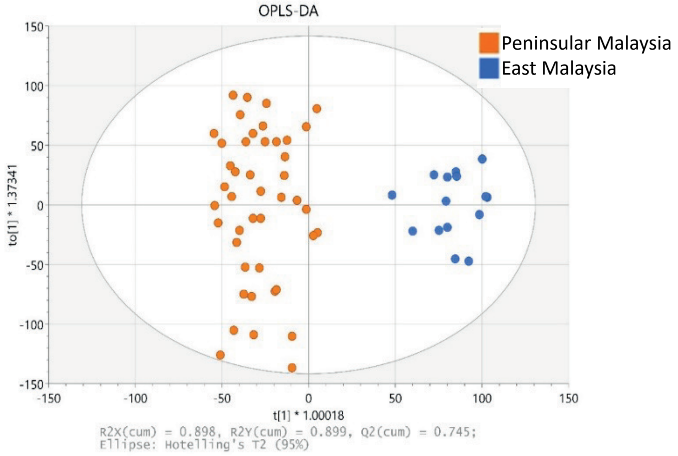


FIG. 1. OPLS-DA score plot of the crude palm oil samples collected from Peninsular and East Malaysia in July.

As the next step, we assessed the ability of the OPLS-DA model to discriminate all 4 geographical locations. This was not fully achieved by the model. Palm oil samples from East Malaysia formed a separate cluster in most of the months, and some of the locations within the Peninsula could be also discriminated one from another, e.g. East Peninsula from the South and Central Peninsula in July (Figure 2).

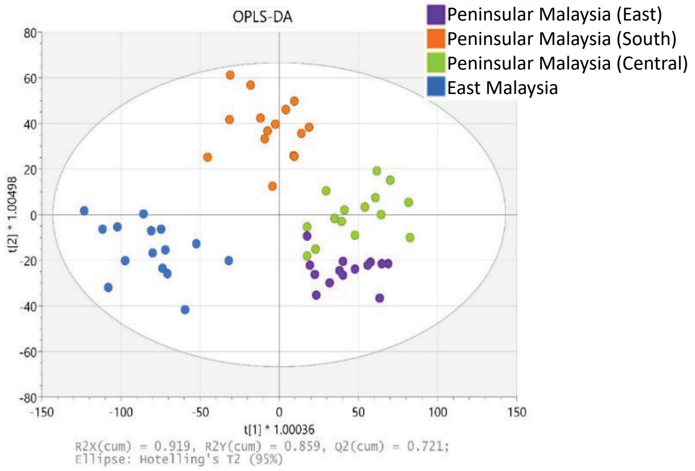


FIG. 2. OPLS-DA score plot of the crude palm oil samples collected from Peninsular (East, South, Central) and East Malaysia in July.

To assess whether OPLS-DA would achieve the discrimination of palm oil samples from the 3 locations in the Peninsula, a new model was generated. The discrimination of samples from Peninsular Malaysia was achieved in most of the months. An example of the OPLS-DA score plot of oil samples collected in July is shown in Figure 3. The goodness of fit (R2X(cum), R2Y(cum)) and the predictive ability (Q2(cum)) values of the 7-fold cross-validated OPLS-DA model were 0.909, 0.917, and 0.816, respectively.

Further work on the geographical discrimination of crude oil samples at FSCL will include the use of Fourier-transform near-infrared (FT-NIR) spectroscopy and stable isotope analysis, which will be presented in future editions of the FSC newsletter.

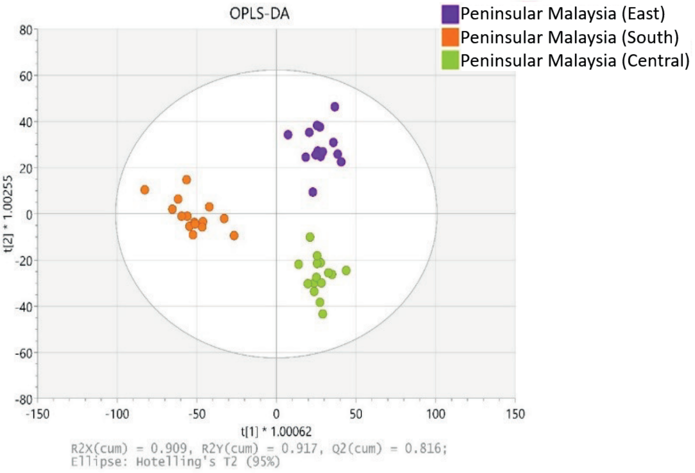


FIG. 3. OPLS-DA score plot of the crude palm oil samples collected from Peninsular Malaysia (East, South, Central) in July.

FSCL Staff

Ms Christina Vlachou took up the position of Head of the Food Safety and Control laboratory on 4 May 2022, joining us from the General Chemical State Laboratory of Greece. Christina has many years' experience in the field of food analysis. Her academic background includes a PhD in chemistry and Master's degrees in quality assurance and toxicology, as well as continuing education in chemical analysis and project management. She has participated in Greek regulatory control systems for more than 10 years, including experience as a laboratory head, specializing in the control of food contaminants and has also conceptualized and managed projects on food authenticity and fraud, focusing on spirits. Christina has also served as a laboratory assessor for the Hellenic Accreditation System, as a tutor for the European Food Safety Authority in its EU-Food Risk Assessment Induction Training regarding chemical risk assessment, as member of several Codex electronic working groups and as a EUROTOX and EFSA fellow. We welcome Christina to the FSCL team, and we are sure that her extensive research, analytical, regulatory and organizational experience will keep driving the applied and adaptive research and capacity building efforts of the laboratory forward, to the benefit of our member States. In welcoming Christina, we also thank Mr Simon Kelly for accepting the role of acting Laboratory Head since August 2021 and performing the necessary duties in addition to maintaining his outputs as our expert technical officer in stable isotope analysis and related fields.

