

## Food & Environmental Protection Newsletter

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Joint FAO/IAEA Programme Nuclear Techniques in Food and Agriculture

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## **To Our Readers**



Peruvian Grapes for the market.

It is our sincere hope and wish that you and your loved ones are in good health during this extended COVID-19 pandemic that has impacted our common activities and service delivery. We commiserate with those that have fallen victim to the pandemic in one way or another and hope for faster recovery. Since our last newsletter in July 2020, which reported on the challenges presented by the COVID-19 pandemic to the work of the Food and Environmental Protection (FEP), the Section has continued to support more than 70 technical cooperation projects (TCPs) and six coordinated research projects (CRPs). A couple of additional new CRPs are in the

pipeline. Please see selected highlights in related articles in this newsletter.

For the TCPs, greater focus was placed on implementing procurement of equipment and laboratory supplies, which has been quite successful. Where supply and delivery of procurements has been prompt, this has greatly facilitated food safety testing activities in Member States. Food safety and quality laboratories have generally remained active in Member States to ensure that consumers receive safe and quality food. Laboratory supplies have therefore been very critical during these times.

While coordination meetings and capacity building activities through physical interactions have not been possible and several have been postponed to 2021, a number of virtual activities have been implemented. For example, an ad-hoc, one-day (2 July 2020) coordination meeting for the regional (Latin America and the Caribbean) food safety project on "Improving Regional Testing Capabilities and Monitoring Programmes for Residues/Contaminants in Foods Using Nuclear/Isotopic and *Complementary* Techniques" (RLA5081) was organized to finetune workplans and address urgent needs. Another two-day virtual meeting held 27 and 29 October on "The Future of Data in Food Safety. Sharing Experiences and Lessons Learned with the European Food Safety Agency (EFSA)" was organized under a related regional project "Strengthening the Regional Collaboration of Official Laboratories to Address Emerging Challenges for Food Safety" (RLA5080).

A two-day (21–22 September 2020) collaborative Global Minor Use Priority Setting Workshop was also held in collaboration with the Minor Use Foundation. More than 180 participants from Asia, Africa as well as Latin America and the Caribbean, among others, attended. Such events helped to enhance global efforts to establish collaboration among food safety partners in identifying priority crops/compounds and planning for generation of residue data needed to set up maximum residue limits and promote fair trade.

Another virtual meeting attended by 29 participants in 18 countries of the Asia and Pacific region was held from 3 to 4 September 2020, to initiate a TC project on promoting food irradiation by electron beam and X ray technologies. Furthermore, an online food irradiation network (FInet) was initiated with specialists in Asia and Pacific countries taking part virtually.

FEP also participated in the 11th meeting of the Emergency Preparedness and Response Standards Committee (EPReSC) held virtually between 2 and 4 November 2020 to consider and approve a range of radiation safety standards and guidelines. We also participated in the review and assessment of food products in Japan as part of the Agency's review of Comprehensive Fukushima reports from Japan (June and October).

On the global scene, FEP joined Member States to commemorate the World Food Safety Day<sup>1</sup> and the World Food Day<sup>2</sup>. FEP also supported Indonesia's commemoration of World Food Day by participating in an international webinar on 22 October 2020 in Jakarta, during which FEP delivered a presentation on "Achieving quality/Safety of Food/Agriculture and Environment to Meet the Needs of People During COVID-19 – A system's perspective".

Undeterred by the COVID-19 challenges, the Food and Environmental Protection Laboratory (FEPL) continued to perform its R&D activities and provide outreach services as well as technical guidance to Member States. This newsletter presents details of the work done including but not limited to: (1) "Chemometrics Add-in for Excel" E-learning course under a regional project RAS5081 for Asia-Pacific (16–20 November 2020); (2) regional project support; (3) webinars; (4) state-of-the-art analytical instrumentation; (5) analytical method development and innovative sample preparation; (6) collaborating centre(s); (7) training courses; and (8) laboratory networking. FEP has continued to support laboratory and food safety networking initiatives in Latin America and the Caribbean, Africa and Asia. Some highlights are presented in this Newsletter.

This newsletter also presents a feature article focussing on food irradiation and our collaboration with Member States including the IAEA Collaborating Centre framework. Collaborating Centres help the Agency in the implementation of targeted IAEA programmatic activities.

It is with mixed feelings to inform you that our leader Mr Zhihua Ye retired from the IAEA at the end of August 2020 after five years of dedicated service to several FAO and IAEA Member States in his capacity as Section Head for FEP and Technical/Project Officer. As a frequent follower of our work, you may have received the last nine newsletters if not more from him. While we are happy that he now enjoys well-deserved rest following several years of service in China and the Joint FAO/IAEA Division, his mature, respectful, ever cordial and caring leadership will be greatly missed.

Mr Ye made significant contributions to the improvement of food safety and control systems in Member States, promoting the comparative advantage of nuclear technology in food irradiation; the control of residues and contaminants in food and the environment; food traceability/authenticity as well as nuclear emergency preparedness and response. At least 119 TCPs and 10 CRPs were implemented or initiated during his tenure. Through his leadership, more than 12 Member States have been able to access, maintain or expand export markets for their food as a result of their improved

<sup>&</sup>lt;sup>1</sup> <u>https://www.iaea.org/newscenter/news/world-food-safety-day-nuclear-techniques-used-to-keep-our-food-safe</u>

<sup>&</sup>lt;sup>2</sup> <u>https://www.iaea.org/newscenter/news/world-food-day-2020-iaea-faos-joint-work-benefits-farmers-and-increases-food-security-worldwide</u>

food safety systems; more than 20 food safety/quality laboratories have been established, many from scratch; over 35 others have been enhanced or modernized; and at least 34 laboratories were accredited. Food irradiation and traceability/authenticity capabilities were also introduced or advanced in more than 35 Member States.

Furthermore, during Mr Ye's tenure as FEP's Section Head, at least three major workshops were organized on: (1) remediation of radioactive contamination in agriculture (Vienna, Austria, 2016); (2) African food safety (South Africa, 2018); and (3) Latin America food safety (Brazil, 2019). He also played a role in the planning of the International Forum on Food Safety and Trade, "The Future of Food Safety – Transforming knowledge into action for people, economies and the environment" (April 2019, Geneva, Switzerland) as well as the First FAO/WHO/AU International Food Safety Conference (12–13 February 2019, Addis Ababa, Ethiopia).

