



Joint FAO/IAEA Programme
Nuclear Techniques in Food and Agriculture

Food & Environmental Protection Newsletter



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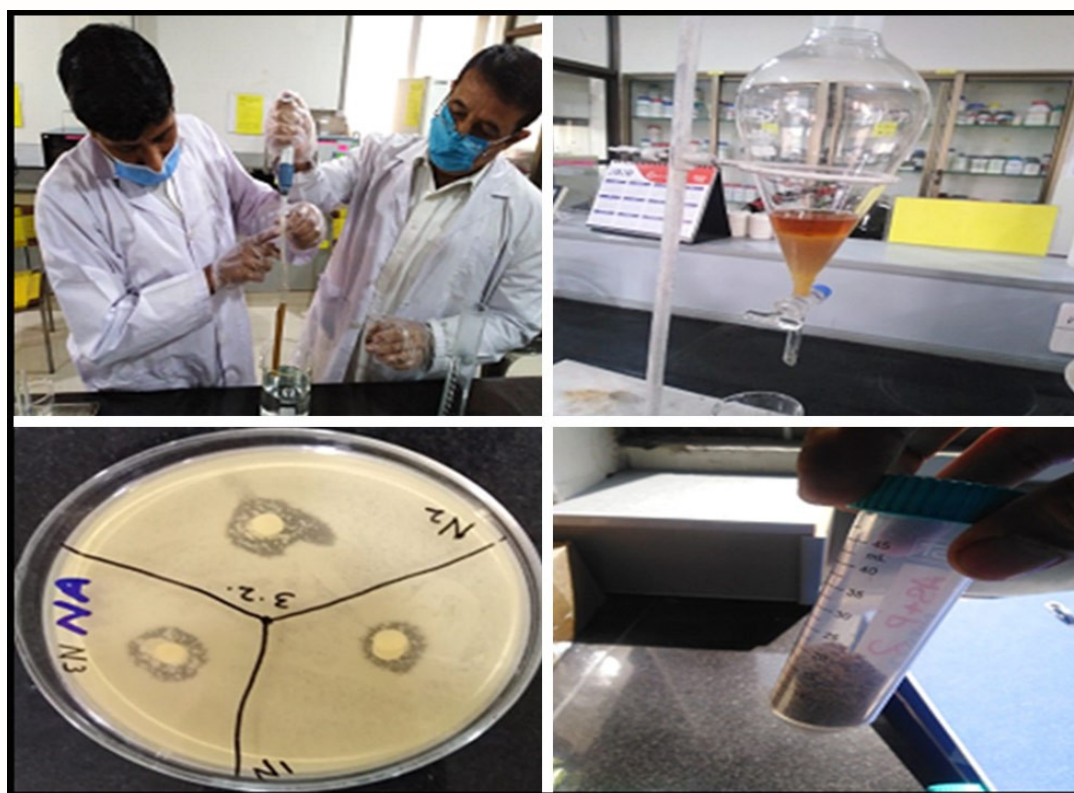
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To Our Readers



Microbiological analysis of irradiated foods by scientists at the Nuclear Institute for Food and Agriculture, Pakistan. (Photo courtesy of Dr Talat Mehmood, Senior Scientist, NIFA, Peshawar, Pakistan)

In line with our goal to help improve food safety control systems, protect consumers and promote trade, we recently reported¹ ISO 17025:2017 accreditation of food safety laboratories in seven Member States (Botswana, Mongolia, Namibia, Nigeria, Pakistan, South Africa and Uganda) following support provided through our capacity

development and research activities. As we celebrate this achievement with counterparts, we hope to help increase the number of accredited laboratories in our Member States. We are therefore continuing to support relevant activities such as enabling institutions to participate in proficiency testing schemes, including the provision of relevant training and

¹ <https://www.iaea.org/newscenter/news/food-safety-labs-in-seven-countries-attain-accreditation-with-iaea-and-fao-support>

transfer of analytical techniques, among others. So far this year, we have supported 27 institutions in 23 Member States in a range of proficiency testing schemes.

Our coordinated research projects (CRPs) are some of the mechanisms through which Member States have enhanced their capabilities and competencies. Two of our six CRPs-D52039 on “Development and Strengthening of Radio-Analytical and Complementary Techniques to Control Residues of Veterinary Drugs and Related Chemicals in Aquaculture Products” and D61024 on “Development of Electron Beam and X ray Applications for Food Irradiation (DEXAFI)” – have concluded. The final research coordination meetings (RCMs) for these two projects were held virtually from 1 to 5 March 2021 and from 7 to 14 April 2021, respectively. D52039 is elaborated in our feature article while another article in this newsletter discusses D61024.

The 3rd RCM for the CRP on “Integrated Radiometric and Complementary Techniques for Mixed Contaminants and Residues in Foods” took place virtually from 26 to 30 April 2021. Originally planned from 6 to 10 April 2020 in China, in partnership with the Chinese Academy of Agricultural Sciences, a virtual version became inevitable due to the COVID-19 pandemic. Another meeting that suffered a similar fate was the 1st RCM for the new CRP D52043 on “Depletion of Veterinary Pharmaceuticals and Radiometric Analysis of their Residues in Animal Matrices”, from 17 to 21 May 2021. This was initially planned for May 2020 but we are pleased to see that the investigators could meet at last virtually. We are happy to report that the 3rd RCM for the CRP D52040 on “Field-deployable analytical methods to assess the authenticity, safety and quality of food” took place from 7 to 18 June 2021.

A virtual consultancy meeting was held from 22 to 26 February 2021 to support the development of a new CRP on “Irradiation Technology for Phytosanitary Treatment of Food Commodities and Promotion of Trade”. Leading experts from Australia, China, Europe, Mexico and the USA took part in the meeting. Plans are under way to seek further approvals and launching of this CRP. Another virtual consultant meeting is planned to advise and develop a new CRP on “Nuclear Techniques to Support Risk Assessment of Biotoxins in Food and Related Matrices as well as Isotope-assisted pathogen detection”. This will take place from 16 to 20 August 2021.

Research, training and technology transfer have continued at the Food and Environmental Protection Laboratory (FEPL) amidst COVID-19 challenges. We are happy to report the development of analytical methods that can be used to: discriminate between Arabica and Robusta coffee and therefore address food fraud; study of the volatile fraction of fresh orange juices and also differentiate between organic and conventional orange juices using nuclear magnetic resonance. Additional work includes technology used for geographical differentiation of Hom Mali rice cultivated in different regions of Thailand using FTIR-ATR and NIR spectroscopy, as well as Volatilomic Profiling of Turmeric Teas by Dual-Detection HS-GC-MSD-IMS, among others. Since the last newsletter, the laboratory has implemented virtual training such as the course on Detection and Control of Organic Contaminants in Food – targeted testing held from 1 to 4 December 2020, as an activity under the Peaceful Uses Initiative project funded by the Government of Japan.

Our work requires partnerships. We are therefore happy to report the redesignation of the Centro de Investigación en Contaminación Ambiental (CICA), University of Costa Rica, as an IAEA Collaborating Centre for the period 2021–2025, and the designation of the Institute for Global Food Security (IGFS), Queen’s University Belfast, United Kingdom, as another collaborating centre on Food and Feed Safety and Authenticity for the period from 2021 to 2025.

Since the last newsletter, Food and Environmental Protection has supported more than 70 active or closing technical cooperation projects as well as the design of 35 national and regional food safety projects for the 2022–2023 technical cooperation cycle.

Finally, on staffing matters, we welcome Ms Aminata Faustmann who joined us as Team Assistant serving the FEPL, Soil and Water Management and Crop Nutrition as well as Animal Production and Health Laboratories. Another colleague, Ms Sofia Rezende, a PhD consultant at FEPL, will continue her work until December 2021.

James Jacob Sasanya

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<http://www-naweb.iaea.org/nafa/fep/index.html>
<http://www-naweb.iaea.org/nafa/fep/fep-laboratory.html>

Feature Article

Contributing to Safety of Aquaculture Products and Production - Conclusion of a Related Coordinated Research Project (CRP)

James Sasanya

A CRP D52039 on “*Development and Strengthening of Radio-Analytical and Complementary Techniques to Control Residues of Veterinary Drugs and Related Chemicals in Aquaculture Products*” initiated in June 2015 has now been concluded with the 4th and final Research Coordination Meeting (RCM) hosted virtually at the IAEA Headquarters in Vienna, Austria between 1 and 5 March 2021. The physical meeting was postponed from 2020 due to the COVID-19 pandemic. The CRP involved 15 research contract and agreement holders from: Argentina, Belgium, Brazil, Cameroon, Canada, Ecuador, China PR, Lebanon, Netherlands, Nigeria, Singapore, South Africa, Turkey and Uganda. Twenty-three participants from these Member States and the IAEA Secretariat attended this meeting.



Sampling Aquaculture production sites in South Africa for residues monitoring.

As a brief background and why the CRP was needed, aquaculture practice (fish and seafood farming) is becoming more widespread for the inexpensive and intensive production of protein rich foods. In the period from 2000 to 2012, intensive aquaculture production increased at an average annual rate of 6.2% from 32.4 million to 66.6 million tons; this has increased. Inevitably, chemical inputs such as veterinary pharmaceuticals and related substances are required to control aquaculture-related diseases and improve yields. Residues of such inputs, plus unintended

natural toxins (in aquaculture products and feeds) and contaminants at production sites, or effluents, pose public and environmental health risks and must be addressed. This calls for robust national regulatory frameworks underpinned by sound laboratories, to among others safeguard consumers and aquaculture production, and enhance international trade in aquaculture products. Research was therefore needed on analytical methods that will strengthen laboratory performance and nuclear and isotopic techniques play an important role. Research was also required to better understand the contamination of aquaculture production sites, with potential public and environmental health implications. This CRP would contribute to strengthening Member State analytical laboratories and national chemical residue monitoring programmes thus contributing to the improvement of food safety, better aquaculture production and management practices as well as enhancement of trade in aquaculture products. New analytical methods would be developed validated and transferred amongst Member States laboratories. The CRP would also contribute to the knowledge-base on contamination of aquaculture production systems.

The purpose of the final RCM was to review individual and group work done including application of research findings to national programmes and the mechanism for transfer of the technology to other Member States as well as determining impact of the CRP. The scope of the meeting and expectations covered power point presentations, discussions and provision of feedback from other participants, and preparation of a final report.

Findings: Overall, expected outputs and outcome were realized and the CRP has been of significant benefit to Member States. In total 36 methods were developed and validated; 36 standard operating procedures (SOPs) prepared, and 19 papers or scientific reports produced. These have contributed to improved analytical technology that is transferable and supports the testing and monitoring of residues and contaminants in aquaculture products and production sites. In eight countries, the established analytical methods have been applied to national residue monitoring programmes both for aquaculture products consumed locally including imports, as well as exports.

New radio receptor assay methods established under the project have been used for cost-effective determination of varying types and levels of residues in aquaculture products. This required primary validation of parameters for reliable testing of several veterinary drug residues in different fish. Three countries involved in this process, followed by transfer to two other laboratories that have in turn performed secondary validation. Further transfer to more countries is ongoing. In a related development, work on radio immunoassay for certain prohibited residues was also initiated.

Elsewhere, research was successfully conducted on the uptake and depletion of a C-14 labelled drug (sulfonamide) in certain fish. This information is important in appreciating the metabolism of the drug and provides a foundation for additional research on depletion of drugs in food animals. Such information is useful in the process of MRL setting.

The research generated information to help confirm that the presence of certain forbidden drugs/substances in food production, is not necessarily due to intentional use, rather due to natural occurrence. For example, a research contract showed that Semicarbazide (SEM) is not a conclusive marker for illegal use of the forbidden antimicrobial nitrofurantoin because SEM (a nitrofurantoin metabolite) was found to be naturally existing in shellfish. Established occurrence-species profile for SEM in shellfish may help in food safety regulatory decisions. Furthermore, analytical methods were developed and used to test mycotoxins in aquaculture inputs and associated matrices. This is critical in understanding exposure to mycotoxin through aquaculture.

Research findings also contributed knowledge and better understanding of chemical contamination/residue levels in water and related matrices (e.g. sediments) as well as potential implication on aquaculture production in general. Isotopic research demonstrated the presence of a wide range of human and veterinary pharmaceuticals in fish and water, as an indication of human/anthropogenic exposure. Further, $\delta^{15}\text{N}$ was used to demonstrate exposure of phytoplankton, shrimp and fish samples collected in three lakes of Córdoba province, to anthropogenic sewage. Thus, research work has helped demonstrate the usefulness of $\delta^{15}\text{N}$ in identifying pollution of water bodies whose water is useful in aquaculture production. Bioaccumulation and bioconcentration of certain drugs used in human medicine such as carbamazepine, in fish for human consumption, under field and controlled laboratory experiments, has been demonstrated through this CRP.

Research also contributed to enhanced laboratory competences according to international standards, such as ISO 17025 accreditation of the participating laboratories in Nigeria, South Africa and Uganda for example. This is crucial since these institutions hold important mandates of ensuring consumer safety and facilitating competitiveness of

exports. Meanwhile, 17 PhD, MSc and Post-Doctoral fellows in four Member States benefitted from this CRP.

The project resulted in some offshoots that may require follow-up. These included (among others), occurrence and effects of using water contaminated with antimicrobials in alfalfa plant cultures. The presence of ciprofloxacin and enrofloxacin in soil and their effect on legume symbiosis with *Rhizobium* species was briefly studied. Soil samples of alfalfa cultures irrigated with contaminated water from the Osmore river (Peru) were collected and analysed in Brazil where ciprofloxacin and enrofloxacin were detected. Another offshoot was work done to determine the concentrations and human exposure assessment of per- and poly-fluoroalkyl substances (PFAS) in farmed marine shellfish in South Africa. PFAS are organic compounds that contain a completely fluorinated hydrophobic carbon chain bonded to variable hydrophilic head. Seafood is a 'vehicle' for consumer exposure to these substances but data on their levels is limited thus necessitating some baseline information.

This CRP has also contributed to initiation of a new CRP on depletion of veterinary pharmaceuticals in food animals that was recently launched. Also, some research contracts have contributed to the development of national TC projects that will be launched in 2022, such as the introduction of monitoring practices for new residues (including pesticides) in aquaculture. More TCPs are expected out of this CRP.



Sampling Aquaculture production sites in South Africa for residues monitoring.

Forthcoming Events

Research Coordination Meetings of FAO/IAEA Coordinated Research Projects and Training Courses

Consultants Meeting (CM) on Nuclear Techniques to Support Risk Assessment of Biotoxins in Food and Related Matrices as well as Isotope-assisted pathogen detection, Vienna, Austria, 16–20 August 2021.

Training Course on the Use of Profiling/Fingerprinting Techniques to Determine Food Origin and Verify Food Authenticity, Seibersdorf, Austria, 16–27 August 2021.

Training Course on the Use of Stable Isotope Techniques to Determine Food Origin and Verify Food Authenticity, Seibersdorf, Austria, 11–22 October 2021.

Joint FAO, IAEA and Latin American and Caribbean Analytical Network Workshop on Food Contaminants Testing and Risk Assessment Programs, Panama City, Panama, 12–14 October 2021.

Second Research Coordination Meeting on the Implementation of Nuclear Techniques for Authentication of Foods with High-Value Labelling Claims (INTACT Food) (D52042-CR-2), Vienna, Austria, 1–12 November 2021.

First Research Coordination Meeting on Irradiation Technology for Phytosanitary Treatment of Food Commodities and Promotion of Trade (D61026-CR-1), Vienna, Austria, 22–26 November 2021.

International Meetings/Conferences

Codex Committee on Residues of Veterinary Drugs in Foods (CCRVDF25), Virtual, 12–19 July 2021.

Codex Committee on Pesticide Residue (CCPR52), Virtual, 26–31 July 2021

Latin American Symposium on Risk Assessment (LARAS2021), virtual event, opening session on 14 October 2021 and closing session on 26 October 2021, with plenary sessions on 18, 19, 20 and 25 October 2021.

International Conference on a Decade of Progress after Fukushima-Daiichi: Building on the Lessons Learned to Further Strengthen Nuclear Safety, Vienna, Austria, 8–12 November 2021.

Past Events

New IAEA Collaborating Centre in the UK to Support IAEA Research on Food and Feed Safety and Authenticity

Andrew Cannavan

The IAEA and the Institute for Global Food Security (IGFS), Queen's University Belfast, United Kingdom, have signed an agreement to formalize their ongoing collaboration on the development and implementation of analytical methods and mechanisms to enhance food safety and quality and promote food integrity worldwide. The IGFS was designated as a Collaborating Centre on Food and Feed Safety and Authenticity from 2021 to 2025. The designation ceremony took place during the IAEA Board of Governors meeting in March 2021.



William Gatward (UK Permanent Mission) receives the Collaborating Centre plaque on behalf of IGFS from IAEA Deputy Director General Najat Mokhtar.