Mr Vamsi Golla joined the laboratory team as an intern in October 2021 from the University of Göttingen, Germany, where he was completing his Master's thesis research in reconstruction of fire history from black carbon in soils of Mediterranean ecosystems using pyrogenic molecular markers such as benzene carboxylic acids and polycyclic aromatic hydrocarbons. Vamsi is currently working with isotope ratio mass spectrometry to assist in the development of stable isotope methods to verify the geographical origin of rice and green coffee beans. Mr Golla is making a valuable contribution to the FSCL team's outputs whilst benefiting from the IAEA internship training and development opportunity.

Ms Sofia Bussalino, from the University of Litoral, Neuquen, Argentina, joined the FSCL in February 2022. Sofia is currently working in the field of chemical contaminants and residues in food, contributing to the development and optimization of multi residue methods for pesticides and mycotoxins in honey and coffee using LC-MS/MS and GC-MS/MS. Additionally, Sofia will be trained in screening technologies such as Raman, FT-IR and NIR spectroscopy, as well as in chemometrics data analysis, and she will provide support to the FSCL staff regarding capacity building activities under a PUI project. In her time at FSCL, Sofia already demonstrated to be a real asset to FSCL in the analytical work and various other laboratory activities.

Mr Evans Rockson Tawiah commenced a one-year internship at FSCL in February 2022. Evans obtained an MPhil degree in Food Science and Technology from the CSIR College of Science and Technology (Accra, Ghana) and has been working at the Food and Drugs Authority of Ghana prior to joining FSCL. Mr Tawiah is making a valuable contribution to FSCL's work regarding the development and transfer to the Member States of analytical screening methods for the verification of food authenticity. During his initial period at FSCL, he has been working on the application of infrared spectroscopy techniques for the authentication of organic strawberries and for the geographical discrimination of green coffee.

During their internship, Vamsi, Sofia and Evans will not only acquire additional professional skills, but they will also provide valuable support to the FSCL work outputs with their positive attitude at work.

Announcements

New Agency Publication on Radioactivity in Food

In our last newsletter we reported on a virtual technical meeting on radionuclides in food and drinking-water in non-emergency situations. A pre-print Safety Report No.114 entitled 'Exposure due to Radionuclides in Food Other Than During a Nuclear or Radiological Emergency. Part 1: Technical Material' was published online (https://inis.iaea.org/search/search.aspx?orig_q=RN:53004342) in January 2022, a little too late to be reported in our January Newsletter. This new publication will appear in print later this year.

The Joint FAO/IAEA Centre has been working for several years on a FAO, IAEA and World Health Organization (WHO) project to develop data and methodologies that could be used to produce criteria that competent authorities could use to assess radionuclides in food in existing exposure situations (i.e., in normal circumstances, not in a nuclear or radiological emergency). Safety Report No.114 provides technical information that can be used as a basis to assess and, if necessary, manage exposure to radionuclides in food in existing exposure situations. This Joint FAO, IAEA and WHO report includes information on the observed distributions of concentrations of key natural radionuclides in various foods, the use of dietary surveys to assess ingestion doses arising from exposure to radionuclides in food, and radionuclide concentrations in natural mineral waters, aquaculture and wild foods. It will serve as a valuable reference to those interested in radionuclides (natural and human-made) that can be detected in the environment and therefore also in foods. A companion publication (Part 2) is currently in-press and builds on the information in Safety Report 114 by outlining proposals for the assessment and management of exposures to radionuclides in food in existing exposure situations according to international standards.

Second International Conference on Applications of Radiation Science and Technology (ICARST-2022)

Carl Blackburn

The IAEA is organizing the ICARST-2022 for new dates in 2022. The conference will take a comprehensive look at the status of radiation science in academia and industry, as well as its ability to meet future challenges. Due to the continuing global response to the COVID-19 pandemic, the conference has been rescheduled and is now due to be held in Vienna, at the IAEA Headquarters from 22 to 26 August 2022. Further information can be found online (https://www.iaea.org/events/icarst-2022).

Vienna, Austria 22–26 August 2022

The 20th International Meeting on Radiation Processing (IMRP20)

Carl Blackburn

This international conference is held every two or three years and is one of the leading international events concerning the science and commercial application of radiation processing. The International Irradiation Association has rescheduled the dates for this international conference. For the first time, it will also include a meeting of the Phytosanitary Irradiation Forum. The meeting will be hosted in Bangkok from 7 to 11 November 2022 by the Thailand Institute of Nuclear Technology. Further information is available online (https://imrp-iia.com/).

The International Meeting on Radiation Processing (IMRP), Bangkok, Thailand, 7–11 November 2022.

Publications

2022

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