Since our last newsletter, a number of success stories have been reported, including: (i) 'Senegal Increases Self-Reliance in Food Safety Control through Nuclear Science'<sup>3</sup>; (ii) 'Boosting Namibia's Fish and Fishery Product Exports through Support from IAEA and FAO'<sup>4</sup>; (iii) 'In Seychelles, Nuclear Science Helps Safeguard Consumers' <sup>5</sup>; (iv) 'Nuclear Techniques Help Thai Food Exports' <sup>6</sup>; and (v) 'Irradiation Secures Viet Nam's Fruit Exports'<sup>7</sup>.



Mr Ye (extreme right, front row) and Ms Battaglia (extreme left, second row) and other FEP staff implementing COVID-19 protocol.

Another colleague, Ms Daniella Battaglia, also left the IAEA, concluding a three-year service as Technical Officer at the Joint FAO/IAEA Division where she supported both FEP and Animal Production and the Health Subprogrammes. She returned to FAO Headquarters in Rome at the end of August 2020. Some of the countries she supported include, among others, Cambodia, Dominica, Morocco, Myanmar, Niger, Pakistan, Syrian Arab Republic and Uganda. She also supported our work on standards and guidelines for radionuclides in food and water. Her services were appreciated, and we wish her all the best in her next assignment.

Once again, may you and your loved ones have good health and better days ahead soon.

James Jabob Sasanya

Acting Head, Food and Environmental Protection Section

<sup>&</sup>lt;sup>3</sup> <u>https://www.iaea.org/newscenter/news/senegal-increases-self-reliance-in-food-safety-control-through-nuclear-science</u>

<sup>4 &</sup>lt;u>https://www.iaea.org/newscenter/news/boosting-namibias-fish-and-fishery-product-exports-through-support-from-iaea-and-fao</u>

<sup>5 &</sup>lt;u>https://www.iaea.org/newscenter/news/in-seychelles-nuclear-science-helps-safeguard-consumers</u>

<sup>6 &</sup>lt;u>https://www.iaea.org/newscenter/news/nuclear-techniques-help-thai-food-exports</u>

<sup>7 &</sup>lt;u>https://www.iaea.org/newscenter/news/irradiation-secures-viet-nams-fruit-exports.</u>

## Staff

## Joint FAO/IAEA Division of Nuclear Techniques in Food and Agriculture

Name	Title	Email	Extension	Location
Qu Liang	Director	Q.Liang@iaea.org	21610	Vienna

### Food and Environmental Protection Subprogramme

Name	Title	Email	Extension	Location
Carl M. Blackburn	Food Irradiation Specialist	C.Blackburn@iaea.org	21639	Vienna
James J. Sasanya	Food Safety Specialist (Veterinary Drug Residues)	J.Sasanya@iaea.org	26058	Vienna
Kyoko Narikawa	Team Assistant	K.Narikawa@iaea.org	26061	Vienna
Malgorzata Rydeng	Team Assistant	M.Rydeng@iaea.org	21641	Vienna
Andrew Cannavan	Laboratory Head	A.Cannavan@iaea.org	28395	Seibersdorf
Simon Kelly	Food Safety Specialist (Traceability)	S.Kelly@iaea.org	28326	Seibersdorf
Britt M. Maestroni	Food Scientist	B.M.Maestroni@iaea.org	28398	Seibersdorf
Alina Mihailova	Analytical Chemist (Food Authenticity)	A.Mihailova@iaea.org	28373	Seibersdorf
Aiman Abrahim	Laboratory Technician	A.Abrahim@iaea.org	28327	Seibersdorf
Marivil Islam	Laboratory Technician	M.Islam@iaea.org	28394	Seibersdorf
Joanna Mletzko	Team Assistant	J.M.Mletzko@iaea.org	28362	Seibersdorf
Serik Permetov	Lab Cleaning Attendant	S.Permetov@iaea.org	28397	Seibersdorf
Shuichi Nakaya	Analytical Chemist (Food Control)	S.Nakaya@iaea.org	27307	Seibersdorf
Florence Maxwell	Laboratory Technician	F.Mawell@iaea.org	27482	Seibersdorf

Food and Environmental Protection Section Vienna International Centre, PO Box 100, A-1400 Vienna, Austria Tel.: (+) 43 1 2600 + Extension; Fax: (+) 43 1 26007; Email: Official.Mail@iaea.org

Food and Environmental Protection Laboratory FAO/IAEA Agriculture and Biotechnology Laboratories A-2444 Seibersdorf, Austria Tel.: (+) 43 1 2600 + Extension; Fax: (+) 43 1 26007; Email: Official.Mail@iaea.org

> <u>http://www-naweb.iaea.org/nafa/fep/index.html</u> <u>http://www-naweb.iaea.org/nafa/fep/fep-laboratory.html</u> <u>http://www.fao.org/ag/portal/age-index.html</u>

## **Feature Article**

## Food Irradiation and Aerial as an IAEA Collaborating Centre

#### Carl Blackburn

The IAEA Collaborating Centres Scheme<sup>8</sup> provides the Agency with a means of designating eligible institutions as partners to assist in implementing selected IAEA programmatic activities. This scheme was initiated by the IAEA Department of Nuclear Sciences and Applications on a three-year pilot basis in 2004. After a successful pilot phase, the IAEA Director General approved the IAEA Collaborating Centres Scheme in 2008 and expanded it to all interested IAEA departments.

Aerial<sup>9</sup> is a not-for-profit, French Technology Resources Centre and Technical Institute for Food Industry. It is located at Illkirch, near Strasbourg, France and is designated as an IAEA Collaborating Centre for multidisciplinary applications of electron beam or X-ray technologies and related dosimetry, especially for food irradiation. Its work is being reviewed following a successful five-year period of formal collaboration and is submitting a request for a renewal of its IAEA Collaborating Centre status. Therefore, this gives me the opportunity to both showcase the achievements of this collaboration and illustrate how the Collaborating Centres Scheme can work in practice.

The formal collaboration started with a ceremony at Aerial on 6 October 2016 during which the IAEA Collaborating Centre plaque was presented to Mr Alain Strasser, General Manager Directeur, by Mrs Nathalie Colinet of the Department of Nuclear Applications of the IAEA at the time. In the months prior to this, a formal written expression of interest had been sent to the IAEA by Aerial. after a positive assessment, representatives of Aerial and the IAEA Food Irradiation Specialist prepared a collaborating centre proposal and workplan. The proposal was presented to the appropriate IAEA Committee for Coordinated Research Activities where it was approved. It was also endorsed by the Permanent Mission of France to the United Nations and International Organizations in Vienna. An agreement document was therefore produced and signed by both parties. The agreement contained, the undertakings of both Aerial and the IAEA, the duration of the designation as an IAEA Collaborating Centre as well as a Work Plan that detailed objectives, activities, plus expected results and outcomes.