The IGFS is a leading research provider to a range of stakeholders, including regulators, governments and multinational industries, tackling food contamination and adulteration and working on a wide range of food integrity projects on the international stage. As such, key aspects of its mandate closely parallel those of the Food and Environmental Protection Subprogramme, within the Joint FAO/IAEA Centre for Nuclear Techniques in Food and Agriculture.

The collaboration will leverage the complementarity of the nuclear analytical techniques developed and disseminated by the IAEA and the novel technologies and applications developed by the IGFS, to provide solutions to real-world food safety and security problems in both developing and developed countries. This will expand the scope of nuclear and complementary analytical methods available to IAEA Member State laboratories to underpin systems for the control of food contaminants, food authenticity and traceability.

The main activities in relation to the collaboration will include:

- Research on the detection of food and feed contaminants and adulterants using a range of spectroscopic and mass spectrometric platforms.
- Research into novel, transferrable methods of analysis for the authentication of high value food commodities.
- Research into data fusion techniques for food authentication, combining data from nuclear (stable isotope, NMR) and complementary (vibrational spectroscopy) techniques.
- To act as a training and reference centre and technical hub for laboratories in SE Asia and Africa. Provide advisory services, on-line training material and webinars on analytical methodology for the detection and control of food contaminants and adulterants.

Najat Mokhtar, Deputy Director General and Head of the Department of Nuclear Sciences and Applications, signed the agreement on behalf of the IAEA. “This will expand the scope of nuclear and complementary analytical methods available to IAEA Member State laboratories to support systems for controlling food contaminants, food authenticity and traceability,” she said.

Professor Chris Elliott, O.B.E., founder and first director of the IGFS and liaison officer for the Collaborating Centre, added that it was a wonderful accolade for his institute to be recognized by the IAEA for its efforts in food and feed safety and authenticity. “We are thrilled by the opportunities this agreement will provide for us to support the vital work of the Agency,” he said.

Professor Nigel Scollan, the current IGFS director, said: “We wholeheartedly support and share (the IAEA’s) vision of using the very best science and technology to make the world a better place and look forward to continued collaboration in the area of food and feed safety and authenticity”.

Utilizing their existing connections with institutes in Africa and South East Asia, the Collaborating Centre will support IAEA programmatic activities focused on building capacity for food safety and security in Member States through applied research, technology transfer and the provision of guidance and advice, with the ultimate goals of protecting consumer health, enabling trade in food commodities through certification of their safety and quality, and tackling issues such as the ever-increasing incidence of food fraud.



The Institute for Global Food Security, Queen's University Belfast, UK, which includes the well-equipped ASSET Technology Centre.

Redesignation of CICA, Costa Rica, as an IAEA Collaborating Centre

Britt Maestroni and Andrew Cannavan

The Food and Environmental Protection subprogramme is pleased to report that the Centro de Investigación en Contaminación Ambiental (CICA), University of Costa Rica, was redesignated as an IAEA Collaborating Centre for the period 2021–2025. The area of collaboration is in food and environmental quality and safety.

CICA consists of six laboratories working on various aspects of food and environmental quality and safety, including water analysis, analysis of pesticide residues and other organic compounds, analysis of greenhouse gases, ecotoxicological analysis, bioremediation of pollutants and isotopic applications. The institute has good training facilities and is well equipped for chemical and isotopic analysis in food and environmental samples.



Centro de Investigación en Contaminación Ambiental (CICA), University of Costa Rica.

CICA is a member of the Red Analítica de Latino America y El Caribe (RALACA), which is a regional, non-profit network of food safety laboratories and associated institutions initiated with the assistance of IAEA. RALACA brings together analytical laboratories in a number of Latin American countries to enhance and harmonize regional capabilities to address issues relating to food safety and environmental sustainability. RALACA currently has more than 50 member institutes in a number of countries in Latin America and the Caribbean, including Argentina, Belize, Brazil, Chile, Colombia, Costa Rica, Cuba, Dominican Republic, Ecuador, El Salvador, Guatemala, Honduras, Mexico, Nicaragua, Panama, Paraguay, Peru, Uruguay and Venezuela. Through its collaborating centre status CICA is in an excellent position to further support and contribute to RALACA, providing an ongoing liaison between the network and IAEA, a conduit for dissemination of IAEA TC and research outputs to the network, and providing services such as support for the RALACA website which were previously provided by IAEA in the formative stages of the network.



Training facilities at CICA.

Public health is the top priority for all national institutions dealing with food control and monitoring and surveillance are essential components of food safety control systems. The IAEA plays an essential role in strengthening such systems through the application of nuclear and related analytical techniques. This is especially important when normal capabilities are weakened by the disruptive effects on the integrity of the food supply and its inherent control measures caused by crises such as the current COVID-19 pandemic. The renewed collaboration with CICA will assist FEP in the implementation of its projects with the objectives of strengthening and supporting the capacities of the Latin American network of food safety and control laboratories while promoting multidisciplinary activities and coordinated research activities. Collaborating Centre activities will include research and development on contaminants in food and the environment in accordance with the FAO/IAEA Food and Environmental Protection Laboratory advice, focusing on problems specific to the Latin America/Caribbean region, and contributions to boosting the activities and growth of the RALACA network. The Collaborating Centre will also act as a technical hub for food safety in the region, creating and making available short online training courses on specific aspects of food quality and safety, and will provide advisory services on food integrity to the RALACA network of laboratories.

Recent Updates from the Analytical Network of Latin America and the Caribbean (RALACA)

Britt Maestroni

The Analytical Network of Latin America and the Caribbean (RALACA) is a non-profit network of laboratories and associated institutions, that aims to improve technical capabilities and encourage cooperation and communication between laboratories belonging to LAC region.

During the COVID-19 pandemic RALACA continued supporting the development and improvement of the capacities necessary to guarantee food safety and a sustainable agricultural environment in the LAC Region by fostering communication and sharing of data and analytical information.

In particular, in the period from January to May 2021, RALACA hosted a cost-free, four day distance learning course, implemented by the members of the ACADEMIA RALACA Committee, on “Introduction to residue and chemical contaminants analysis”. More than 200 participants attended the training and valued the training course as most relevant to their jobs. ACADEMIA RALACA was formed in 2020 as a new Committee and is planning to organize further training courses for the Latin American and the Caribbean (LAC) region based on specific request by regional laboratories. RALACA also hosted a webinar on 9 February on “Veterinary drugs in food of animal origin: from single-class to multi-residue LC-MS analysis. How to improve reproducibility and recovery of some analytes” prepared by Dr Serena Lazzaro from Italy.

On 18 May 2021, on the occasion of the 8th Latin American Pesticide Residue Congress, RALACA hosted its 4th general meeting, and prepared a generic presentation on the outputs of RALACA during 2019–2021. On 21 May, RALACA, in conjunction with EFSA, delivered a free course on the topic, data sharing. More than 120 participants from the LAC region attended the course and showed a deep interest through a series of interactions in the Q&A session. The FEPL helped with the technical organization of the course and preparing the agenda.

The RALACA board would like to encourage the active participation of all RALACA Institutions in the work of the different Committees and invite all Institutions to host online presentations of their own activities and infrastructure. Please contact the RALACA board for further information (ralacaboard@gmail.com).

Networking and Working Towards Accreditation

James Sasanya

As part of the African food safety network (AFoSaN), we have continued to provide support to testing laboratories in proficiency testing. Through the Progetto test veritas PT scheme 24 institutions in 21 countries are participating in PTs including veterinary drug and pesticide residues, mycotoxins, histamines, toxic and essential metals among others in a range of foods including meat (poultry, beef, swine), milk, eggs, cereals and nuts, fish/shrimps, fruits and vegetables; coffee, cocoa, honey and soft drinks. In a separate regionally tailored scheme provided the National Metrology Institute of South Africa, 18 countries/institutions are also participating Aflatoxins in peanut slurry and milk, contaminants in water and targeted regional fruits, among others. Participants will share experiences following the PTs and help each in addressing loopholes.

Virtual Training Course on the Detection and Control of Organic Contaminants in Food – Targeted Testing

Britt Maestroni and Andrew Cannavan

The control of unwanted chemicals in food, such as residues of pesticides or veterinary drugs used in food production, or natural contaminants such as mycotoxins, remains an area of high importance to Member States. Adequate monitoring and surveillance procedures must be in place to enable rapid and effective response to food safety incidents and emergencies. To be effective, a food control system must include the ability to apply targeted monitoring using validated, quality-controlled analytical laboratory methods to detect, quantify and confirm the presence of contaminants in food products. The scientific data generated enables decision makers to apply risk-based controls to protect consumer health, facilitate trade and respond effectively to food safety incidents and emergencies.

A variety of analytical techniques can be applied to test food commodities for contaminants. These include both nuclear-related and non-nuclear physico-chemical techniques. Amongst the most useful and widely applied techniques are those employing liquid- or gas-chromatography with detection by triple-quadrupole mass spectrometry. These techniques, often used in isotope dilution format with stable-isotope



RALACA/EFSA post-congress course on data sharing, hosted on 21 May through the LAPRW web platform.

labelled compounds as internal standards, provide the sensitivity, precision and the capability for confirmation of identity of a target analyte necessary for food control. Expertise in the principles of the instrumentation and practical application of the analytical methods is a prerequisite for the generation of reliable results and data.

To help meet these needs, a virtual training course on the Detection and Control of Organic Contaminants in Food – Targeted Testing was held from 1 to 4 December 2020 as an activity under the Peaceful Uses Initiative project ‘Enhancing Capacity in Member States for Rapid Response to Food Safety Incidents and Emergencies’, which is funded by the government of Japan.

The virtual course was a partial replacement for the hands-on course originally scheduled to be held at the FAO/IAEA Laboratories at Seibersdorf, Austria, from 20 to 30 April 2020. It was necessary to postpone and reformat the course because of travel restrictions and national responses to the COVID-19 pandemic. In order to maintain support to Member States, the original course content was divided into two parts: theoretical aspects that would have been covered in classroom lectures, and practical laboratory sessions. The virtual course in December 2020 focused on the theoretical and background aspects of the detection and control of organic residues and contaminants in food. To accommodate participants in various different time zones across the world, the course was implemented using recorded lectures that were available to the course participants offline between 1 and 4 December, with a ‘Teams’ online question and answer session on 7 December. The lectures were prepared as narrated power-point presentations by four guest lecturers; Dr Veronica Cesio, Associate Professor of Pharmacognosy & Natural Products in the Faculty of Chemistry of the University of the Republic (UdelaR), Montevideo, Uruguay, Dr Suzanne Eckroth, a chemist from the Swedish Food Agency, Dr Ivan Pecorelli, Head of the Contaminants Unit at IZSUM, Italy, and Dr Serena Lazzaro, Italy, who has previously collaborated with FEPL in the delivery of training on food contaminant control. The training materials comprised 19 lectures in four topics; organic contaminants in food, testing methodologies, analytical instrumentation and planning of experiments, method validation, and quality control measures, and were made available to course participants via IAEA NUCLEUS.

The virtual training course had 24 participants from institutes in 24 developing countries (Algeria, Argentina, Bangladesh, Brazil, Chad, Ecuador, Ethiopia, Ghana, India, Jordan, Lao P.D.R., Lebanon, Myanmar, Namibia, Oman, Pakistan, Paraguay, Seychelles, Sri Lanka, Thailand, Tunisia, Uruguay, Uzbekistan and Viet Nam). All participants completed the course and passed a multiple-choice examination, which was available offline on 14 December 2020. Successful completion of the virtual course made the participants eligible for a second course focusing on the application of chromatography – mass spectrometry techniques to detect, quantify and confirm the presence of organic chemicals such as pesticides and

veterinary drug residues in foods. It was intended that this second course would be a hands-on event held in the FEPL at Seibersdorf, but unfortunately the ongoing disruption caused by COVID-19 has made this impossible, and the second course was also reformatted to be held as a virtual event in June 2021.

The ongoing COVID-19 pandemic has highlighted the importance of building resilience in food control systems in Member States to the disruption caused by events such as pandemics, extreme weather caused by climate change and other crises and emergencies. The virtual training course strengthened the participants’ understanding of the underlying principles of analytical methodologies and chromatographic–mass spectrometric techniques to underpin food safety control systems and to respond to food safety-related incidents or emergencies by providing data on the presence and levels of targeted contaminants in food commodities.

The 14th Session of the Codex Committee on Contaminants in Foods, 3–7 May 2021

Carl Blackburn

Mr Blackburn participated at the 14th session of the CCCF, one of the major food safety and quality standard setting committees of the Codex Alimentarius Commission. Due to the COVID19 pandemic, the 14th Session was postponed from May 2020 to May 2021. The meeting was held virtually from 3 to 7 May 2021 with the meeting report adopted on 13 May.

The Food and Environmental Protection Section provide a report on Joint IAEA/FAO programmatic work because it is of interest to food control authorities that are represented at the CCCF. Also, the IAEA is responsible for international radiation safety standards and Codex is the international body for food standards. Therefore, the Joint FAO/IAEA Centre has a role in keeping Codex and the CCCF informed of a project concerning radioactivity in food and the development of international radiation safety guidance. Readers will also recall that at the previous meeting of the CCCF in 2019, a proposal for the formation of a CCCF electronic working group (EWG) on radioactivity in food in non-emergency situations was also accepted by the meeting. The CCCF established an EWG with Mr Blackburn providing any necessary technical input and support to the production of the discussion document for consideration by the CCCF.

At the CCCF meeting in May 2021, formal remarks were given on Joint FAO/IAEA programmatic activities of interest to the CCCF with reference to a written submission

which is available online². Mr Blackburn summarized recent activities of interest to the committee related to IAEA technical cooperation projects, international research projects and research laboratories. He referred to the ongoing international work on radionuclides in food, feed and drinking water and linked it with the information presented in the EWG discussion paper produced for consideration by CCCF. He mentioned that work at international level in this area is currently developing methodologies that can be used to produce criteria to assess these radionuclides in food. He thanked the EWG, the Chairs of the EWG and the Codex secretariat for this excellent discussion paper.

The European Union, as Chair of the EWG, introduced the item on radionuclides in food, drinking water and feed in non-emergency situations. Following information provided by Mr Blackburn, the 13th session of the CCCF had agreed that explorative work should be undertaken on food safety and trade issues associated with radionuclides in food (including drinking water) and feed in non-emergency situations. The EWG had produced a discussion paper to increase the understanding of the presence of radioactivity in food and feed in non-emergency situations and to enable CCCF to take an informed decision on possible follow-up actions at this session. The EWG Chair indicated that in the EWG comments were made as regards the need to have a stronger case made to CCCF to work further on this issue, to clarify the relation between the work to be possibly undertaken by CCCF and work already and planned to be undertaken by international organizations, and to clarify the terms used and to ensure consistent use these terms. The discussion paper³ as presented to the CCCF takes into account these comments. The EWG Chair noted that in the discussion paper it is concluded that naturally occurring radionuclides (i.e. mainly ⁴⁰K, ²¹⁰Po, ²¹⁰Pb, ²²⁸Ra and ²²⁶Ra) are found in many different foods and tend to give radiation doses higher than those provided by artificially produced radionuclides (such as ¹³⁴Cs, ¹³⁷Cs, ¹³¹I and ⁹⁰Sr) in situations not affected by a nuclear emergency situation in the past, but no specific safety problem for food, feed or drinking water due to the presence of naturally occurring radionuclides had been identified. Furthermore, no international trade issues had been identified due to the presence of naturally occurring radionuclides in food, feed and drinking water.

After discussions, the CCCF agreed that no further work is required to be done by CCCF at this time given that naturally occurring radionuclides in food, feed and water do not seem to be an issue for food safety and trade. It also agree to welcome the offer of IAEA to elaborate with the collaboration of FAO and WHO an informative document for the food safety regulators community, providing the state

of the art of natural radioactivity in food, feed and water. The committee also asked to be kept informed of any developments in the field of naturally occurring and artificially produced radioactivity, in particular on the FAO/IAEA/WHO work to develop methodologies that can be used to produce criteria with which to assess radionuclides in food.

Food Integrity Online Conference 19–23 April 2021: Panel Discussion on the Biggest Challenges and Solutions in Food Fraud and Analysis

Simon Kelly

The European Framework 7 Integrated Project “FOOD INTEGRITY” had its final conference in Nantes in 2019. The success of the annual FOOD INTEGRITY conferences, over the project’s 5-year duration, led to “New Food” Magazine continuing to host the conference with the first post-project conference scheduled for 2020 in London, UK. Unfortunately, the event was cancelled due to the COVID-19 pandemic and restrictions on global travel. Consequently, the conference organizers launched the Food Integrity 2021 conference as a fully virtual, interactive five-day event, retaining many of the topics of the original 2020 agenda and including additional topics, covering food integrity, food safety, food regulations, supply chain vulnerability and authenticity, herb and spice authenticity, food fraud, laboratory testing, allergens, consumer labelling concerns and technology updates. The conference explored the global food safety and authenticity challenges that the food industry is facing, with leaders from the food and beverage sector sharing and debating the issues, the lessons learned and possible solutions. More than 75 expert speakers, 1000 participants from across the globe, and exhibitors from industry attended the virtual event.

During the conference, on the 21st April 2021, Mr Simon Kelly participated in a panel discussion on “The Biggest Challenges and Solutions in Food Fraud and Analysis” moderated by Prof Chris Elliott, Queen's University Belfast (QUB), UK. The other panel members were Dr Franz Ulberth, European Commission DJ Research; Professor Michele Suman, Barilla; Dr Clare Menezes, Global Director of Food Integrity, McCormick; and Dr Mario Gadanho, Thermo Fisher Scientific. The discussion attracted 265 viewers from the conference participants and focused on:

- What are the current challenges in food fraud?

² http://www.fao.org/fao-who-codexalimentarius/sh-proxy/en/?lnk=1&url=https%253A%252F%252Fworkspace.fao.org%252Fsites%252Fcodex%252FMeetings%252FCX-735-14%252FWDs-2021%252Fcf14_04e.pdf

³ http://www.fao.org/fao-who-codexalimentarius/sh-proxy/en/?lnk=1&url=https%253A%252F%252Fworkspace.fao.org%252Fsites%252Fcodex%252FMeetings%252FCX-735-14%252FWDs-2021%252Fcf14_14x.pdf

- Testing techniques to combat fraud and adulteration.
- Extending authenticity analysis capabilities.
- What is targeted and non-targeted analyses and how can they help?
- Validating analytical techniques.

Mr Kelly introduced the Food and Environmental Protection Laboratory (FEPL) and explained its role in applying nuclear and complementary techniques to assist Member States to improve their food safety and authenticity control systems. Mr Kelly explained how this is achieved by carrying out applied and adaptive research, transferring methods and building capacity through provision of equipment (via technical cooperation projects), training and advice, and also providing funding for coordinated research in stable isotope analysis and other complementary techniques. He explained that the ultimate goal is to protect consumers from food fraud and any of its unintended consequences that often impact on food safety and human health, especially in developing countries, as well as helping Member States protect and promote their food products and facilitate international trade. Some of the major challenges in food fraud relate to verifying claims of production and geographical origin, which add value to food products and incentivize fraud, but at the same time can be some of the most difficult to address analytically, for example, confirming organic (or bio) cultivation and rearing, claims of wild versus farmed, free-range, fair-trade, Protected Geographical Indications, and so on. This is because natural products are inherently variable and for comparative assessments of authenticity and verification of added value it is essential to have databases compiled from genuine foods, characterizing the variability within those products. Mr Kelly suggested that openly sharing good quality data from standardized methods is essential, but is not commonplace due to several factors, such as intellectual property issues, database curation, quality control and cost-related sustainability issues. Regarding testing techniques, there is a challenge and an opportunity to make testing cheaper, faster and more portable through the use of hand-held techniques such as vibrational spectroscopy, especially for presumptive screening in supply chains. This is also very important in developing economies to make testing more accessible and routine. Again, linked with this, is the challenge of databases for comparative testing and the use of multivariate statistical models. Mr Kelly acknowledged the challenge of using sophisticated multivariate mathematics in any kind of enforcement scenario. He also suggested that, though it may be unrealistic for financial reasons, more routine authenticity surveillance of samples from retail food outlets at all levels, should be completed to identify fraud and potential safety issues.

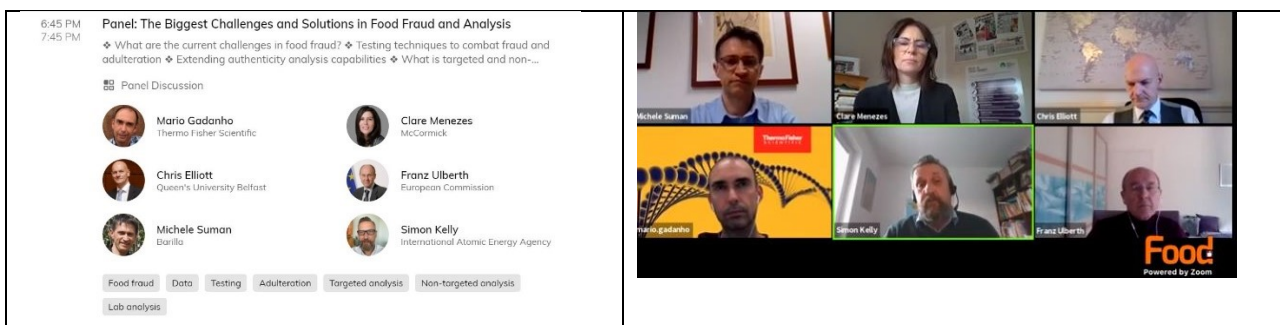
Elaborating on the importance of databases compiled from genuine products and openly sharing data, Mr Kelly explained that economically motivated adulteration of food comes in many forms. For example, the addition of

melamine to milk powder or Sudan dyes to spices is readily detected and there is no acceptable threshold for their use in foods destined for human consumption. Consequently, databases are not required to assess the natural variation because these chemicals should not be present at any concentration. However, there are many food authenticity analyses that are comparative and rely on databases, such as subtle extension of foods with chemically identical equivalents, or verifying added-value claims such as organic, free-range or protected denomination of origin. Databases of genuine products are essential to assess natural variation in the chemical, elemental or isotopic composition of a food. That variation might be linked factors such as year of production, climatic conditions, and the location of production, which is linked to different environmental and geological conditions. Variation due to differences in agricultural practice, variety of crop or breed of animal, or permissible technological processing effects all need to be considered and characterized. A great deal of effort is required to collect and analyse genuine food samples, to ensure that when the measurements are used in mathematical models of the authentic products characteristics, reliable assessments of authenticity of suspect adulterated or substituted foods can be made. The required effort means that databases are expensive and time consuming to compile. This can present a major stumbling block to setting up authenticity testing, especially for developing Member States. Consequently, after providing equipment and training there can still be a significant delay, while databases are compiled, before routine authenticity testing can be implemented. Moreover, if a country is a net importer of food it simply may not be feasible to obtain authentic food samples from overseas to verify the origin of imported food. Even if databases can be initiated through specific research projects, curating, and maintaining them can prove to be a major challenge. Over the years, efforts to establish global databases have faced major issues related to IP rights, data sharing, curation & sustainability amongst existing private or open databases for product protection. Mr Kelly proposed that ultimately what is needed is a major commitment for the compilation of a global, independent, trusted, reference dataset that Member States could routinely use to compare suspected fraudulent and counterfeit foods against.

Regarding FEPL's role in making food authenticity testing more accessible to developing countries, Mr Kelly explained that the food we eat comes from national and often global supply chains and indeed significant amounts of food are sourced from developing countries. These exports are very important for low and medium income country (LMICs) economies and there are often compliance issues, which add to pressures to ensure exports are of required standards and don't become the subject of export bans or national reputational damage due to fraud issues. At the same time, there may be limited laboratory resources available to check the quality and authenticity of food products. Mr Kelly went on to describe how FEPL addressed this issue through an international coordinated research project (CRP) that started

four years ago bringing together researchers and instrument suppliers from both developed and developing economies, to work on vegetable oil and milk authenticity using hand-held, field-portable and bench top systems based on molecular spectroscopic techniques that provide end-users with rapid, low-cost, and easy-to-use “screening” techniques. These technologies, such as near-infrared, mid-infrared, and Raman spectroscopies; ion-mobility spectrometry; multi-spectral imaging; and X ray fluorescence spectroscopy, can be used by stakeholders across the entire food supply chain to quickly screen samples for uncharacteristic anomalies or safety issues. Within the project a spectral library has been established for the users and is hosted by the Walloon Agricultural research Centre in Belgium. A technical sub-project has also produced sealed milk powder and vegetable oil inter-laboratory calibration

materials. A Russian statistics group participating in the project produced a free software add-in for excel that performs a number of multivariate analyses such as principal component analysis and soft independent modelling by class analogy to process spectral data and build models to detect adulterated and counterfeit foods. Another participating institute, QUB, organized a global inter-laboratory study involving twenty-seven institutes that demonstrated the potential of a low-cost handheld near-infrared spectrometer to detect adulteration of oregano herb. Mr Kelly concluded that all of these activities undertaken by FEPL and its collaborators are helping to provide low-cost sensors, training, databases, and free statistical tools to permit LMICs to conduct meaningful screening of food adulteration and related food safety issues.



Online panel discussion on “The Biggest Challenges and Solutions in Food Fraud and Analysis”.

International Food Irradiation Symposium 9–11 March 2021

Carl Blackburn

The symposium was fully dedicated to food irradiation and included legislation, food quality, dosimetry and technical presentations on practical applications for the food industry and also phytosanitary uses. It was held online over three days in March. Hosted and organized by our IAEA Collaborating Centre at Aerial in France with Ion Beam Applications S. A. (IBA) and Bühler Group, the symposium was implemented with the support of the Joint FAO/IAEA Centre for Nuclear Techniques in Food and Agriculture and the International Irradiation Association.

The sessions on food quality provided a balanced overview of the possibilities and limits of the technology. The pros and cons of food irradiation were discussed with practical examples. A session on regulation began with a global overview from Mr Blackburn; this was followed by presentations that covered legislation in the European Union, the Americas (with a focus on the USA) and Asia (with a focus on China). Practical workshops were also delivered online. Bühler Group presented on low energy electron beam treatment of coriander as an example of a novel technology for microbial decontamination. The use of

high energy X ray irradiation to provide phytosanitary Treatments was illustrated by Aerial and IBA using mango irradiation as the practical example and highlighting the important dosimetry aspects of the process. Information provided on the practical aspects of food irradiation and process control built on hands-on experience and new tools to support good irradiation practices included the use of computer simulations. For example, IBA showed the potential of Monte Carlo simulations to study the impact of density and spatial arrangement of products for high energy beam irradiation and the Bühler Group illustrated some of the challenges in simulating low energy electrons for surface irradiation treatments, as an example of low energy beam Monte Carlo approaches.

This was a very well-organized symposium, and virtual coffee breaks enabled participants to interact informally and discuss the different aspects of food irradiation. One of the interesting aspects was that there is a great deal of support for the establishment of an international food irradiation network. The possibilities for such a network are being investigated further. Through our involvement with this symposium we were able to support the online participation of a number of participants and help ensure that the symposium was a truly global event.

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 A. Cannavan
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
D61025	Innovation of Irradiation Technologies on Surface Treatment of Food Commodities	C.M. Blackburn
D61026	Irradiation Technology for Phytosanitary Treatment of Food Commodities and Promotion of Trade	C.M. Blackburn

Consultants Meeting for the CRP Concept “Nuclear Techniques to Support Risk Assessment of Biotoxins in Food & Related Matrices Nuclear as well as Isotope-assisted Pathogen Detection” 16–20 August 2021

James Sasanya

The purpose of the event is to discuss the subject matter including feasibility of the CRP concept; develop a detailed research proposal and provide relevant recommendations including on: implementation strategy; participants; partnerships and resources.

First RCM for D52043 “Depletion of Veterinary Pharmaceuticals and Radiometric Analysis of their Residues in Animal Matrices”, Virtual, 17–21 May 2021

James Sasanya

The 1st RCM was held virtually from 17 to 21 May 2021 and attended by 25 participants from Bangladesh, Brazil, Burkina Faso, Canada, Chile, China, Costa Rica, Iran, Islamic Republic of, Morocco, Pakistan, Korea, Republic of, Sudan, Uganda, the USA and Uruguay. Mr Sasanya was both scientific secretary and event moderator.

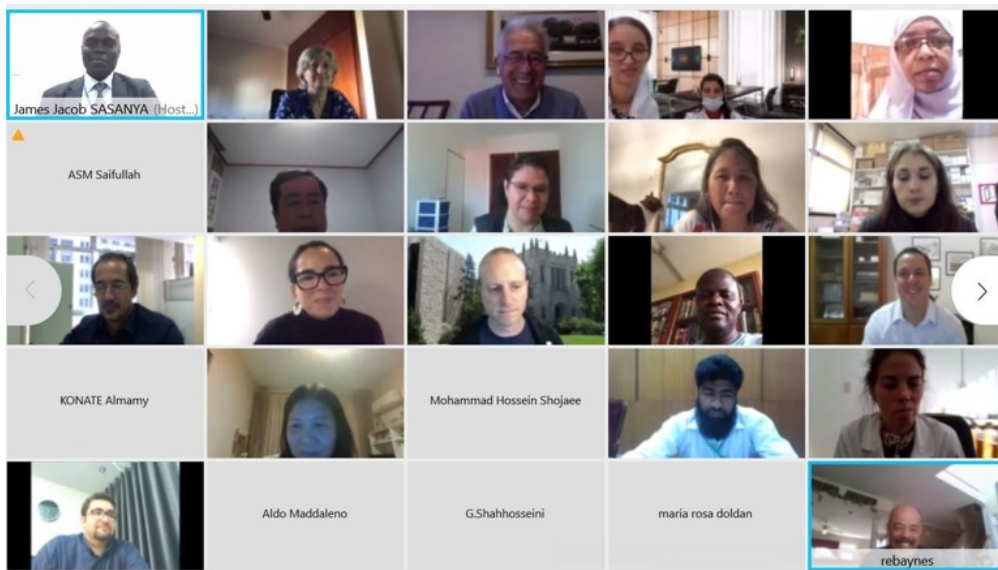
Participants deliberated on a range of topics; received feedback from others and prepared/finetuned workplans. Collaborative strategies as well as research needs were

discussed and solutions identified for individual national research plans. The participants shared various experiences and material.

The scope includes depletion of a range of veterinary drugs including amoxicillin, doxycycline, ivermectin, levamisole, emamectin benzoate, ampicillin, ivermectin, permethrin, diminazene, imidocarb, flavophospholipol, enramycine and diaveridine, ethion, chlorpyrifos, fipronil, florfenicol, sulfadiazine, trimethoprim, oxytetracycline as well as antiparasitics such as buparvaquone, ethidium bromide, melarsenoxide cysteamine a range of food-producing animals including fish, poultry, sheep and goats etc.

One of the specific research activities will involve depletion of amoxicillin and its toxic metabolites amoxicilloic acid in poultry and development of methodology for the evaluation of their metabolism using both accelerator mass spectrometry and liquid scintillation counting (to determine mass balance). In-vitro metabolism will be studied using animal hepatic microsome and hepatocytes to generate metabolic profile. Meanwhile, progress has been reported on disposition and residue depletion of Diaveridine (DVD) in pig and broiler based on radiometric analysis. So far, [3H] diaveridine has been synthesized from 3-bromo-4, 5-dimethoxybenzaldehyde. A radioactive tracing technique coupled with LC-MS-IT-TOF method was developed for discovery and identification of DVD and its metabolites in swine. Tritium-labelled DVD was administered orally to the swine followed by collection and analysis of urine, feces, plasma, bile and edible tissues.

Finally, some of urgent need were identified for most CRP participants, including sourcing and procurement of radiolabelled molecules as well as certain associated equipment.



Some of the participants during the 1st RCM for the CRP D52043.

Third RCM for the CRP D52041 “Integrated Radiometric and Complementary Techniques for Mixed Contaminants and Residues in Foods” Virtual, 26–30 April 2021

James Sasanya

The 3rd RCM for the CRP was held virtually from 26 to 30 April 2021 and attended by 22 Scientists from Botswana, China, Colombia, Ecuador, North Macedonia, Nicaragua, Pakistan, Peru and Uganda. Others included Italy, South Africa, Spain, and the USA. For IAEA staff Ms Malgorzata Rydeng provided administrative support while and Mr James Sasanya was scientific secretary and event moderator.

The purpose of the RCM was to review work done since the 2nd RCM in held in Botswana, and plan for the third phase of the project; to guide researchers; discuss needs and challenges and to identify ways forward. New opportunities the researchers could evaluate would also be discussed.

The meeting noted that innovative research on radio receptor assay has been advanced. This includes characterization of in-house developed receptors reported earlier; new method development for enhanced affinities of multi-contaminants for their respective receptors; standardization of multi-contaminants including tetracyclines, aflatoxin (AFL) M1, chloramphenicol (CAP), gentamicin analysis in milk. The suitability for screening of AFL B1, B2 and CAP in wheat. Plans are underway to upscale production of these receptors.

Work was also reported on development, optimization and validation of isotopic LC-MS/MS methods for

determination of veterinary drug residues, OP- pesticides and mycotoxins in milk, urine and meat. Application of this method for routine testing is ongoing. Additional work initiated on non-targeted screening of a wide range of molecules (up to 600) fruits/vegetables by high-resolution mass spectrometry has been initiated with improvement is required.