Mr Alain Strasser, Aerial General Manager Directeur and Ms Nathalie Colinet of the IAEA at the formal inauguration ceremony of the Collaborating Centre in 2016.

The work undertaken in this collaboration included research to develop improved dosimetry techniques and practices, studies into food irradiation using low energy beams, work on the detection of irradiated food, evaluation of foods irradiated with high energy X-rays, radiation processing and cryogenic applications and the Faisceau d'Electrons Et Rayonnement Ionisants X) (FEERIX) project to build a new irradiation facility specifically designed for research, innovation and training activities.



The new rhodotron electron accelerator installed at Aerial in 2019.

<sup>&</sup>lt;sup>8</sup> https://www.iaea.org/about/partnerships/collaborating-centres

<sup>&</sup>lt;sup>9</sup> https://www.aerial-crt.com/en/

The FEERIX resulted in a new facility at Aerial housing a rhodotron electron accelerator with two beam lines that can operate at high energies of up to 10 MeV for electrons and 5 or 7 MV for X-rays. Therefore, Aerial now has four irradiation facilities that span low, medium and high energy beam irradiation and can be used to support both food and non-food activities to strongly reinforce its capabilities for research, training and technology transfer to IAEA Member States in radiation processing.

During the 2016–2020 designation period, Aerial has been quick to respond to the IAEA's requests for support. For example, it has assisted in reaching industrial collaborators when designing new coordinated research activities related to innovating radiation processing of food with low energy beams from machine sources. Technical outputs to support the food irradiation activities have included: two book chapters, 13 manuscripts in scientific journals, 15 posters presented at technical meetings and 17 conference presentations. Through working as a collaborating centre, Aerial has also provided technical support to colleagues in NAFA concerning dosimetry of low energy X-rays for the sterile insect technique. The institute accomplished the workplan that was established in 2016 and its contribution to IAEA programmatic activities have included:

- Technical input at six IAEA meetings (including the Agency's International Conference on Applications of Radiation Science and Technology and the meeting of IAEA Collaborating Centres);
- Helping to design two Coordinated Research Projects (Innovating radiation processing of food with low energy beams from machine sources, plus radiation effects on polymeric materials);
- Performing two dosimetry inter-comparison exercises for 12 countries;
- Providing cost-free training on good dosimetry practices;
- Undertaking original research on the potential of high-energy X-rays to induce radioactivity in food (in collaboration with Institut pluridisciplinaire Hubert Curien, a national research centre). This is in support of extending international food irradiation standards of the codex Alimentarius to increase the X-ray energy for the irradiation of foods from 5 MeV to 7.5 MeV;

- Hosting a Ph.D. studentship and producing a thesis on high energy X-ray activation;
- Hosting the three-yearly conference on radiation processing. The 2019 International Meeting on Radiation Processing (IMRP19) was held in Strasbourg, France and attended by over 500 international delegates;
- Constructing and qualifying a new high-energy irradiation facility. The designation as an IAEA collaborating Centre helped Aerial to cement financial support from both national and local level funding organizations;
- Engaging with the European Commission on the need for new European Union food irradiation directives.

The Aerial Collaborating Centre also assisted FAO and IAEA Member States by participating in activities funded by the IAEA Department of Technical Cooperation such as hosting eight scientific visits and IAEA fellowships, undertaking nine expert missions to assist institutions in IAEA member states and a further twenty missions to install dosimetry equipment and provide related training.

The Food and Environmental Protection subprogramme currently receives programmatic support from four collaborating centres and a further two applications are in the pipeline. The four current collaborating centres are: The National Center for Electron Beam Research at Texas A&M Agri Life Research (College Station, USA) for collaboration on Electron beam technology for food, health and environmental applications; Centro de Investigación en Contaminación Ambiental (Ciudad Universitaria Rodrigo Facio, Costa Rica) centre for collaboration on eLearning and accelerated capacity building for food and environmental protection; The Advanced Radiation Technology Institute, Korean Atomic Energy Research Institute (Jeongeup, Republic of Korea) collaborating centre on radiation applications for environmental remediation, advanced materials, foods and biotechnology; and Aérial (Illkirch, France, for multidisciplinary applications of electron beam and X-ray technologies and related dosimetry, especially for food irradiation.

## **Forthcoming Events**

### **Research Coordination Meetings of FAO/IAEA Coodinated Research Projects and Training Courses**

Consultancy Meeting on Irradiation Technology for Phytosanitary Treatment of Food Commodities and Promotion of Trade, Vienna, Austria, 22–26 February 2021.

Final Research Coordination Meeting on Development and Strengthening of Radio-Analytical and Complementary Techniques to Control Residues of Veterinary Drugs and Related Chemicals in Aquaculture Products (D52039- CR- 4), Vienna, Austria, 1–5 March 2021.

Fourth Research Coordination Meeting on the Development of Electron Beam and X-Ray Applications for Food Irradiation (DEXAFI) (D61024-CR-4), Vienna, Austria, 6–9 April 2021.

Third Research Coordination Meeting on Integrated Radiometric and Complementary Techniques for Mixed Contaminants and Residues in Foods (D52041-CR-3), Vienna, Austria, 26–30 April 2021.

First Research Coordination Meeting on the Innovation of Irradiation Technologies on Surface Treatment of Food Commodities (D61025-CR-1), Vienna, Austria, 17–21 May 2021.

First Research Coordination Meeting on Depletion of Veterinary Pharmaceuticals and Radiometric Analysis of their Residues in Animal Matrices (D52043-CR-1), Vienna, Austria, 17–21 May 2021.

Training Course on the Detection and Control of Organic Contaminants in Food — Laboratory Component, Seibersdorf, Austria, 7–11 June 2021.

Third Research Coordination Meeting on Field-deployable Analytical Methods to Assess the Authenticity, Safety and Quality of Food (D52040-CR-3), Vienna, Austria, 14–18 June 2021. Second Africa Food Safety Technical Meeting, Johannnesburg, South Africa, 28 June–2 July 2021 (Virtual).

Consultants Meeting (CM) on Nuclear Techniques to Support Risk Assessment of Biotoxins in Food and Related Matrices, 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), Kingston, Jamaica, 1–5 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**

Regular Meeting of the Inter-Agency Committee on Radiation Safety (IACRS), Vienna, Austria (TBC).