The validation of an isotopic LC-MS/MS analytical method developed for a group of 29 veterinary drugs and pesticide residues in animal products has also been advanced. Further, two analytical methods were developed and validated for multi-residue determination of pesticides and veterinary drugs (including antimicrobials and antivirals) in eggs and chicken. One scientific paper was published, and one master dissertation completed under this research contract. With regard to research on simultaneous determination of mixed residues/contaminants (veterinary drugs, pesticides and mycotoxins) in chicken liver by dispersive liquid-liquid microextraction/LC-MS/MS, a technique has been developed as a greener sample preparation and/or preconcentration method. Further improvement using stable isotopes is underway.

A multiresidue method for quantification and confirmation of antimicrobial residues and mycotoxins in animal waste was developed and validated. Part of this work is relevant to matters of antimicrobial resistance (AMR). Establishing potential correlation of residues levels in food and waste is being evaluated. Finally, improvement and expansion of a multi-class/analyte method for mycotoxins and pesticides in quinoa is ongoing using a wide range of stable isotopes. The CRP is generally affected by the COVID-19 pandemic as laboratory activities couldn't be implemented as planned during the 2nd RCM.



Some of the participants at the 3rd RCM for the CRP D52041.

Final Research Coordination Meeting of CRP D61024: Development of Electron Beam and X ray Applications for Food Irradiation, Virtual, 7–14 April 2021

Carl Blackburn

The fourth and final Research Coordination Meeting (RCM) of Coordinated Research Project (CRP) on the Development of Electron Beam and X ray Applications for Food Irradiation (DEXAFI) was held as a virtual meeting. This CRP involves 16 participating institutions from 13 countries (China, Egypt, France, Indonesia, Japan, Republic of Korea, Pakistan, Poland, Portugal, Syrian Arab Republic, Thailand, USA, Viet Nam). The overall objective of the research is to accelerate the development and facilitate the implementation of practical techniques to irradiate food and agricultural products using electron beam (EB) and X ray ionizing radiations.

A publication in the IAEA TECDOC series is being produced. The research results achieved at the close of the CRP were reviewed during the meeting in terms of the expected research outputs that were agreed at the start of the project in 2015. Publications and presentations on new concepts of electron beam and X ray machines customized for food irradiation, included reports on low energy electron beam irradiation of foods such as spices (irradiation using < 300 keV electrons); the use of cabinet X ray food irradiation to ensure “clean-food” for the immune-compromised in hospitals (low energy X ray irradiation of 160 kV energy); the development of a cabinet X ray irradiation system for phytosanitary irradiation (low energy X ray of 160 kV). There is renewed interest in developing and using low energy beam devices. There is growing interest in the industry to use low energy electron beams as is shown by the food engineering group Bühler and its newly developed in-line electron beam surface decontamination unit. Meeting participants commented that

they have also noticed that there are other companies now working on low energy X ray applications and several potential end users of such a device have already been identified through research activities. There seems to be a commercial opportunity for low energy X ray irradiators.

The CRP also developed improved dosimetry protocols for low energy X ray food irradiation, and validation of existing dosimetry. In addition, new imaging tools to support product dose mapping for heterogenous products were developed in China and Viet Nam. These new technologies can be used to quickly determine mass thickness of items before irradiation processing, and predict electron beam dose distributions. A number of CRP participants also produced scientific publications detailing examples of irradiation by electron beam or X ray of specific categories of food with pre- and post-irradiation requirements. In addition to specific categories of food, there are valuable research outputs related to phytosanitary treatments. For example, comparisons of dose rate studies and of the use of electron beam, X ray, and gamma irradiation. Many participants also worked directly with industry to assist in the commercial development of food irradiation.

Research in France investigated the issue of activation of radioactivity by the choice of the maximum energy of the electron beam that produces X rays. The higher the energy, the more efficient the process (X ray production and food treatment), but it is essential to consider the possibility of photonuclear activation which can lead to the production of radioactive nuclei inside the irradiated material. International food standards give a maximum X ray energy of 5 MeV for food irradiation, but some countries allow 7.5 MeV X rays. Research to date has conclude that food irradiated with X rays up to 7.5 MeV is safe and efficacious. Any induced radioactivity is short lived, and the levels are very low, well below natural radioactivity levels in non-irradiated food. Levels are so low that simulation methods are needed to estimate activation levels as the radioactivity is difficult to measure. The research in France under this CRP has confirmed this using up-to-date modelling and practical experiments. The major finding was

that modelling can predict levels of induced radioactivity and this research has produced a method to validate simulations. Mr Blackburn has asked for international food and phytosanitary standards to be revised to increase the maximum energy for the X ray irradiation of food from 5 to 7.5 MeV. The Codex Alimentarius Committee on Food Hygiene have agreed to consider this as a potential future work item at their next meeting. The international phytosanitary standards of the International Plant Protection Convention also give a maximum X ray irradiation energy of 5 MeV for phytosanitary irradiation treatments. The relevant standard is being revised and has also been redrafted to propose an increase to the maximum energy for X ray irradiation of food commodities from 5 to 7.5 MeV.

Development of a New Coordinated Research Project on Phytosanitary Irradiation Treatments, Virtual, 22–26 February 2021

Carl Blackburn

A virtual was held to develop a new IAEA research proposal for Coordinated Research Project (CRP) on Irradiation Technology for Phytosanitary Treatment of Food Commodities and Promotion of Trade. Leading experts from Australia, China, Europe, Mexico, and the USA were invited

to advise the secretariat about the CRP proposal and to develop its scope and content.

Phytosanitary treatments are official pre-shipment or quarantine processes recognized internationally and used by National Plant Protection Organizations to mitigate biosecurity risks associated with plants or plant-based products. Several techniques have been developed and these include treatments based on temperature, chemical fumigation and ionizing radiation. Ionizing radiation is a relatively recent commercial treatment. Although commercial irradiation treatments have been used on a continual basis over the past 25 years, its use for trade in fresh fruits and vegetables has increased significantly since 2006. More irradiation facilities are being built to treat foods mostly in the Americas, and Asian and Pacific countries.

A proposal for a new CRP was produced at the meeting. The agreed overall objective of the research was to validate the radiation doses proposed for at least five generic phytosanitary irradiation treatments using large numbers of regulated pests. Factors that might affect treatment efficacy will also be investigated. The specific research activities should focus on finding examples of the most radio-tolerant species in several pest groups important for trade (Table 1) and conducting tests with large numbers of individuals of these species to confirm efficacious irradiation doses. The proposal will be submitted to the appropriate IAEA Committee for their consideration and we hope to make an announcement soon on a new CRP in this area.

TABLE 1. PROPOSED GENERIC MINIMUM TREATMENT DOSES FOR IMPORTANT PEST GROUPS

Proposed generic dose (Gy)	Regulated pest group
150	Weevils of the family Curculionidae
200	Leaf miners of the family Agromyzidae
250	Mealybugs of the family Pseudococcidae
250	Scale insects of the family Diaspididae
250	Eggs and larvae of the order Lepidoptera
300	All regulated insects except for pupa and adult Lepidoptera
400	Pupae of the order Lepidoptera
400	Mites of the family Tetranychidae
500	Mites in families other than Tetranychidae

Since the adoption of the International Plant Protection Convention (IPPC) Guidelines for the Use of Irradiation as a Phytosanitary Measure⁴ (ISPM No. 18), a great deal of commercial experience has been gained to confirm the efficacy and utility of ionizing radiation. Research coordinated by the Joint FAO/IAEA programme over a series of four coordinated research projects (CRPs) spanning

from 1986 to 2015 has contributed to the 16 phytosanitary irradiation doses for irradiation treatments currently in the annexes of the IPPC International Standard on Phytosanitary Treatments for Regulated Pests⁵. The most recent CRP (D61028) ended in 2015 and the results were published as a journal special issue⁶. It developed irradiation doses against 34 species in 10 insect families, 3 mite families and 1 snail

⁴ IPPC, Guidelines for the Use of Irradiation as a Phytosanitary Measure ISPM No. 18, IPPC 2003; <https://www.ippc.int/en/core-activities/standards-setting/ispm/>

⁵ IPPC, International Standard on Phytosanitary Treatments for Regulated Pests (ISPM 28 and its annexes) <https://www.ippc.int/en/core-activities/standards-setting/ispm/>

⁶ Development of Generic Phytosanitary Irradiation Doses for Arthropod Pests. Florida Entomologist, Vol. 99, Special Edition 2, October 2016

family. This body of work is leading to new irradiation standards. Although several generic doses for large groups of pests were developed they have not yet been adopted as there is a need for further research to fully validate these

generic doses and supplement proposals to support their acceptance in the International Standards for Phytosanitary Measures (ISPM).

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
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
Bahamas	BHA5001	Developing laboratory capacity for testing contaminants in animal and related products including fish in Bahamas	J.J. Sasanya
Bahrain	BAH5002	Establishing a National Quality Control Standard for Foodstuffs and Fishery Products	J.J. Sasanya
Botswana	BOT5020	Enhancing Capabilities for a Holistic Approach to Testing 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
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
Dominica	DMI5002	Enhancing Capacity to Monitor Agrochemical Residues in Foods and Related Matrices	J.J. Sasanya
Dominican Republic	DOM5005	Strengthening National Capacities to Ensure Food Authenticity	S.D. Kelly

Country/Region	Project No.	Title	Technical Officer
Ecuador	ECU5030	Reducing Post-Harvest Losses of Native Potatoes and other Fresh Foods by Irradiation	C.M. Blackburn
Eritrea	ERI5012	Developing Analytical Capabilities for Food Safety	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	FIJ5004	Establishing a Food Safety Laboratory for Analysis of Pesticide Residues in Fresh Fruits, Vegetables and Root Crops	B.M. Maestroni
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
Cote d'Ivoire	IVC5041	Strengthening Capabilities to Monitor Contaminants in Food and the Environment	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
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
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 Fooder Crops and Authenticity of Local Honey Using Nuclear and Related Technologies	A. Cannavan
Mauritius	MAR5027	Building Capacity to Analyse Veterinary Drug Residues and Related Chemical Contaminants in Animal Products	J.J. Sasanya

Country/Region	Project No.	Title	Technical Officer
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
Mongolia	MON5024	Enhancing Food Safety Analytical Capabilities for Veterinary Drug Residues and Related Contaminants Using Isotopic Techniques	J.J. Sasanya
Montenegro	MNE5004	Strengthening Technical and Institutional Capacities of the National Reference Laboratory for Food and Feed Control	A. Cannavan
Mozambique	MOZ5010	Strengthening Confirmatory Analytical Capabilities for Veterinary Drug Residues and Related Contaminants in Animal Products	J.J. Sasanya
Namibia	NAM5015	Developing Capacity of the National Standard Institution and Agro-Marketing and Trade Agency in the Areas of Food Safety	B.M. Maestroni A. Cannavan
Namibia	NAM5018	Strengthening Animal Health and Food Safety Control Systems	J.J. Sasanya
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
Vanuatu	NHE5002	Strengthening Agro-Food Laboratory Quality Infrastructure	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
Philippines	PHI5035	Advancing Laboratory Capabilities to Monitor Veterinary Drug Residues and Related Contaminants in Foods	J.J. Sasanya
Rwanda	RWA5002	Strengthening Laboratory Capacity to Analyse and Monitor Food Contaminants by Standards Board	J.J. Sasanya

Country/Region	Project No.	Title	Technical Officer
Seychelles	SEY5010	Strengthening Laboratory Capabilities to Enhance Food Safety Using Nuclear and Complimentary Analytical Techniques	J.J. Sasanya
Sri Lanka	SRL5048	Strengthening National Capability for Food and Feed Safety	A. Cannavan A. Mihailova
Sudan	SUD5039	Enhancing the Capacity to Monitor Pesticide and Veterinary Residues in Food Using Nuclear and Complementary Techniques	J.J. Sasanya
Sudan	SUD5040	Strengthening the Evaluation of Quality, Monitoring and Control Programmes for Food Contaminants	J.J. Sasanya
Thailand	THA5056	Strengthening Food Safety Laboratory Capacities	J.J. Sasanya
Uganda	UGA5042	Strengthening Capabilities of Two Central Food Safety Laboratories and Selected Regional Veterinary Centres of Public Health	J.J. Sasanya
Uganda	UGA5040	Strengthening Multi-Sectoral Food Contaminant Monitoring Programmes Through the Effective Use of Nuclear, Isotopic and Complementary Techniques	J.J. Sasanya
Viet Nam	VIE5022	Promoting Interlaboratory Comparison and Accreditation in Testing Chemical Contamination for Food Safety	B.M. Maestroni
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	RAF5084	Strengthening Food Contaminant Monitoring and Control Systems and Enhancing Competitiveness of Agricultural Exports using Nuclear and Isotopic Techniques (AFRA)	J.J. Sasanya
Asia	RAS5078	Enhancing Food Safety Laboratory Capabilities and Establishing a Network in Asia to Control Veterinary Drug Residues and Related Chemical Contaminants	J.J. Sasanya
Asia	RAS5081	Enhancing Food Safety and Supporting Regional Authentication of Foodstuffs through Implementation of Nuclear Techniques (RCA)	S.D. Kelly

Country/Region	Project No.	Title	Technical Officer
Asia	RAS5087	Promoting Food Irradiation by Electron Beam and X Ray Technology to Enhance Food Safety, Security and Trade (RCA)	C.M. Blackburn
Latin America	RLA5079	Applying Radio-Analytical and Complementary Techniques to Monitor Contaminants in Aquaculture (ARCAL CLXXI)	J.J. Sasanya
Latin America	RLA5080	Strengthening the Regional Collaboration of Official Laboratories to Address Emerging Challenges for Food Safety (ARCAL CLXV)	B.M. Maestroni A. Cannavan
Latin America	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	RLA5084	Developing Human Resources and Building Capacity of Member States in the Application of Nuclear Technology to Agriculture	J.J. Sasanya

Virtual Training to Support Veterinary Drug Residue Monitoring Programmes in Latin America and the Caribbean: TCP RLA5081, 22 March and 12 May 2021

James Sasanya

One of the outputs of the ARCAL project RLA5081 “Improving Regional Testing Capabilities and Monitoring Programmes for Residues/Contaminants in Foods Using Nuclear/Isotopic and Complementary Techniques” is established or improved residue monitoring programmes. In the absence physical meetings, a series of virtual training workshops were organized for several project counterparts and stakeholders, with the help of two external experts, between 22 March and 12 May 2021. This covered a range of topics including: developing a residues plan; food safety legislation (old and new); analytical requirements; matrix ranking of substances to establish risks; sampling procedures and investigating non-compliant test results of authorized and nonauthorized substances, among others. More than 50 counterparts including laboratory and regulators benefitted from this training and they provided very good feedback. Plans are underway to support other residues/contaminants.

Training on Food Safety Regulatory Framework – TCP RAF5084, 6–7 April 2021

James Sasanya

Under a regional food safety project for Africa, RAF5084 “Strengthening Food Contaminant Monitoring and Control Systems and Enhancing Competitiveness of Agricultural Exports using Nuclear and Isotopic Techniques”, a virtual training course to enhance the food safety regulatory framework was organized from 6 to 7 April 2021. This was attended by over 50 participants from 25 countries in the African region, who are involved in different aspects of the safety control system.

Topics covered included, among others: the national food safety legal/regulatory framework: a fundamental element of an efficient national food safety control system; the building blocks and operationalization of a the framework; the international regulatory context of food safety from the perspective of the WTO/SPS/TBT, Codex; Importance of an efficient national food safety control system: Impact on public health, food security, trade and socio-economic development. Participants also discussed Codex and its relevance to developing countries, using the importance for Codex standards and Codes of Practice to various commodities such as spices, fruits and vegetables, coffee, tea and animal products, as examples. The linkages between the legal/regulatory frameworks and the national residue/hazard monitoring and control programmes,

including the role of stakeholders in complementing policy makers in strengthening food safety were addressed with each country sharing their experiences and challenges. Two case studies from India on market access were also presented

and used to guide discussions and make comparisons. Areas of need were identified for follow up action.



Some RAF5084 participants at the training workshop on food safety regulatory framework.

Enhancing Laboratory Quality Management at Rwanda Standards Board (RSB)

James Sasanya

Under a national food safety TC project RWA5002 “Strengthening Laboratory Capacity of the Rwanda Standards Board to Analyze and Monitor (Veterinary Drug Residues and Related Contaminants) chemicals in foods”, a virtual national training course on quality management system was organized from 23 to 31 March 2021 with 32 participants from different sections participating. Training support, included demonstrations, lectures and group discussions covering areas such as (but not limited to): General requirements for testing and calibration laboratory with emphasis on ISO/IEC 17025:2017 – training, implementation and auditing; preparation for integrating a laboratory information management system (LIMS) into a routine testing laboratory; attaining/maintaining accreditation; mock laboratory assessment; and common challenges with ISO standards (s) as well as resolving non-compliance.