Codex Committee on Residues of Veterinary Drugs in Foods (CCRVDF25), San Diego, USA, (TBC).

IAEA Technical Meeting Innovating Radiation Processing of Food with Low Energy Beams from Machine Sources, Dresden, Germany, (TBC).

## Past Event

## International Collaboration to Support MRL-Setting Initiatives— Virtual Global Minor Use Foundation Workshop, 21–22 September 2020

#### James Sasanya

A two-day (21–22 September 2020) collaborative Global Minor Use Priority Setting Workshop was held through the Minor Use Foundation and attended by more than 180 participants including growers, industry/product manufacturer's bodies, researchers, minor use programme managers, government officials and laboratory personnel from 38 countries in Asia, Africa, Europe, Latin America, North America and Oceania.

The workshop participants met to discuss and make decisions on the top ten minor use projects to progress beginning in October 2020 through September 2021. A wide range of tropical, temperate and greenhouse crops, among others were addressed. The list of over 4000 global crop protection needs from at least 56 countries has now been prioritized to ten. These include include: 1) Tuta absoluta on tomatoes; 2) Western flower thrips on ornamentals; 3) Spotted Wing Drosophila on raspberry; 4) Downy mildew on dry bulb onion; 5) Downy mildew on hops; 6) Spotted Wing Drosophila on blueberry; 7) Downy mildew on green onion (where possible to integrate with dry bulb); 8) Phytophthora on avocado; 9) Anthracnose on mango; and 10) Phytophthora on pineapple. There is also room for flexibility.



Fig. Pineapple, one of the crops targeted for data generation to support MRL setting.

A detailed analysis of the work required and capacity available to conduct the relevant research and data generation for new crop protection solutions for these priority needs is now ongoing and has drawn interest from several stakeholders.

Events such as this workshop help to enhance global efforts to establish collaboration among food safety partners in identifying priority crops/compounds and planning for generation of residue data needed to set up maximum residue limits (MRLs) and promote fair trade.

## Virtual Panel Discussion on Isotope Fingerprints: The Common Thread Between Forensic Applications—from Food and Beverage Adulteration to Crime Scenes Investigations, 6 October 2020

#### Simon Kelly

ThermoFisher Scientific organised an open access webinar in the form of a panel discussion on "Isotope Fingerprints: The common thread between forensic applications—from food and beverage adulteration to crime scenes investigations", which took place on 6 October 2020. The webinar was hosted by Dr Mario Tuthorn and Dr Christopher Brodie from ThermoFisher Scientific and the panellists were; Dr Christy Mancuso, Forensic Isotope Specialist, University of Mexico; Dr David Psomiadis, Head of the Laboratory and Business Development Manager, Imprint Analytics GmbH, Austria; and Mr Simon Kelly, IAEA Food and Environmental Protection Laboratory (FEPL). Over one hundred and twenty registered delegates attended the panel discussion through on-line participation.

The webinar was designed to provide background information on the use of stable isotope analysis in forensic biogeochemical covering studies cycles, archaeological/criminalistic materials, foods and beverages, officially recognised methods for foods, criminal forensics and interlaboratory data comparison, drawing on expertise from private laboratories, governmental agencies and academic research institutes. The panellists explained how isotope fingerprints are incorporated and preserved in foods and forensic materials, and shared their experience with state-of-the-art applications of isotope ratio mass spectrometry analysis and their views of future perspective in forensics investigations. The audience members then had the opportunity to ask for expert advice on the methodology, compliance with official methods, the challenges of laboratories working towards the same standards, and discuss the importance of detecting origin and authenticity in the context of real-world application. Mr Kelly was invited to give a pre-recorded scientific lecture on the principles of light-element stable isotope systematics in biogeochemical cycles and how these underpin the

application of stable isotope analysis in forensic studies. The presentation covered understanding how we can use hydrogen and oxygen stable isotopes to obtain geographical information for our forensic studies on food, and other biological materials: how we can exploit the distribution of carbon-13 in different reservoirs in the environment; a focus on how the use of synthetically derived nitrogen in fertilisers impacts on the stable nitrogen isotope values in conventionally cultivated versus organic crops; and finally the complexity of sulfur isotope signals and how these can be interpreted to obtain an indication of geographical origin. Mr Kelly also presented information about new food matrix stable isotope reference materials that were characterised by a consortium including FEPL and officially recognised stable isotope methods from the European Committee for Standardization (CEN), the Association of Official (AOAC), Analytical Chemists the International Organisation of Vine and Wine (OIV) and the International Codex Alimentarius Standard 234. The other panellists. Dr Christy Mancuso, presented the use of stable isotope analysis in criminal forensics with reference to a particular case where it was successfully applied to unravel mysteries around unidentified human remains at the Great Salt Lake near Salt Lake City, USA. Dr David Psomiadis presented the application of stable isotope methods, in a commercial setting, applied to the verification of the geographical origin of fruits and vegetables, olive oil; the authenticity of wine and the control of vanilla flavour and caffeine.



Contributors to the virtual Panel discussion on Isotope Fingerprints: The common thread between forensic applications - from food and beverage adulteration to crime scenes investigations, 6 October 2020.

Mr Kelly then participated in a 20-minute live question and answer session with the other panel members. Viewers of the webinar asked questions related to the panel members' presentations covering what other official methods for food authenticity are in the pipeline; opinions about creating database for authenticity verification versus using material of known origin for direct comparison; the application of isotope analysis used in legal actions in criminal forensics; choosing the right instrument setup when measuring several isotopes on one system; and compound-specific analysis in complex matrices and identifying compounds in such samples. The feedback obtained by the organisers was very positive. In addition, the webinar is free to view on-demand<sup>10</sup>:



Mr Kelly presenting the variation in sulfur isotope ratios in the marine and terrestrial environment during the Virtual Panel discussion on Isotope Fingerprints, 6 October 2020.

### International Fruit and Vegetable Juice Association Technical Webinar: Authenticity, 15 October 2020

#### Simon Kelly

The International Fruit and Vegetable Juice Association (IFU) Technical Webinar on authentication methods took place on 15 October 2020. The webinar was organized as a replacement for the IFU Technical Workshop in 2020, which had to be cancelled as a live event due to the Corona pandemic. IFU Technical Workshops are the meeting point for the national and international juice industry, including research and development, quality, laboratories, suppliers and universities to discuss the latest developments in methodology related to the safety, quality, and authenticity of fruit juice. Over one hundred and fifty delegates attended the webinar through on-line participation.

Mr Simon Kelly was invited to give a pre-recorded scientific lecture on a new rapid method using hydrogen stable isotope analysis to detect undeclared addition of sugar to fruit juice. The improved procedure for the isotope analysis was developed in the FEPL and utilizes a simple one-step chemical reaction that makes sugars sufficiently volatile to be separated and measured by gas chromatography coupled to isotope ratio mass spectrometer. The conversion of the derivatised sugars into the measuring gas is achieved using a high temperature chromium-silver reactor that retains carbon, oxygen and fluorine whilst releasing hydrogen gas for stable isotope measurement. The new procedure has

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https://event.on24.com/wcc/r/2665786/9158FE201606FA6C0EFE25044 5CB04FD/1565634?mr=s

advantages over methods using nitro-sugar derivatives, sugar degradation products and fermentation in terms of ease of use, analysis time and sensitivity. The potential of the technique for detecting economically motivated adulteration of foods and beverages was discussed with illustrations of the differences between the isotope abundance of the nonexchangeable hydrogen in sugars from authentic pineapple juice and those of beet and cane sugars/syrups, which permits the presence of these potential adulterants to be rapidly detected. Other speakers in the webinar covered topics such as quality and authenticity observations in the juice market, new analytical insights for lemon and lime juice analysis and other new isotopic techniques for the analysis of pineapple juice.

Mr Kelly then participated in a 30-minute live question and answer session with Dr Susanne Koswig of Sure Global Fair (SGF) Producers Association. Mr Markus Jungen of SGF. and Mr Mikko Hofsommer of Gesellschaft für Lebensmittel-Forschung (GfL) GmbH. Viewers of the webinar asked questions related to the Mr Kelly's presentation regarding the comparability of measurements from the new rapid method to measurements using the previously described offline methods. Mr Kelly referred to the findings of the FEPL study published in Food Chemistry (2020; 126413), which demonstrated the comparability of results from the new method, using trifluoroacetate-sugar derivatives, for the hydrogen isotope analysis of carbon-bound nonexchangeable hydrogen on saccharides with that obtained with nitro-sugar derivatives. Mr Kelly also answered questions regarding the analytical set up of the gas chromatography—isotope ratio mass spectrometry system and the technical modifications made to make the system more resilient against hydrogen fluoride and fluorine gas.



Mr Kelly giving an invited lecture at the International Fruit and Vegetable Juice Association (IFU) Technical Webinar: Authenticity, 15 October 2020.



From the top left, Mr Kelly (IAEA), Mr Markus Jungen (SGF), Mr Mikko Hofsommer (GfL) and Dr Susanne Koswig (SGF) with the Webinar host Ms Aintzane in a live question and answer session at the at the International Fruit and Vegetable Juice Association (IFU) Technical Webinar: Authenticity 15 October 2020.

## International Webinar: World Food Day 2020 Commemoration in Indonesia, 22 October 2020

#### James Sasanya

Indonesia joined the rest of the world to commemorate the 2020 World Food Day and one of the events held was an international webinar under the theme "Achieving food quality/safety of food/Agriculture and Environment to Meet the Need of The People During COVID-19". This was organized by the Indonesian Environmental Expert Forum Association, locally referred to as Perkumpulan Forum Tenaga Ahli Lingkungan Indonesia (P-TALI) in cooperation with three other institutions: Indonesian Center for Agricultural Land Resource Research and Development; Indonesian Agency for Agricultural Research and Development; and Ministry of Agriculture.

An estimated 264–300 participants attended the event, whose aim was to among others, increase public awareness of the importance of handling food in a sustainable and environmentally friendly manner and especially in COVID-19 times. A number of presentations were delivered with the Officer speaking on "Achieving Quality/Safety of Food/Agriculture & Environment to Meet the Need of The People During COVID-19 2020"—A systems' perspective. The talk included the effect of COVID-19 on the food supply, access and inputs; as well as continued need for safe and quality food in emergency and normal situations. The Officer also elaborated on the food safety/quality chain and the role of the various stakeholders from production/farm through harvest (pre- and post-); storage and distribution; transportation; handling; factory/industry; markets and at home.

The Officer also presented on the unique role of the laboratory in ensuring safe and quality food supply including supporting emergency food supplies for the vulnerable. He noted that the laboratories, however encountered challenges such as: over-seas or cross boarder capacity building trainings that couldn't be implemented affecting skillimprovement. Therefore, laboratories in-country must work towards being competent enough in preparation for difficult situations such as COVID-19, and train analysts locally. Similarly, installation, maintenance or service of equipment were affected due to COVID-19 related restrictions. Routine operation of the laboratories was also affected due to reduction in staffing; socio-distancing in laboratories (not so easy to do), as well as the urgent demand to conduct multiple tasks. For instances some food safety laboratories were required to participate in COVID-19 testing as well. Another challenge highlighted was the need for increased laboratory inputs and consumables in some cases, although access, and supply were disrupted. Simple items such as gloves and masks, RT-PCR tools and inputs among others suddenly became scarce. Shortage of cargo flights resulting in slower delivery of procurements also impacted shelf-life of reagents. Field sampling was significantly affected too due to restricted personnel-movements and transportation. As such, overall laboratory operational costs increased. Lastly, participation in proficiency testing schemes continued although at a slower pace in some laboratories.

In conclusion the Officer advised that countries need to establish and strengthen food safety control systems that are also prepared for and can respond to challenging times. Today it is COVID-19 but who knows what would happen in future. He also encouraged stakeholders to increase visibility of and awareness in the public on the role and importance of sound food safety systems, especially laboratories; how these should be sustainable and the need for continuous data/information gathering or generation on food safety and quality in a country.

The webinar came up with a number of conclusions (and recommendations), one of which was that the COVID-19 pandemic has impacted the supply chain of raw materials and the performance of the food and beverage processing industry and resulting in decreased food availability (quantity, safety/quality and continuity) in some countries. It was also noted that there is a tendency for some food-producing countries to save their food stocks, limiting exports to an extent.



International Webinar: World Food Day 2020.