The RSB personnel gained better understanding of the new requirements in ISO7025:2017, how to integrate LIMS in routine laboratory testing as well as developing and implementing an effective and efficient management system, among others. Participants were also equipped with

knowledge and skills in developing and implementing a management system in line with the new standard. Using a process mapping exercise, they also realized how different units in a laboratory depend on each for an effective management system. The training also helped enhance knowledge on lab competence, provision of timely results to clients, due diligence and effective communication so that quality is not compromised or affected at any point in the testing process.



RSB participants during the virtual training in quality management.

Virtual Workshop to Review and Amend the Draft National Food Safety Standards and Codes of Practice for Oman, and Develop and Improve National Food Control Strategies, March 21–25, 2021

Simon Kelly

Food safety has been identified as a priority sector in the 2016–2020 Oman National Plan and consequently in the IAEA 2018–2023 Country Programme Framework (CPF). As part of the Technical Cooperation Project (TCP) OMA5008 “Enhancing National Omani Capabilities in Food Safety and Traceability” a 5-day Virtual Workshop supporting the Oman Government's recent decision to create a national Food Safety and Quality Centre, under the auspices of the Ministry of Regional Municipalities and Water Resources, was organized. At present, there is a baseline of capacity to analyse food contaminants in Oman, but this capability is dispersed amongst institutions under several different Ministries. Furthermore, none of the existing laboratories, associated with the Ministries, have a fully-fledged quality system such as ISO17025. There is also the need to update Oman's food legislation to strengthen food control and consumer protection, and create a national food control agency. In order to address these requirements three highly experienced experts were contracted to deliver the workshop. These were; Dr Bert Pöpping, managing director of the strategic food consulting company FOCOS; Dr Mark Woolfe, retired Head of the Food Authenticity Programme of the UK's Food Standard's Agency; and Dr Nasr Hasanain, Food Expert in the Qatar Ministry of Public Health. The subject matter covered in the 5-day Workshop, in response to the above situation, included:

- International Agreements and Standards, and their impact on national food legislation,
- Regulatory frameworks for making and reviewing food control legislation,
- Models for food laws,
- Models for Food Control Agencies,
- Food labelling and standards, food fraud and food safety, and traceability,
- Food irradiation – purpose and methods of irradiation, methods for detecting irradiated foods, and control of food irradiation.

There were 14 participants in the workshop mainly from the Ministry of Agriculture, Fisheries and Water Resources. The presentations prepared by the experts were given in both English and Arabic and the Workshop was delivered between 10:00 to 14:30 on all 5 days with a short 30 minute break using Microsoft Teams. All of the PowerPoint

presentations were stored onto a dedicated TCP OMA5008 SharePoint site, with restricted access for the workshop participants, through registered IAEA Nucleus accounts after each day's session. The specific topics covered by Dr Hasanain were International Context and the World Organization for Animal Health, National Regulatory Frameworks for Food, Making and Reviewing National Laws, Toward Integration, incorporating a Biosecurity Approach, Model Food Laws and Challenges of developing and improving a National Food Control Strategy. Dr Woolfe covered the topics of International agreements and standards (WTO and Codex), Elements of a National Food Control System, Models for Food Control Agencies and Codex Standards on Food Labelling, food fraud and food safety, and traceability. Finally, Dr Bert Pöpping delivered presentations on Reasons for irradiation of agricultural products and food, Types of irradiation, Good Practices of irradiation, Detection of irradiated food, and Legislation on irradiated foods in selected countries.

The Workshop generated several high level recommendations to establish the Oman National Food Safety and Quality Centre including working with other Government bodies to revise and draft a new food safety law focusing on consumer protection. There was full participation on each day of the Workshop and the participants had ample opportunity for discussion and questions. The Workshop was well received, and provided the information for the new National Food Safety and Quality Centre to begin its journey into providing food safety and consumer protection for the Omani population.

Virtual Training in Multivariate Data Analysis Using the Chemometrics Add-in for Excel (CAFE) Software: RAS5081, 14–18 December 2020 and BGD5032, 1–15 February 2021

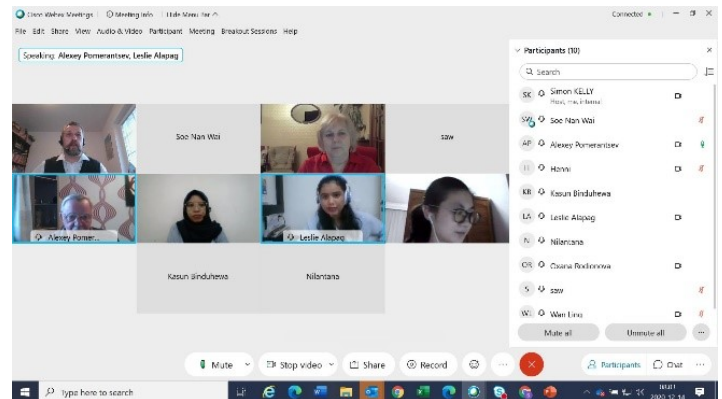
Simon Kelly

The IAEA technical cooperation project RAS5081, which falls under the Regional Cooperative Agreement for Asia and the Pacific (RCA) is building on the previously developed regional capacities and capabilities in the application of nuclear and isotopic techniques for food authenticity, origin and safety and the use of integrated approaches to develop systems for the protection and promotion of foods with a strong national or regional character. An important part of the overall implementation of these techniques is the ability to apply robust statistical models that permit the many variables generated by stable isotope, trace element and complementary analyses to be combined. During the COVID-19 pandemic, and the severe restrictions on international travel, it has been a priority to maintain the momentum of the regional training programme through alternative modalities, such as on-line distance learning events. To this end a Virtual Regional Training

Course (VRTC) in multivariate data analysis using the Chemometrics Add-in For Excel (CAFE) software was organized from 14 to 18 December 2020. The aim of the course was to provide the participants with a powerful, easily accessible, and low-cost tool for basic multivariate data analysis (MDA). MDA is an essential part of complex analytical experiments and chemometrics is intensively used for processing of various nuclear data e.g. stable isotope and trace element (SITE) data for food authentication and to verify the geographical origin of food to support traceability systems. The trainers, Professors Oxana Rodionova and Alexey Pomeranstev, from the N.N. Semenov Federal Research Centre for Chemical Physics, prepared seventeen narrated PowerPoint tutorials, that were made available ‘on-demand’ through an IAEA Nucleus SharePoint site along with the Excel software add-in, guidance manuals and Excel templates with examples of data from food authenticity studies. The dedicated distance-learning SharePoint site was made available to fifteen participants from Australia, Bangladesh, China, India, Indonesia, Lao P.D.R., Malaysia, Mongolia, Myanmar, Philippines, Korea, Republic of, Singapore and Sri Lanka. The VRTC was the fifth in a series from the technical cooperation project for Asia-Pacific “Enhancing Food Safety and Supporting Regional Authentication of Foodstuffs through Implementation of Nuclear Techniques” (RAS5081). Participants viewed the software tutorials on-demand and then undertook computer exercises using Excel matrix calculations, regressions, and determining basic statistical functions before moving on to more complex classification problems, typically found in food authentication and provenance studies, involving data evaluation with Principal Component Analysis, Soft Independent Modelling by Class Analogy and Partial Least Squares Discriminant Analysis. The participants were given plenty of opportunities to ask questions about all aspects of the course through an on-line plenary session hosted by Mr Kelly through the IAEA’s Webex platform and through scheduled one-to-one question and answer sessions hosted by the trainers. The participants completed relevant assignments throughout the course. Care was taken to ensure that participants understood the required pre-processing of data and when to remove outliers or other non-essential data. The course finished with another plenary session and participants were sent electronic IAEA RAS5081 course certificates. The software support from the trainers then continued for another month via e-mail correspondence to ensure that participants had additional time to cement the training and ask questions that might arise from chemometric analysis of their own data. The overall feedback on the training course by the participants was excellent and the participants were highly motivated to learn and interact with each other despite the challenges of distance learning. All participants were well prepared for the course in advance with availability of the on-demand tutorials and coped well with the Excel style format of the software enabling them to have effective and efficient training in a familiar ‘software environment’. The course

laid the foundation for an essential part of SITE data elaboration, stressing the importance of building reliable and robust multivariate models before moving to the testing phase for food authentication methods.

The format for this training was also successfully applied within the national technical cooperation project BGD5032, ‘Building Capacity in Improving Food Safety Using Nuclear and Other Complementary Analytical Techniques’, the objective of which is to improve the national food traceability and authenticity system using nuclear and other complementary techniques.



Participants from the Virtual Training in Multivariate Data Analysis using the Chemometrics Add-in For Excel (CAFE) software.

Developments at the Food and Environmental Protection Laboratory

Discrimination between Arabica and Robusta Coffee Using Multispectral Imaging

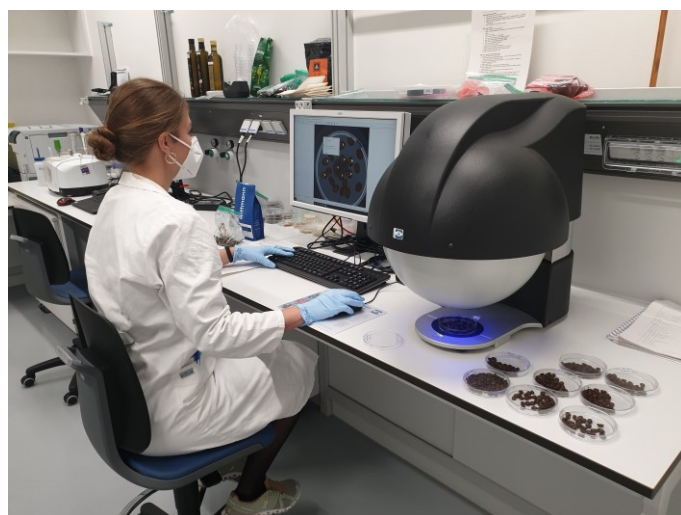
Alina Mihailova, Beatrix Liebisch, Marivil Islam

Various spectroscopic and mass chromatographic techniques have been applied for the testing of food authenticity. There is, however, still a significant need to develop faster, cheaper and more efficient methods to complement currently available techniques. Multispectral imaging (MSI) is an innovative and non-destructive technique that combines imaging and spectral technologies with advanced digital image analysis and machine learning. Using strobed light-emitting diode (LED) technology the MSI system combines measurements at 20 different wavelengths into a single high-resolution spectral image, permitting fast and accurate characterization of foods in terms of colour, surface chemistry, texture, shape, and size without touching the sample and often with no sample preparation. This technique can complement the various other analytical approaches that are being developed and tested at FEPL under CRP D52040 “Field-deployable Analytical Methods to Assess the Authenticity, Safety and Quality of Food” and CRP D52042 “Implementation of Nuclear Techniques for Authentication of Foods with High-Value Labelling Claims” for transfer to the Member State laboratories.

One of the food authenticity applications of MSI currently being developed at FEPL, is a rapid approach for the differentiation of Arabica and Robusta coffee beans. Cultivated coffee beans are among the most widely traded commodities in the world and are frequently targets for fraud. The two main species of cultivated coffee are *Coffea arabica* and *Coffea canephora* (commonly known as Robusta coffee). Arabica beans, which account for nearly 70% of global coffee production, are prized for their superior smooth flavour. However, Arabica plants are more prone to disease than Robusta plants, which, though more disease-resistant, produce beans that yield a stronger, harsher and more bitter drink, and are therefore sold at lower prices compared to Arabica. This price differential offers the potential for unscrupulous traders to make economic gain by partially or wholly substituting Arabica beans with Robusta. Therefore, there is an urgent need for rapid, low-cost and efficient analytical techniques that can be used to monitor the authenticity of Arabica coffee and screen the samples in the supply chain.

FEPL has been developing a rapid screening method for the differentiation between Arabica and Robusta coffee samples using the VideometerLab 4 MSI system.

Overall, 10 types of Arabica and 8 types of Robusta roasted coffee, mostly of single origin (India, Uganda, Brazil, Mexico), were obtained for this study from a specialized coffee store. No sample preparation was required for the MSI analysis. Spectral data were obtained in 100% reflectance mode between 365 and 970 nm. Image data (e.g. bean width, length, area, roundness) were obtained by using the VideometerLab Blob tool. For each coffee type, 10 samples were analysed, each comprising of 20 coffee beans. The spectral and image data were obtained for each coffee bean individually, and then the data from 20 beans were combined and averaged. Further, the spectral and image data of each sample were scaled using Unit-Variance (UV) scaling and subjected to chemometrics analysis.



MSI analysis of Arabica and Robusta coffee at FEPL.

Principal component analysis (PCA) was used to assess the initial coffee sample groupings. The PCA model (Figure 1) showed the tendency of coffee samples to group according to their botanical origin. The goodness of fit ($R^2X(\text{cum})$) and the predictability ($Q^2(\text{cum})$) values of the obtained PCA model were 0.892 and 0.850, respectively.

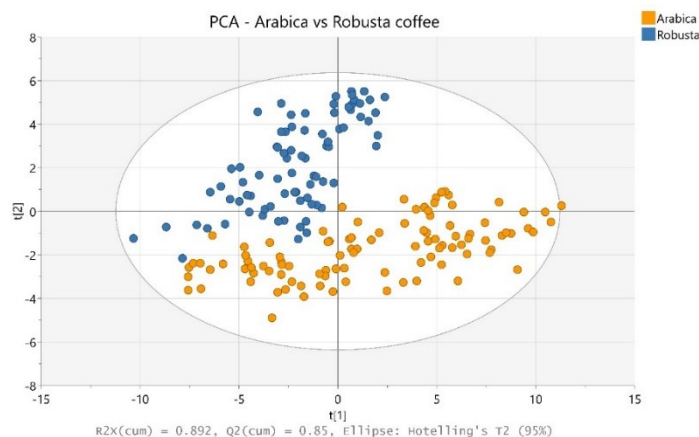


FIG. 1. PCA plot of Arabica and Robusta coffee.

A supervised multivariate data analysis approach, orthogonal projections to latent structures discriminant analysis (OPLS-DA) with seven-fold cross-validation, was used to build a discriminative model, which successfully discriminated the Arabica and Robusta coffees (Figure 2). R2X(cum), R2Y(cum) and Q2(cum) values of the obtained cross-validated OPLS-DA model were 0.957, 0.899 and 0.890, respectively.

The available sample set was split into a training set (n=121) and a test set (n = 59). The OPLS-DA model was constructed using only the training dataset and subsequently used for the prediction of the botanical origin of “unknown” coffee samples from the test dataset. The OPLS-DA model allowed 100% correct prediction of both Arabica and Robusta coffee samples.

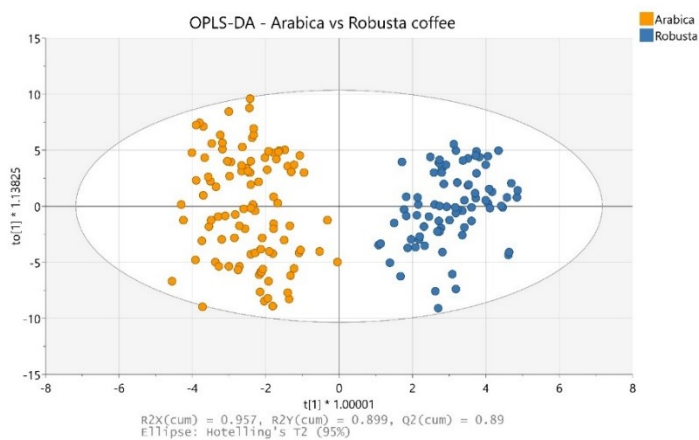


FIG. 2. OPLS-DA plot of Arabica and Robusta coffee.

The major spectral and image features responsible for the differentiation of Arabica and Robusta coffee were examined using the Variable Importance in Projection (VIP) scores from the OPLS-DA model. The VIP plot is shown in Figure 3. The major differences between the two coffee types were observed in coffee bean shape, width-to-length ratio, area, as well as wavelength regions of 570–590 nm and 630–690 nm.

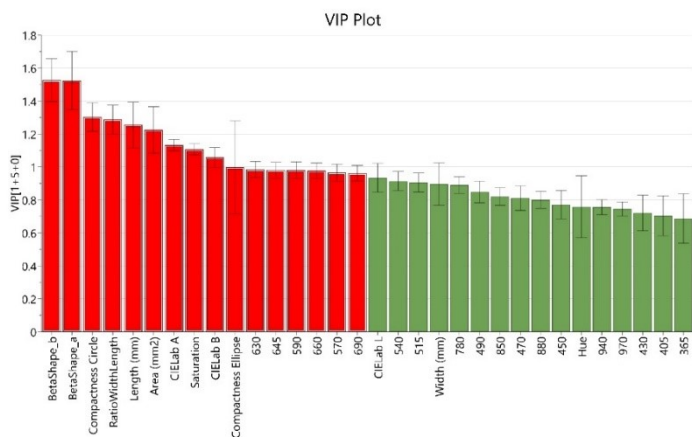


FIG. 3. Variable importance in projection (VIP) plot for the OPLS-DA model of Arabica and Robusta coffee.