### Sixth International Forum on Food Authenticity Technology and Industry Development, Beijing, China, 1–2 November 2020

#### Simon Kelly

The sixth International Forum on Food Authenticity Technology and Industry Development (IFFATID) took place on the 1st and 2nd of November 2020 at the Beijing Friendship Hotel and virtually through teleconferencing facilities. The forum was organized by the Chinese National Centre of International Research on Food Authenticity Technology, in affiliation with the NHC Key Laboratory of Food Safety Risk Assessment, and Sinolight Food Inspection and Certification Company. One hundred and sixty delegates attended in person and over 4800 attended through on-line participation. The forum was designed to address the requirement for the development of new methods and techniques to verify the quality, authenticity, and safety of food. Much of this requirement is driven by the frequency of major food scandals, globalisation of food sales and the increasing length and complexity of international supply chains. It also reflects changing consumer preferences in China and the growing number of addedvalue claims that are attached to food production such as the demand for organic, locally produced, natural, authentic, and Geographically Indicated foods, but also new analytical challenges and new risk factors. Fortunately, this has coincided with significant advances in instrumentation, where nuclear and complementary techniques play a key role. A need also exists to address the issue of collating. managing, and exploiting the large amounts of isotopic, elemental, metabolomic, proteomic and genetic data that is produced by the latest analytical equipment. Although this data is digitized, its rate of production and complexity often exceeds the limitations of traditional data management tools. To address all these different topics the forum programme was divided into the following five sessions:

- Responding to Economic Interests to Drive Fraud, Global Cooperation and China's Leading Strategy Research;
- Food Authenticity Technology Empowers High-Quality Industry Development;
- Food authenticity certification and brand release
- Food integrity: integrating quality, safety and authenticity;
- Analysis on the development of stable isotope and metabolomic research in the field of traceability of agricultural products.

Mr Simon Kelly (FEPL), was invited to give a pre-recorded scientific lecture at the forum in the "Food integrity: integrating quality, safety and authenticity" session on "The use of Stable Isotope and Trace Element (SITE) analysis to verify the origin of foods with Geographical Indication (GI) status". This is of particular importance to Chinese researchers at the moment because on 20 July 2020 the European Council adopted decisions on an agreement between the European Union (EU) and the government of the People's Republic of China on geographical indications (GIs) relating to premium foods. This is the first significant bilateral trade agreement signed between the EU and China that ensures 100 EU agri-foods with Geographical Indications receive protection on the Chinese market. Similarly, 100 Chinese products will be protected in the EU, thereby ensuring mutual respect of foods with strong regional and/or national identities from both agricultural traditions. Four years after its entry into force, the scope of the agreement will expand to cover an additional 175 GI names from both sides. The agreement also includes a mechanism to add more geographical indications thereafter. Unfortunately, the added-value attached to foods with GIs can simultaneously incentivise fraudulent substitution with inferior products or adulteration. This in turn can lead to unintended food safety issues, reputational damage and can contribute to barriers to international trade and ultimately poverty. The Food and Agriculture Organisation of the United Nations (FAO) and the European Bank for Reconstruction and Development (EBRD) have identified "the promotion of linkages between local producers, their local areas and their food products through geographical indications as a recognized pathway to nutritious food systems and sustainable development for rural communities throughout the world". The EU already recognises ten GI foods produced in China through its Protected Geographical Indication (PGI) and Protected Denomination of Origin (PDO) legislation, these include:

- Longjing tea (CN/PDO/0005/0621)
- Jinxiang garlic (CN/PGI/0005/0622)
- Yancheng lobster (CN/PGI/0005/0625)
- Zhenjiang vinegar (CN/PGI/0005/0630).

Consequently, there is a recommendation from the EU that newly registered GIs should contain a reference to a suitable analytical procedure to verify the provenance and/or essential qualities of the product that relate it to its *terroir*.

Mr Kelly presented the theoretical basis underpinning the use of combined stable isotope and trace element profiling in SITE analysis, which arguably offers the best hypothesis driven technique to specifically determine the geographical origin of food, through an understanding of the links between isotopic composition of the food and systematic variation of stable isotopes in the biogeochemical cycles of the environment. He also presented concrete examples of the application of SITE analysis to Protected Denomination of Origin (PDO) Grana Padano and Parmigiano Reggiano Italian cheeses, Basmati rice (which is currently undergoing GI recognition in the EU), British Pork and Slovenian milk products.



Mr Kelly giving an invited lecture at the Sixth International Forum on Food Authenticity Technology and Industry Development, Beijing, China, 1 November 2020.

Mr Kelly then participated in a 30-minute live question and answer session with Dr Carsten Fauhl-Hassek from the German Federal Institute for Risk Assessment. Participants at the forum asked questions related to the Technical Officer's presentation regarding the availability of databases of SITE profiles of authentic foods that would permit authenticity and origin evaluation of suspect counterfeit European products.



Mr Kelly (bottom left) with Dr Carsten Fauhl-Hassek (top left), from the German Federal Institute for Risk Assessment, participating in a live question and answer session at the Sixth International Forum on Food Authenticity Technology and Industry Development, Beijing, China, 1 November 2020.

Mr Kelly explained that in order to apply this technology to questions of food origin, and independent verification of traceability systems, it is necessary to characterise the natural variation in the SITE fingerprint for authentic GI food products over a number of growth/harvest cycles. This information can then be incorporated into a reference library or food authenticity database for comparison of suspected counterfeit or adulterated food products. Although methodology and technology is available, the lack of existing databases and/or the limited interoperability between them is the key stumbling block for enabling methodology to implement SITE fingerprinting for food authenticity testing. He then went on to explain that Member States could invest in developing their own SITE libraries, but that an internationally shared open-access database would be the most effective and efficient solution, in a similar way to the IAEA's Global Network of Isotopes in Precipitation (GNIP) database. In addition, the FEPL is taking steps in this direction through its new Coordinated Research Project (D52042) to establish an open access SITE database in collaboration with the U.S. National Institute of Standards and Technology (NIST). Other questions from the meeting delegates relating to the techniques used for stable isotope analysis and other nuclear complementary techniques were also answered by the Technical Officer.

### Updates from the Red Analitica de Latino America y el Caribe (RALACA)

#### Britt Maestroni

The Red Analitica de Latino America y el Caribe (RALACA) network is active in more than 54 institutions in 21 participating countries in the Latin American and Caribbean (LAC) region. The network's objective is to help and sustain efforts for food safety and quality and enhance environmental sustainability. The work of RALACA is conducted through working groups (committees) that synergistically interact on the technical subjects and help its members meet the relevant guidelines, standards, practices and recommendations for the production and control of safe and quality foods.

Since May 2020, RALACA contributed to the analytical community nine webinars and two virtual training events. The webinars addressed a series of topics that are very relevant in the Latin American and the Caribbean region. To cite a few, caring for bees and honey production in times of pandemics; analytical and diagnostic capacity in Ecuador; practical aspects of the production of reference materials; workflows for the determination of pyrrolizidine alkaloids in native flora and as a contaminant in honey; determination of the degradation efficiency of a pesticide bio-purification system by means of multi-residue analysis; analysis of pesticide residues in medicinal plants and spices; APIStrip: a new tool for sampling environmental pollutants through beehives; metrological traceability and reference materials, and interlaboratory comparison tests: a regional experience for the quality of analytical laboratories. The presentations are available online<sup>11</sup>. With an average participation of 55– 60 members at the webinars, a record 139 participants was logged on the occasion of the two-day training on the future of data sharing, a learning opportunity that the European Food safety Authority (EFSA) kindly offered to the LAC region through RALACA, among others.

Since the legal registration of RALACA as a foundation for enhanced capacity building in the LAC region in May 2020, the network has created two very important new committees, the Data Sharing Committee (DSC) and the Academy.

The DSC incorporates counterparts from 19 countries in the LAC region that will start collecting and managing a common repository of food safety related data under the framework of a regional ARCAL project on Strengthening the Regional Collaboration of Official Laboratories to Address Emerging Challenges for Food Safety (ARCAL CLXV; TCP RLA5080).



Ms Maestroni (FEPL, top left) participating in a virtual meeting of the RALACA Board during the COVID-19 lockdown.

## **Continued Support for Food Safety Networks in Africa and Asia**

#### James Sasanya

The FEP has continued to support networking activities among food safety stakeholders in Africa (African Food Safety Network - AFoSaN) and Asia (Food Safety Asia Network). A number of common activities such as interlaboratory studies, among others were organised. Interlaboratory studies are important components of laboratory quality management. They help in validation of methods, assessing proficiency of laboratories, estimating uncertainty of measurement etc and may include comparison studies or proficiency testing (PT). Thus, interlaboratory comparisons (ILCs) were organized in a number of institutions in Botswana, Kenya, Madagascar, Rwanda, Senegal, Seychelles, South Africa, Sudan, Tunisia, Uganda and Zambia. These are focussing on screening and confirmatory analysis of: Sulfonamides, Quinolones, Tetracyclines, Lincosamides and Macrolides in kidney and related animal products; Chloramphenicol in milk and Synthetic steroids in bovine liver. Additional veterinary drug residues include avermectins and benzimidazoles in liver and kidney. Other areas of work include testing of Aflatoxins in feed and heavy/toxic metals (Lead, Cadmium, Arsenic) in food. Furthermore, 34 institutions in 27 countries participated in proficiency testing involving a wide range of residues, contaminants and matrices. Plans are underway to share experiences from the PTs through a technical meeting.

<sup>&</sup>lt;sup>11</sup> <u>http://www.red-ralaca.net/2-uncategorised/482-webinar-2020</u>

These ILCs and PTs are critical to helping the network members meet their goal of ensuring 10 laboratories are accredited in the next two years.

Networking laboratories in Burundi, DR Congo, Ivory Coast, Mauritania, Niger and Rwanda were provided common video-material and analytical methods to facilitate analysis of veterinary drug and pesticide residues as well as mycotoxins in a range of food products. This provides an opportunity for future interlaboratory studies as well as exchange of method protocols and staff.

Three webinars were organized on minor use/crops; mycotoxins and laboratory standards. Details of the minor use are presented in a related article in this newsletter. A related webinar on mycotoxins was held on 11 November 2020 and attended by over 85 participants. It was also the maiden webinar of the Mycotoxicology Society of Nigeria.

Through these two networks (Africa and Asia) in collaboration with partners in Latin America and the Caribbean, several standard operating procedures for analysis of veterinary drug and pesticide residues, mycotoxins and selected toxic metals in food, feed and water have been compiled into a booklet. These will be shared among the network laboratory members and other interested laboratories elsewhere. Furthermore, the Asian group through Indonesia organized an international webinar to commemorate the world day as detailed in a related article in this newsletter.



Preparation for interlaboratory comparision analyses.

## **Coordinated Research Projects**

CRP Reference Number	Ongoing CRPs	Project Officer
D52039	Development and Strengthening of Radio-Analytical and Complimentary Techniques to Control Residues of Veterinary Drugs and Related Chemicals in Aquaculture Products	J.J. Sasanya
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
D61024	Development of Electron Beam and X ray Applications for Food Irradiation (DEXAFI)	C.M. Blackburn
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

An Update on a New Coordinated Research Project (CRP) to Support Work on Maximum Residue Limits (MRLs) for Veterinary Drugs and Related Pesticide (Dual Use) Compounds

James Sasanya

A five-year CRP, D52043: "Depletion of Veterinary Pharmaceuticals and Related Substances and Radiometric Analysis of their Residues in Food" has been launched. This aims at helping Member States generate scientific datasets that will facilitate the setting of food safety standards such as missing MRLs for veterinary drugs and related substances e.g. pesticides of animal and plant use (dual use). This will contribute to promoting trade and safeguarding consumers. The project immediately benefits Codex Member Countries especially through the Codex Committee on Residues of Veterinary Drugs in Food (CCRVDF) and to the extent the compounds concern both animal and plant health, also to the Codex Committee on Pesticide Residues (CCPR) as well as the Joint FAO/WHO Expert Committee on Food Additives (JECFA) for the safety risk assessment of veterinary drugs and where appropriate, the Joint FAO/WHO Expert Meeting on Pesticide Residues (JMPR) for the risk assessment of pesticides or compounds for dual use.

The project arises from needs expressed by several Member States during the 23rd and 24th sessions of CCRVDF. The CCRVDF23 (2016) agreed to establish an electronic working group co-hosted by Costa Rica and the United States of America, to: consider the complete results of a global survey in order to identify priority veterinary drugs for which MRLs are needed for protect public health and to facilitate international trade; and to identify information gaps for a successful and comprehensive assessment by JECFA. Thus, CCRVDF24 (2018) presented a database on countries' needs for MRLs indicating the compounds for which MRLs are needed and the data gaps to allow their safety assessment by JECFA. CCRVDF called upon Codex Member States and observer organizations to provide the missing data identified in the database as/when available so that the compound / commodity combination(s) listed in the database can be scheduled for evaluation by JECFA in order to provide the scientific basis for the establishment of MRL for consideration by CCRVDF.

So far 16 research institutions from: Bangladesh, Brazil (x3), Burkina Faso, Canada, Chile, China, Republic of Korea, Morocco, Pakistan, Sudan, Uganda, Uruguay and USA (x2), are now taking part in this CRP although several others have expressed interest. With additional funding, the project will encourage more participation with wider geographical representation. Particular attention will also be placed on involvement of Small Island Developing States (SIDS) in order to boost their contribution to Codex work.