This work is being continued to assess whether the approach can also be successfully applied for the determination of the

level of adulteration of Arabica coffee with Robusta. Further model validation will be performed using a larger number of retail and authentic Arabica and Robusta coffee samples.

Discrimination between Arabica and Robusta Coffee Using Bench-Top Nuclear Magnetic Resonance (NMR) Spectroscopy

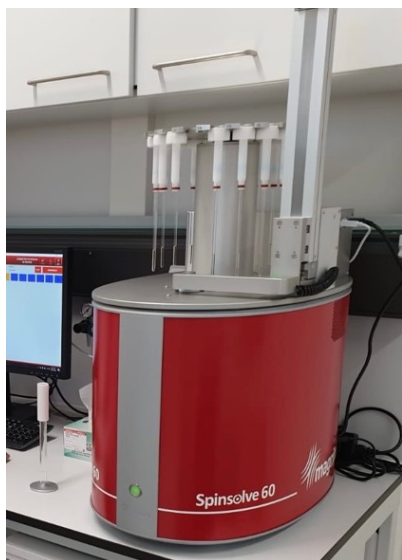
Ignacio Miguez, Alina Mihailova, Beatrix Liebisch, Simon Kelly

Foods are very complex matrices that consist of thousands of primary and secondary metabolites. The variability in metabolism, as well as their composition are reflected in their NMR spectra. These spectra are sometimes difficult to interpret due to the complexity of the food samples which necessitates the use of multivariate statistical methods to identify the compounds responsible for the differentiation between samples.

Coffee beverages have been consumed for centuries, but they are still gaining popularity across the world due to coffee's flavour and reported health benefits. However, adulteration is a problem within the supply chain. One of the major fraudulent practices involves the mixture of different quality coffee beans from two different species; Arabica and Robusta. Arabica beans are more expensive beans being widely acknowledged to have better taste, while Robusta beans are easier to cultivate and have a higher yield. This makes Arabica coffee more susceptible to economically motivated adulteration with cheaper Robusta. In order to protect consumers and stakeholders in the coffee supply chain from fraud, it is important to develop robust and reliable methods to detect the adulteration of Arabica beans.

Last year, the FEPL acquired a Magritek Spinsolve 60 MHz benchtop NMR system and started method optimization for the rapid discrimination between Arabica and Robusta coffee beans. To increase the analytical capacity of the system, the spectrometer has recently been coupled to an autosampler. This improvement allows the automated analysis of a greater number of coffee samples, vastly improving the efficiency of research for the development of a multivariate statistical model for the discrimination of the two coffee species.

The spectral data obtained from the coffee extracts was processed and its quality assessed prior to its use in statistical models to allow the chemometric comparison between the two coffee species. Principal component analysis (PCA) was used to assess the initial sample groupings and the quality of the data. The performance values of the PCA model, the goodness of fit (R2X(cum)) and the predictability (Q2(cum)), were 0.996 and 0.966, respectively. A PCA model is considered robust if $Q2 > 0.5$ and $R2 > 0.5$, hence these results are very encouraging.



Spinsolve 60 MHz bench top Nuclear Magnetic Resonance spectrometer with automatic NMR sample tube changer in the Food and Environmental Protection Laboratory.

Orthogonal projections to latent structures discriminant analysis (OPLS-DA) (supervised approach) was performed to differentiate the samples. The sample set was split into a training set (2/3 of the total number of samples), used for building the model, and a test set (the remaining 1/3 of the samples), used for the model validation. The OPLS-DA model (Figure 1) was able to discriminate Arabica and Robusta coffee samples and achieve 100% correct prediction of the samples from the test dataset. The performance indicators of the OPLS-DA model demonstrated that it had performed well, R^2X (cum), R^2Y (cum) and Q^2 (cum), being 0.817, 0.975 and 0.961, respectively.

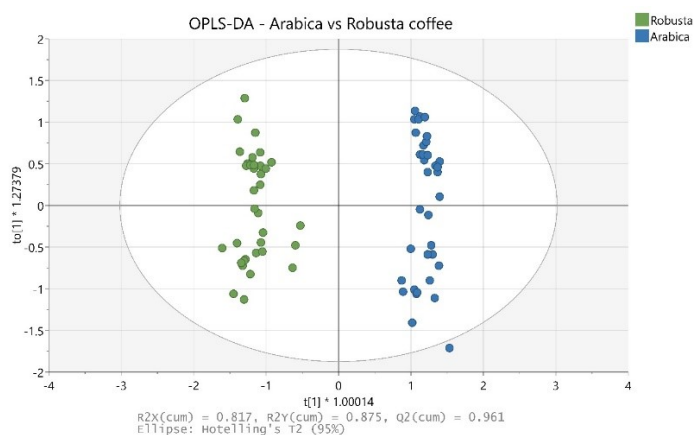


FIG. 1. OPLS-DA model of Robusta and Arabica coffee using 1H NMR spectral data.

One of the spectral features identified as being responsible for the differentiation of the coffee varieties was the signal of the 16-*O*-methylcafestol, which is found mainly in Robusta beans, and has been proposed as a phytomarker for the differentiation between the two species.

The signal corresponding to 16-*O*-methylcafestol is a singlet that, given the conditions of the resonance experiment, presents good resolution from the rest of the signals in the

spectrum. Considering the characteristics of this signal, it was possible to build a simple linear regression model (Figure 2) with different adulteration levels of Robusta in Arabica coffee (from 0% to 100% (w/w) Robusta coffee in Arabica). The model was constructed using 5 replicates at each level of adulteration and further tested with 2 replicates at each adulteration level. The regression mean square error (RMSE) and the mean percentage prediction error (PPE) of the model were 3.6% and 3.9%, respectively. The current model allows the prediction of the % w/w of adulteration in samples that contain at least 10% w/w of Robusta beans. At levels below this, the incentive for economic adulteration is low.

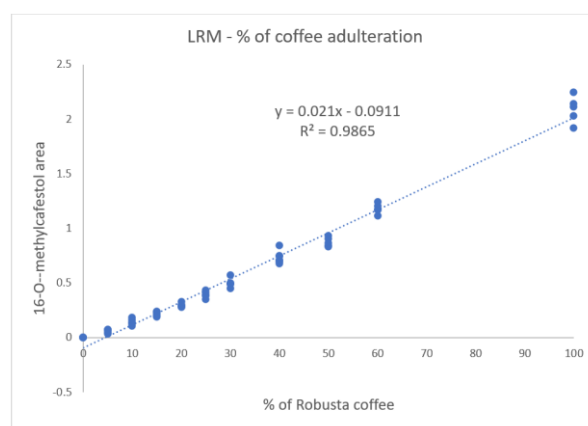


FIG. 2. Linear regression model for the prediction of the % w/w of Robusta coffee in an Arabica coffee.

Future work will include the analysis of new commercial and authentic coffee samples from Costa Rica and Jamaica to assess the robustness of both predictive models (discrimination and percentage of adulteration) and perform further model validation.

Training on Detection of Heavy Metals in Turmeric Teas Using Screen Printed Electrodes and Voltammetry

Britt Maestroni and Sofia Rezende

Excessive accumulation of heavy metals in plant foods grown in contaminated areas can seriously affect public health, food quality and safety. Roots and tubers make a significant contribution to the diets of people worldwide. A number of studies have reported turmeric as a source of lead (Pb) exposure in South Asia. Since 2011, there have been 13 international recalls due to excessive Pb concentrations in certain brands of turmeric from Asia. Lead is toxic and even low levels can impair normal cognitive development, especially among young children.

Spices, including turmeric, are among the top five most commonly adulterated food products. Economically motivated adulteration (EMA) frequently occurs at the processing phase, from rhizome to powder, prior to trade. Examples of EMA include the addition of toxic colorants such as dyes to turmeric powders. Another recent discovery

was the addition of lead chromate to turmeric during processing to enhance the colour of dried turmeric root. A recent study indicated that lead chromate is being added to turmeric by local polishers, who are untrained in basic health concepts and unaware of the neurotoxic effects of Pb. This process is often imprudently implemented to meet wholesalers' requests for very yellow roots, driven by consumer demand for turmeric, which is considered a very healthy spice due to its antioxidant, anti-inflammatory, antimicrobial, and antimutagenic properties.

To help the farmers community meet food safety principles, the FEPL recently initiated a study on contamination of turmeric. One of the results was the development of a multi-contaminant/ multi-residue targeted analytical method for the detection and quantification, by liquid- and gas chromatography – mass spectrometry, of several contaminants in turmeric, including dyes. That method was reported in previous issues of the Food and Environmental Protection Newsletter in 2019–2020, and was published in the peer-reviewed scientific literature (Food Control, 121, 107579). Further studies were initiated to address the authenticity and adulteration of turmeric. One of the goals of the study was to identify and verify the application of a suitable rapid and cost-effective screening technique to improve accessibility of food safety testing in the field. Electroanalytical methodology was identified as a potential solution.

Members of the FEPL staff participated in a virtual training course (Figure 1) for determination of heavy metals in turmeric teas using screen printed electrodes and voltammetry. The training was offered by the University of Oviedo and consisted of a practical demonstration in the laboratory of the principles and application of square wave voltammetry using a portable potentiostat, (see Figure 2), and utilized as a rapid screening method for Pb, Cadmium (Cd) and Zinc (Zn) in turmeric teas. The objective of the training was to familiarize FEPL staff with the use of screen-printed electrodes (SPE) and the detection system.

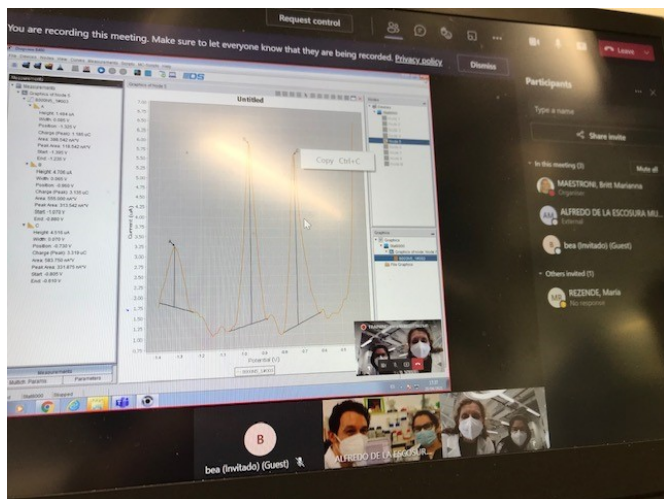


FIG. 1. Virtual training helped FEPL staff gain knowledge about a rapid screening technology for detection of Zn, Cd and Pb in turmeric teas.

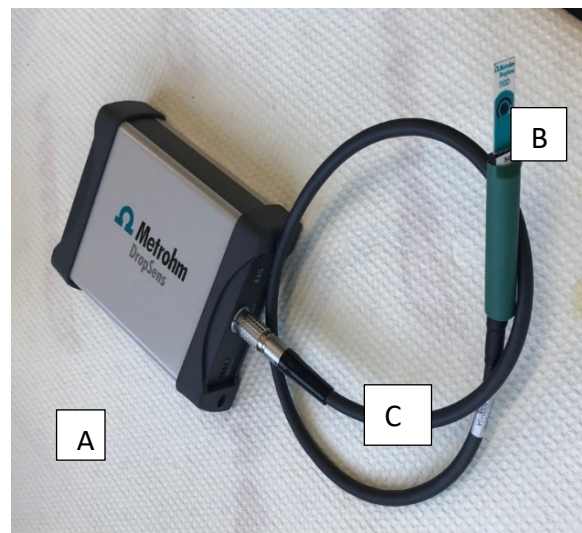


FIG. 2. The potentiostat (A) used at FEPL for implementing square wave voltammetry using SPE (B) via a transducer cable (C).

A turmeric tea sample was prepared according to home tea preparation guidelines and fortified at 1 mg/kg with a mixture of Zn, Cd and Pb. The teas were left to cool down to room temperature. A small volume (50 μL) of turmeric tea was applied, using an automatic pipette, to the working electrode (see Figure 3) of the SPE, as shown in Figure 4. The potentiostat was connected to the SPE via a transducer cable, and the signal generated using dedicated software. This preliminary investigation demonstrated the potential of the technique, and further work at FEPL will include the validation of this rapid screening method and its application to turmeric tea samples.

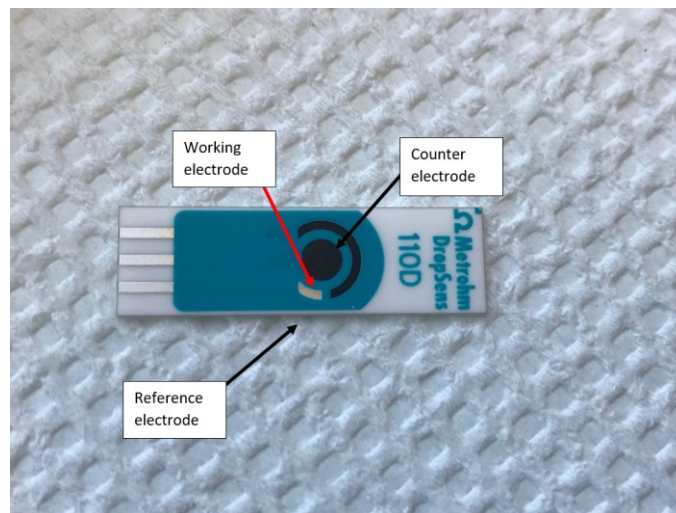


FIG. 3. The screen-printed electrode used at FEPL.



FIG. 4. Ms Rezende applying 50 μ L of spiked turmeric tea to the SPE electrodes, connected via a transducer to the potentiostat.

Study of the Volatile Fraction of Fresh Orange Juices

Britt Maestroni and Sofia Rezende

Orange juice is one of the most frequently consumed beverages worldwide. Among all common juice products, ‘not from concentrate’ (NFC) orange juice is considered as being the best quality, accounting for an important share of orange juice consumption in developed countries, while its shares continuously increase in emerging countries. Economically motivated juice adulteration is a well known practice, particularly when significant value can be added, for example by labelling a product as ‘organic’. Orange juice is generally allowed to be mixed with small amounts of other citrus juices or juice cells, according to the Codex General Standard for Fruit Juices and Nectars (CODEX STAN 247-2005), to adjust the taste and flavour before sterilization and packaging. This aspect, in addition to the potential to fraudulently claim organic production, provide enormous challenges for the analytical chemists responsible for testing the marketing claims and ensuring food safety (e.g. absence of non-compliant concentrations of pesticide residues and their metabolites). Numerous analytical methods have already been developed to detect adulteration in fruit juices, most often in conjunction with multivariate data analysis. However, the diversity in adulteration techniques, the natural variation of fruits of different geographical and varietal origin, along with the different storage conditions and processing techniques, make the detection and prevention of juice adulteration a very complicated task. In this sense, effective, reliable and rapid food authentication methods represent a valuable and irreplaceable tool for enabling authorities to set up control systems to ensure juice quality and safety and to combat fraud.

The FEPL, largely through the work of the RALACA network, continues to support research studies on good agricultural practices, as it is important to ensure both the safety of food products and a sustainable agricultural environment. As a component of this research, a study on the characterization of organic versus conventional

production was initiated in FEPL, and additional methods were developed and validated to ensure food safety in a range of different commodities that have socio-economic importance in Member States. One of the techniques applied is the use of a dual-detection headspace-gas chromatography-mass spectrometry-ion mobility spectrometry (HS-GC-MSD-IMS) system. This system was applied to test the volatile fraction of orange juice samples, and to putatively characterize the headspace fraction of injected orange juice samples. A variety of different orange juices, originating from Latin America, were considered in this study, which also included the optimization of the headspace method and of the GC conditions. The initial IMS results are presented in Figure 1. Selected IMS areas, corresponding to specific spectral regions, were selected and their signals used to run principal component analysis (PCA) on the whole batch of samples as shown in Figure 2.

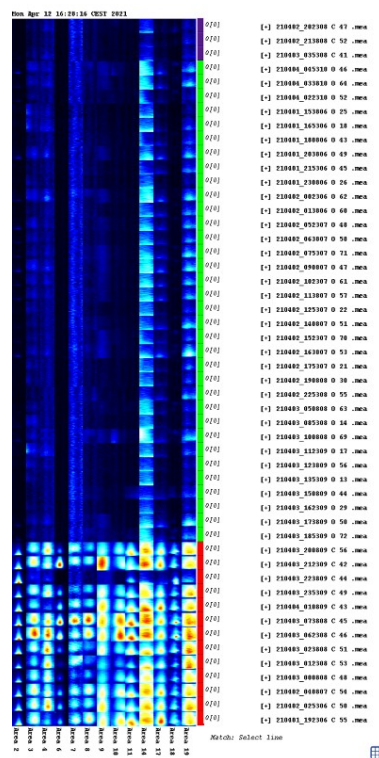


FIG. 1. Selected IMS areas corresponding to specific spectral regions were selected and the signal was recorded for multivariate data analysis.

Figure 2 provides a first indication that selected volatile compounds may be able to distinguish between organic and conventional orange juices, in this set of samples. Future work includes a validation of the multivariate model and further refinements using a variety of additional samples collected from different regions, different cultivars and different storage conditions and processing techniques.

The set of orange juices samples were also tested using gas and liquid chromatography coupled to tandem mass spectrometry (GC-MSMS and LC-MSMS) against a targeted list of pesticides. The results are still being analysed and will be used in a multivariate data fusion experiment contributing to providing further evidence on the organic versus conventional orange juice separation model.

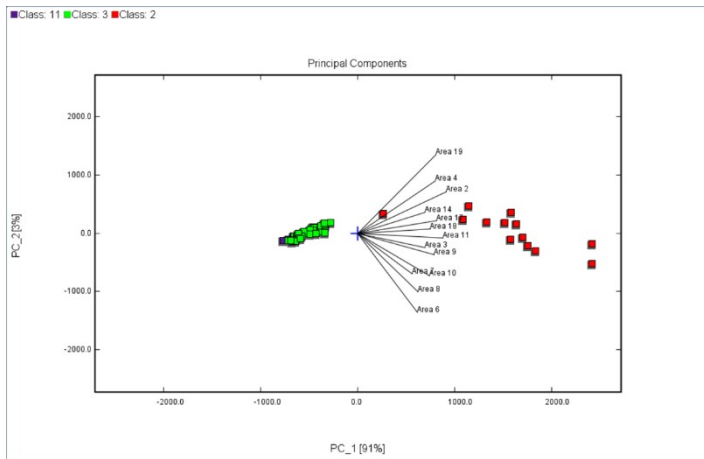


FIG. 2. PCA analysis of the orange juice samples. Green dots correspond to fresh organic orange juice samples, red dots correspond to conventionally produced fresh orange juice samples, violet dots correspond to concentrated orange juice samples.

Differentiation between Organic and Conventional Orange Juices Using Benchtop ^1H NMR Spectroscopy

Ignacio Miguez, Alina Mihailova, Beatrix Liebisch, Simon Kelly

Global food regulations require that the provenance of food can be traced from farm to fork. Currently no routine authenticity testing is carried out on the organic products before they reach the consumer. The authentication of organic foods relies predominantly on certification processes and regular farm inspections. This creates an incentive for unfair producers and retailers to mix organic and conventional produce or even fully substitute organic produce with conventional for financial gain. Significant cases of fraud, where conventional produce was mislabelled and passed off as organic, have been reported in recent years worldwide. The issue concerns the whole agri-food sector, from small local farmers' markets to supermarket chains and global large-scale retailers. To support the integrity of the whole organic food and drink supply chain it is therefore highly important that, in addition to traceability and certification schemes, the authenticity of organic foods can be analytically verified in an objective and independent way.

Although, various analytical techniques have been applied for the authentication of organic food products over the past decade, the lack of reliable markers and the diversity of the organic and conventional cultivation strategies presents a challenge for the development of robust methods that can be adopted for use in routine control practices. Various primary and secondary metabolites can serve as markers for production origin of food and aid the verification of the labelling claims.

The application of ^1H NMR spectroscopy for the authentication of organic food products is still at an early stage. Nevertheless, several recent studies reported the high discriminative power of NMR spectroscopy and its potential

for discrimination between foods from organic and conventional production systems.

In 2020 FEPL commenced work on developing analytical methods for differentiating organic and conventional orange juices, using samples from Mexico supplied by the non-profit fruit juice industry association, "Sure Global Fair" (SGF). Initial analyses were carried out using FTIR spectroscopy. In addition to FTIR, the potential of benchtop ^1H NMR spectroscopy to differentiate the juices from organic and conventional production was investigated. ^1H NMR spectra were acquired using a Magritek Spinsolve 60 benchtop NMR spectrometer. Spectral data were pre-processed using the following algorithms: zero filling, phase correction, baseline correction, referencing, and spectral alignment. Data were normalized, Pareto-scaled and subjected to chemometrics analysis.

Principal component analysis (PCA) was used to assess the initial juice sample groupings. The PCA model (Figure 1) showed the separation of the juice samples according to their production system, i.e. organic or conventional. The goodness of fit (R^2X (cum)) and the predictability (Q^2 (cum)) values of the obtained PCA model were 0.976 and 0.957, respectively.

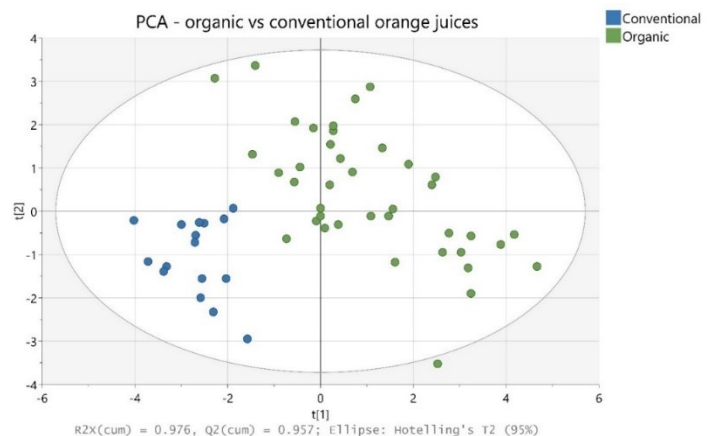


FIG. 1. PCA model of organic ($n=40$) and conventional ($n=16$) orange juice samples from Mexico.

The data were analysed using the supervised multivariate method, orthogonal projections to latent squares discriminant analysis (OPLS-DA), with seven-fold cross-validation. The OPLS-DA model (Figure 2) successfully discriminated between the two groups, organic and conventional juice samples. The performance indicators of the OPLS-DA model, R^2X (cum), R^2Y (cum) and Q^2 (cum), were 0.886, 0.878 and 0.848, respectively.

Using the limited number of samples available, an OPLS-DA model was constructed using a training dataset and subsequently used for the prediction of the production origin of the juice samples from a test dataset not used for model construction. The OPLS-DA model gave 100% correct prediction of both organic and conventional juice samples.

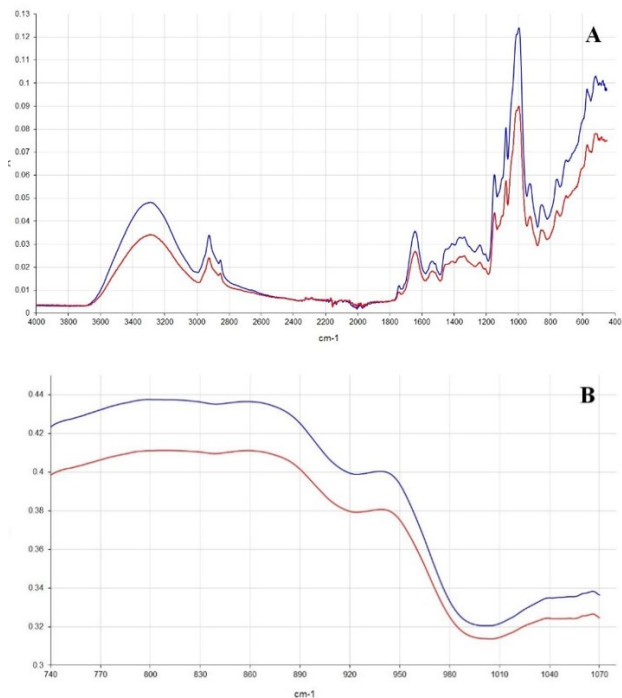


FIG. 1. Representative FTIR-ATR (A) and NIR (B) spectra of Thai Hom Mali rice. Blue spectrum - north-eastern region; red spectrum - northern region.

The full dataset from each production year was divided in randomized order into a training set ($n = 47$ (2018); $n = 67$ (2019)) and a test set ($n = 23$ (2018); $n = 33$ (2019)). OPLS-DA with seven-fold cross-validation was used to build the discriminative models for the differentiation of northern and north-eastern region in the training dataset. The performance of the models was assessed using the goodness

TABLE 1. BENCHTOP FTIR-ATR: GOODNESS OF FIT AND PREDICTIVE ABILITY OF OPLS-DA MODELS FOR THE 2018 AND 2019 DATASETS.

Spectroscopic technique	Year	N (train. set)	N (test set)	R2X (cum)	R2Y (cum)	Q2 (cum)	Correct classification rate of the test set, %		
							Northeast	North	Total
Benchtop FTIR-ATR	2018	47	23	0.919	0.981	0.776	100	100	100
	2019	67	33	0.637	0.929	0.477	96.65	100	96.97
Hand-held NIR	2018	47	23	0.992	0.771	0.409	94.12	66.67	86.96
	2019	67	33	0.969	0.734	0.488	91.30	70.00	84.85

N – number of samples; train. set – training set.

The predictive ability of the OPLS-DA model, generated using the larger dataset of 2019, was challenged by using the samples of 2018 as “unknowns”. Due to the fact that the samples from north-eastern region came from different provinces in 2018 and 2019, only samples from the North (common provinces in both years) were used for this additional model validation. The OPLS-DA model was able to correctly predict 100% of the “unknown” samples from the North from 2018.

The NIR spectra, obtained using a hand-held NIR spectrometer, also allowed differentiation between the rice samples from the northern and north-eastern regions in both

of fit (R^2) and predictability (Q^2) values. The OPLS-DA models, built using the training dataset, were used to predict the geographical origin of the samples from the test dataset. The predictive ability of the models was assessed using the correct classification rate of samples from each region. For additional validation, the OPLS-DA model, obtained using the 2019 dataset (largest dataset), was used to predict the samples from the northern region (the only region with common provinces in both years) from the 2018 dataset. In addition, the effect of sample preparation (ground samples vs no sample preparation) on the performance of OPLS-DA models, obtained using NIR spectral data, was assessed.

The major differences in the FTIR absorbance bands between the samples from the north-eastern and northern regions were observed around 1030 and 1100 cm^{-1} (C-O stretching), which is associated with the presence of polysaccharides, around 1540 cm^{-1} (N-H bend, C-N stretch) and 1640 cm^{-1} (C=O stretch), associated with the presence of proteins, and around 2930 cm^{-1} (CH_2 symmetric stretch), associated with the presence of lipids.

OPLS-DA allowed clear differentiation between the rice samples from the northern and north-eastern regions in both production years (Figure 2). The goodness of fit (R^2) and the predictive ability (Q^2) of the OPLS-DA model are shown in Table 1. External model validation was performed using the test dataset comprising of samples that were not used in the construction of the model. The correct classification rate of samples from the test dataset was 100% and 96.97% in 2018 and 2019, respectively.

production years. The goodness of fit (R^2) and the predictive ability (Q^2) of the OPLS-DA model (Figure 3) constructed using NIR data are shown in Table 1. External model validation was performed using the test dataset comprising samples that were not used in the construction of the model. The correct classification rates of samples from the test dataset were 86.96% and 84.85% in 2018 and 2019, respectively. The predictive ability of the OPLS-DA model, generated using the larger dataset of 2019, was challenged by using the samples of 2018 as “unknowns”. The OPLS-DA model was able to predict 100% of the “unknown” samples from the North from 2018.

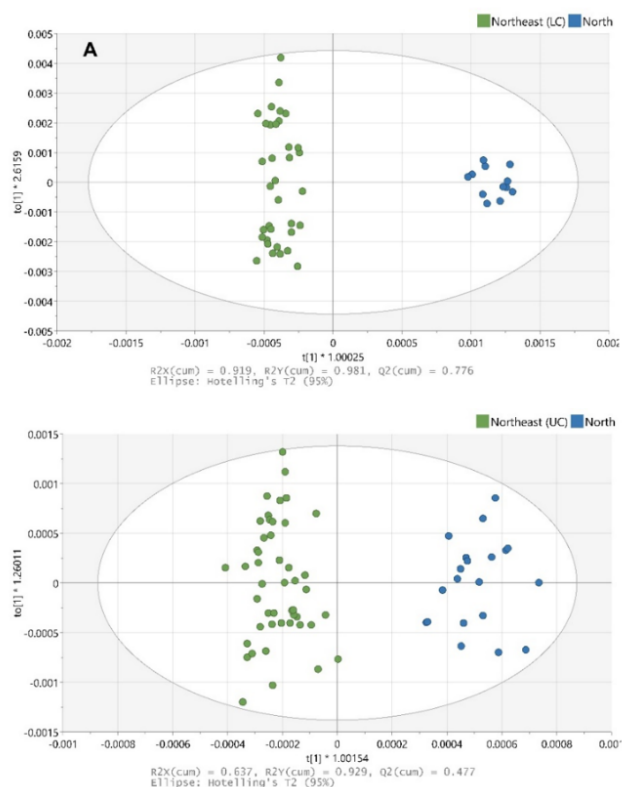


FIG. 2. OPLS-DA models of the training dataset analysed with FTIR-ATR: year 2018 (A) and 2019 (B).

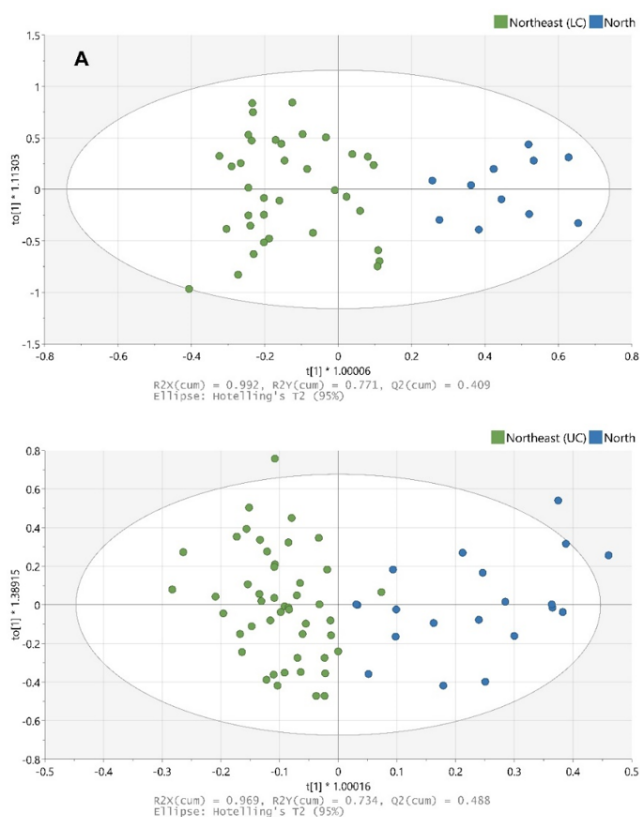


FIG. 3. OPLS-DA models of the training dataset analysed with NIR: year 2018 (A) and 2019 (B).

In addition to ground rice ($n=100$), whole rice grain samples ($n=100$) from 2019 were analysed by hand-held NIR, and the effect of both sample preparation techniques on the performance of OPLS-DA models was assessed. No

decrease in the performance of the model was observed for whole grain rice samples as compared to ground rice powder. The correct classification rates of samples from the test set were 87.88% and 84.85% for whole grain and ground rice samples, respectively. These results demonstrate that in addition to its low cost, small size and ease of use, the hand-held NIR spectrometer is suitable for the analysis of dehulled polished rice without the need of any sample preparation, which makes the approach very rapid and straightforward.

This study demonstrated that FTIR-ATR and NIR spectroscopy, combined with OPLS-DA, are promising analytical tools for geographical differentiation of Thai Hom Mali rice. This opens up the possibility for these types of rapid spectroscopy approaches to be used for cost-effective sample screening at a farm or retail level before committing to more sophisticated and time-consuming SITE techniques for confirmatory or orthogonal analysis.

Volatilomic Profiling of Turmeric Teas by Dual-Detection HS-GC-MSD-IMS

Britt Maestroni and Sofia Rezende

There is increasing interest in natural food products rich in bioactive phytochemicals with healthy properties, and turmeric (*Curcuma longa*), is among those natural food products. Beneficial health properties of turmeric products are mainly attributed to the presence of curcuminoids, phenolic acids, flavonoids, terpenoids, phenylpropanoids and sesquiterpenes, among other compounds. Turmeric has been widely recognized as important for its medicinal properties. As a medicine, turmeric is used for both prevention of, and therapy for diseases. The main clinical targets of turmeric are the digestive organs: in the intestine, for treatment of diseases such as familial adenomatous polyposis; in the bowels, for treatment of inflammatory bowel disease; and in the colon, for treatment of colon cancer. As a food it is used as a spice; as a food supplement it is adopted for its antioxidant, anti-inflammatory, antimicrobial, and antimutagenic properties.

Given the economic importance of spices and this medicinal plant that grows primarily in tropical regions including India, Thailand, Pakistan, China, Sri Lanka, Peru, Haiti and Jamaica, and the fact that many Asian countries consume a large amount of the worldwide turmeric production, the FEPL recently published a method for the analysis of multiple residues and contaminants in turmeric powders. However, economically motivated food fraud is also an important aspect of the work around herb and spices. It is well known that economically motivated adulteration (EMA), involving mixtures and dilutions of authentic spices with low-cost adulterants to increase profit margins, is frequent. Therefore, the FEPL initiated a new study on the profiling of turmeric teas. Initially the goal is to characterize and putatively identify the volatile fraction of turmeric tea samples made from authentic turmeric rhizomes from either

organic or conventional agriculture. This will enable further studies on the differentiation between authentic turmeric and adulterated turmeric samples, that are present in the market, especially when sold in the form of fine powders.

The characterization and authentication of a wide range of food products are often carried out in a straightforward way using instrumental techniques such as stable isotope analysis, and a range of spectroscopic methods which are especially suitable for the analysis of large sets of samples. In this study a novel dual-detection headspace–gas chromatography–mass spectrometry–ion mobility spectrometry (HS-GC-MSD-IMS) system will be used for the analysis of the volatile profile of turmeric teas without requiring any specific sample preparation process or pre-treatment of the samples, except for the processing of the turmeric rhizomes into fine powders with a homogeneous particle size. The volatile phase of samples, prepared by agitation of turmeric agitated with water at a specific temperature (turmeric ‘tea’) in the headspace apparatus, were analysed by GC-MS and GC-IMS simultaneously. The MSD was programmed to analyse the samples in scan mode and Agilent software for unknown data analysis was efficiently used together with mass spectral libraries to putatively identify the volatile compounds present in the headspace of turmeric teas. A retention index library was used on the IMS side to tentatively identify the injected compounds. The dedicated IMS software helped in characterizing the turmeric tea samples.

Several samples of turmeric teas were analysed, using three different time/temperature conditions for turmeric tea preparation. Figure 1 is an example of the IMS plot areas for the different turmeric teas; the data and further considered for multivariate data analysis.

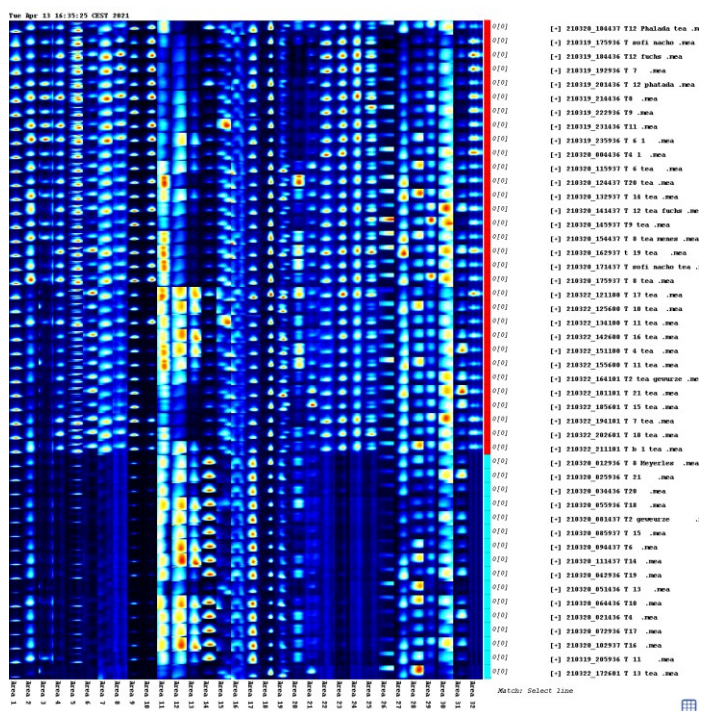


FIG. 1. IMS plot areas selected and used for multivariate analysis of turmeric teas.

The sensitivity of the MSD run in scan mode was too low for detecting pesticide residues. However, dedicated deconvolution software applied to the scan data and a comparison to NIST mass spectral libraries provided a good indication of the possible matrix compounds present in the volatile fraction of teas from authentic turmeric rhizomes, due to the fact that the secondary metabolites are present at very high concentrations compared to, for example, potential residues of pesticides. Principal components analysis (PCA) was applied to the results from IMS signals and the result is shown in Figure 2.

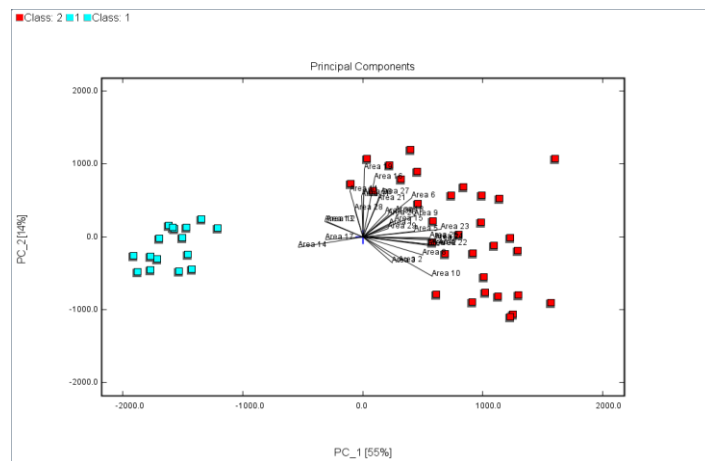


FIG. 2. A PCA plot of the turmeric teas using the IMS plot areas. The red squares correspond to the analysis of teas made from conventional turmeric teas; the light blue squares correspond to organic turmeric teas.

Initial results indicated that the analysis of the volatile fraction from turmeric teas might be a useful technique to distinguish between organic and non-organic (from conventional agriculture) turmeric teas. The model will also require further validation work using more samples from different sources.

In addition the work will focus on the development of a robust enough method using HS-GC-MSD-IMS possibly in combination with other rapid techniques that is able to distinguish with a high confidence between turmeric teas prepared from adulterated and authentic rhizomes.

“Elements of Truth” – the Food and Environmental Protection Laboratory’s New Shimadzu Bench-Top Energy Dispersive X ray Fluorescence (EDXRF) Spectrometer for Food Authenticity and Safety Analyses

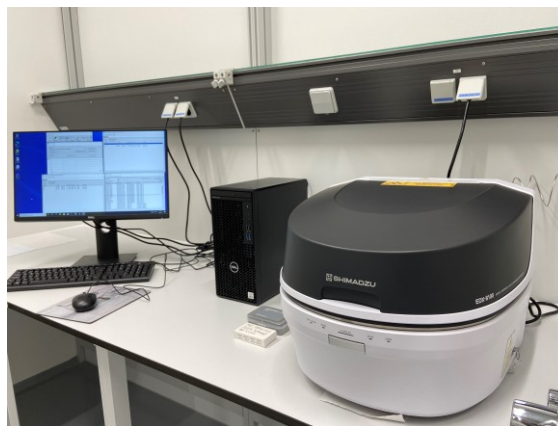
Simon Kelly

In April 2021 the Food and Environmental Protection Laboratory installed a Shimadzu 8100 bench-top Energy Dispersive X ray Fluorescence (EDXRF) spectrometer to conduct rapid elemental profiling of foods to confirm their authenticity and to screen for excessive concentrations of some elements that may pose a threat to food safety. The

instrument was procured under the Peaceful Uses Initiative project ‘Enhancing Capacity in Member States for Rapid Response to Food Safety Incidents and Emergencies’, which is funded by the government of Japan. In recent years, significant technological progress has been made in the development of EDXRF spectrometers in terms of their portability, meaning that in addition to traditional floor-standing instrumentation, bench-top and hand-held versions are available. This makes the technique amenable to on-site and point-of-contact analyses, respectively.

One of the main advantages of the EDXRF technique is that it permits the measurement of elements from sodium to uranium with minimal sample pre-treatment. Solid food samples must be dried and milled to obtain a homogeneous powder. This is markedly quicker and simpler than sample preparation for other elemental analysis techniques such as Inductively Coupled Plasma – Mass Spectrometry (ICP-MS), where the food must be dried, milled, microwave-digested in concentrated acid and diluted prior to analysis. Usually, for EDXRF, the dried and powdered food is simply pressed to form pellets that are irradiated and the elemental composition measured under vacuum. The Shimadzu 8100 EDXRF system in the FEPL has the further advantage that it can measure ‘loose’ powders directly, in an inert helium gas atmosphere, without the need to compress the food sample into a pellet. This can be particularly advantageous for the rapid screening of foods already sold in powdered form, such as dried milk powder, cereal flour, cocoa and spices such as turmeric and saffron powder. Furthermore, liquid and semi-liquid foods, such as honey, can be directly measured without the need for sample pre-treatment. However, it should be noted that whilst these advantages have the potential to further speed up the food sample elemental screening process this can have the effect of reducing the sensitivity of the analysis, taking some elements below the detection limit of the instrument. Nevertheless, EDXRF is characterized by limits of detection (LODs) in the low mg/kg (ppm) level, which are often adequate for elemental profiling to obtain characteristic ‘fingerprints’ that are useful for both geographical origin determination and questions of production origin, such as organic versus conventional, when the multi-element data is processed with the aid of chemometrics. Very recently, EDXRF has been successfully applied to the unsupervised elemental pattern recognition between organic and conventional retail samples of bovine milk, basmati rice, cane sugar, chocolate, cinnamon, coffee, coconut water, honey, paprika powder and wheat flour. The technique has also been successfully applied to the geographical origin discrimination of Protected Designation of Origin (PDO) chestnut honeys from different parts of Spain.

Despite these recent food applications, generally EDXRF is mainly applied to the analyses of soils, sediments, minerals, alloys, metals and inorganic matrices. The FEPL will be applying this the state-of-the-art bench-top EDXRF to analyse organic food matrices for authentication purposes and transferring the technology to members states so that it can be eventually deployed for rapid screening and enforcement work to confirm if added value claims about food origin and production methods are indeed truthful. To this end the EDXRF system will initially be used to generate elemental fingerprints for a wide range of premium foods, produced by developing Member States, in the CRP D52042, for example; Taliouine saffron (GI) from Morocco; Chinese Jinxiang Garlic (PGI); Jamaican Blue Mountain Coffee (GI); and Thai Jasmine rice (GI). In addition, it will be used to develop heavy and toxic metal screening methods for powdered milk samples from CRP D52040 “Field-deployable analytical methods to assess the authenticity, safety and quality of food”.

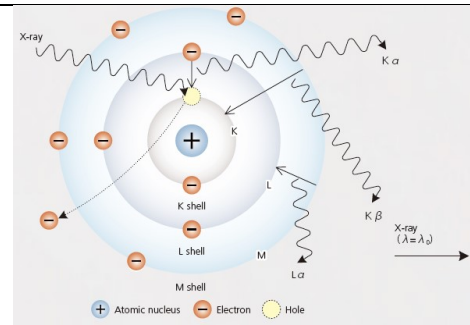


The new Shimadzu bench-top Energy dispersive X ray fluorescence (EDXRF) 8100 spectrometer in the Food and Environmental Protection Laboratory at the IAEA Yukiya Amano Laboratory, Seibersdorf.

Visiting fellows, trainees and interns will also have an opportunity to learn operational and analytical methods on the EDXRF, thereby supporting Member State capacity building and raising awareness of the technique. As the supply chains that deliver foodstuffs to our doors grow ever more complex, bringing incredible choice to consumers, so it becomes more difficult to ensure that we can trust those foods. EDXRF is a technique which can be used to rapidly detect fraudulent or mislabelled premium and protected foods and reduce the risks of unintended food safety incidents associated with adulteration and toxic metal contamination. The new EDXRF system also presents the opportunity for collaboration with the other FAO/IAEA Agriculture & Biotechnology Laboratories for research projects that require elemental analysis.

Principle of Fluorescent X ray Generation

When a sample is irradiated with X rays from an X ray tube, the atoms in the sample generate unique X rays that are emitted from the sample. Such X rays are known as "fluorescent X rays" and they have a unique wavelength and energy that is characteristic of each element that generates them. Consequently, qualitative analysis can be performed by investigating the wavelengths of the X rays. As the fluorescent X ray intensity is a function of the concentration, quantitative analysis is also possible by measuring the amount of X rays at the wavelength specific to each element of interest.



Electron paths and the principle of fluorescent X ray generation expressed as a Bohr model of the atom. (Reproduced with permission of the Shimadzu Corporation)

FEPL Staff

Since the start of 2021, there has been one staffing change in FEPL. In March, Ms Joanna Mletzko, who provided administrative support as team assistant for the three laboratories in the Yukiya Amano Laboratories (YAL); FEPL, the Soil and Water Management and Crop Nutrition Laboratory (SWMCNL) and the Animal Production and Health Laboratory (APHL), took advantage of the Agency's staff mobility mechanisms to move to the Office of Procurement Services on a temporary development

reassignment. Ms Aminata Faustmann took over Joanna's duties in supporting the staff in YAL. We wish Joanna all the best in her current assignment and welcome Aminata to the YAL team.

We are pleased to report that Ms Sofia Rezende, from the University of the Republic, Uruguay, who joined FEPL as a PhD consultant in January 2020 initially for a one-year period, has extended her stay with FEPL until the end of 2021. This will allow Sofia to complete her research, which was interrupted in 2020 due to COVID-19 disruptions.

Announcements

One Day Virtual African Food Safety Workshop, 4 August 2021

James Sasanya

This half-day event will be held in partnership with the National Metrology Institute of South Africa (NMISA). The purpose is to deliberate on current food safety technical matters related to consumer safety and trade, as preamble for a physical Africa-wide food safety workshop planned for June/July 2022, a follow-up of a similar event held in June 2018. This became a biennial event but couldn't held in 2020 and 2021 due to COVID-19.

This workshop will among others address the following: Role of food safety laboratories in the African Continental Free Trade Area (AfCFTA) and associated challenges including standards and export rejections; reliable testing laboratory services and role of non-technical stakeholders. Participants will also discuss how laboratories align with sanitary and phytosanitary regulatory systems, including relevant requirements, implementation and corrective measures. Other areas to deliberate on will include: regional

efforts in contributing to the process of setting standards for residues and contaminants as well as public-private partnerships that can enhance food safety laboratory operation and sustainability.

Call for Nominations for the Joint FAO, IAEA and Latin American and Caribbean Analytical Network (RALACA) Workshop on Food Contaminant Testing and Risk Assessment Programmes, 12–14 October 2021

Britt Maestroni

Effective food control systems are needed to ensure a safe and wholesome food supply on a global basis. One key element of such a system is the provision of feedback on the effectiveness of agricultural practices in producing food that is safe and meets the requirements for international trade. In this regard, analytical laboratories are essential to help establish or contribute to ongoing monitoring and surveillance schemes. Testing of agricultural produce is important to maintain consumer confidence, ensure food

safety, and facilitate international trade and the provision of safe food supplies. Data on the concentrations of chemical residues and contaminants in food are required to enable the estimation of population exposure to those chemicals. In general, food is analysed as it is commercialized, and the major objective is the enforcement of legal limits. Implementing monitoring schemes is challenging, especially for developing countries, and requires strong networking, research, human resource development and capacity building in the field of food control systems and risk assessment, and efficient food testing infrastructure.

To help Member States in this regard the Joint FAO/IAEA Centre will hold a Joint FAO, IAEA and Latin American and Caribbean Analytical Network Workshop on Food Contaminant Testing and Risk Assessment Programmes, hosted by MIDA, Panama City on 12–14 October 2021. The purpose of the workshop is to share and communicate scientific and technological developments, to identify gaps in knowledge and to discuss ways in which nuclear and

isotopic methodologies can improve food safety testing and risk assessment programmes. The workshop will focus on rapid techniques for food contaminant testing applied to pesticide residue analysis in food, and the contribution of analytical data to risk assessment. Experts will be available to discuss current approaches and provide advice to help facilitate the implementation of relevant programmes and guidelines. The workshop will also provide a forum for networking and will highlight current initiatives of the “Red Analitica de Latino America y el Caribe (RALACA)” network. Intensive theoretical training and laboratory demonstrations will be provided during the workshop.

The call for nominations is currently open for analysts from laboratories authorized by Governments to perform analyses for official control of food, or responsible for risk assessment programmes. All persons wishing to participate in the event must be designated by an IAEA Member State or should be members of organizations that have been invited to attend.

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2021

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