#### The CRP's specific objectives include: To

- Conduct animal studies using radio-labeled veterinary pharmaceuticals;
- Develop/validate fit-for-purpose analytical methods;
- Conduct radiometric (& complimentary) analysis of residues;
- Report, publish & disseminate findings; technology transfer;
- Information dissemination and knowledge enhancement on standards/MRL setting and implementation of Codex Standards and Guidelines on residues of veterinary drugs and related pesticides to protect animal, plant and human health and contribute to promoting the One Health Approach. This is critical in view of the global importance of antimicrobial resistance and the role in development of Codex food safety standards.

#### The expected results include (but not limited to):

- Radio-label animal tissue data generated: Residue depletion, disposition & distribution on selected veterinary drugs & pharmacologically active substances in edible tissues;
- Safe/regulatory levels set/established: targeted drug residues safety in various foods;
- Analytical material: Labelled drugs, incurred tissues to: facilitate future research, radio-validation of analytical methods & capacity building activities;
- Reports & research capabilities: Publications, TECDOC, manuals for: researchers, regulatory institutions, risk assessors e.g. JECFA, JMPR & risk managers (Member States, CODEX/CCRVDF and CCPR) to set national & international food safety standards residues of veterinary drugs and targeted pesticides as appropriate;
- Robust risk assessment for residues of veterinary drugs and to the extent such veterinary drugs are also used as pesticides, enabling sound, evidence-based risk management decisions for consumer protection; facilitate national/international standards setting;
- Reduced over-dependence of developing countries on external facilities/institutions, laboratories, to conduct radio-labeled studies for risk assessment data;
- Enhanced capabilities of Member States (particularly developing countries) to meaningfully contribute to standards-setting process (national or international);

- International network of researchers involving in supporting Member States (particularly developing countries) generate data required for MRL setting for veterinary drugs and targeted pesticides as appropriate;
- Validated analytical methods (including radiovalidation). Validated methods for enforcement of MRLs will greatly contribute to safe production of food and facilitate trade as has been a concern raised by Codex Member States in both CCRVDF and CCPR;
- Offshoots: Residue depletion studies will help obtain information about habitual high-level consumptions in some populations e.g. lung tissue (or offal in general). MRLs in offal is a new concern to Member States as shown in the latest discussions in CCRVDF and CCPR as offal are highly consumed in certain countries/regions and their potential for.

The expected outcome is: Enhanced technical (research) capabilities of Member States undertake to: depletion/disposition studies for veterinary drugs and related substances used in food production; generate scientific/residue data and facilitate setting of national or international safe residue levels in foodstuff and feed: facilitate risk assessment and public health programmes and promote trade. This is relevant to Sustainable Development Goals (SDGs) 2 and 3.

Research work will include a wide range of veterinary drug and related substances and animal species such as (but not limited to) goats, cattle, poultry, horse, donkey, sheep, fish and bees etc.

Selected topics so far identified include:

- Depletion of Veterinary Pharmaceuticals and Radiometric Analysis of their Residues in Aquaculture;
- Depletion of Veterinary Drugs Using Radiolabelled Compounds and Determination of Pharmaceuticals in Water, Sediments and Native Fish;
- Assessing Beta-Lactam Residues in Broiler Chickens: Full Evaluation of Metabolism and Depletion of Amoxicillin and Ampicillin in Edible and Non-Edible Tissues;
- Depletion kinetics and residues of Oxytetracycline, Ampicillin, Diminazene and Ivermectin in goats' meat, milk, liver, kidney, skin and offal;
- Antiparasitic radio-labelled depletion studies for MRL establishment in food from animal origin;
- Radiotracer studies to determine pharmacokinetics behaviour of veterinary drugs in food-producing animals;

- Depletion of Veterinary Pharmaceuticals (Flavophospholipol, Enramycin and Diaveridine) and Radiometric Analysis of their Residues in poultry;
- Disposition and Residue Depletion of Diaveridine in Pig and Broiler Based on Radiometric Analysis;
- Depletion study of Amoxicillin, Ciprofloxacin and Ivermectin residues in edible tissues of Sonali Chicken using liquid chromatography coupled with tandem mass spectrometry
- Depletion and Development of Methodologies for Evaluation of Residues of Amoxicillin, Ampicillin, Imidocarb
- Depletion of Targeted Radiolabeled Antitrypanocide and Anti-Piroplasm Drugs used in Cattle and Camels
- Investigating Conjugation of Veterinary Pharmaceuticals and Depletion of Radiolabelled Residues in Food-Animal Matrices

- Radiolabelled studies of veterinary pharmaceuticals and related substances commonly used in livestock production in Burkina Faso and radiometric analysis of associated residues.
- Depletion of Veterinary Pharmaceuticals and Radiometric Analysis of their Residues in Animal Matrices"/Estimating antibiotic (tulathromycin, florfenicol) depletion in goat milk samples.
- Validation of multi-class analytical methods for the determination of veterinary drug residues in minor species

The first research coordination meeting planned for this year first in May and then November 2020 couldn't take place and tentatively is rescheduled for March 2021.

The project is seeking partnerships to support the various research topics and offset costs of radiolabelled material and use of animal experimental facilities. There is a huge demand among the Member States for such projects and with additional funding, more institutions can be involved. Therefore, any public-private partnerships are most welcome.

## **Technical Cooperation Projects**

Country/Region	Project No.	Title	<b>Technical Officer</b>
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. 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	J. J.Sasanya
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 Capabilities to Ensure Food Authenticity	S.D. Kelly
Ecuador	ECU5030	Reducing Post-Harvest Losses of Native Potatoes and other Fresh Foods by Irradiation	C. M. Blackburn
Eritrea	ERI5012	Developing Analytical Capabilities for Food Safety	J. J. Sasanya

Country/Region	Project No.	Title	<b>Technical Officer</b>
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	M. Groening S. Kelly
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 Crpps 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
Mauritania	MAU5008	Strengthening Laboratory Capacity to Analyse and Monitor Residues and Contaminants in Foods	J. J. Sasanya

Country/Region	Project No.	Title	<b>Technical Officer</b>
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 A. Cannavan
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. 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. 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
Sri Lanka	SRL5048	Strengthening National Capability for Food and Feed Safety	A. Cannavan A.Mihailova

Country/Region	Project No.	Title	<b>Technical Officer</b>
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
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
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. Kelly
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

## Promoting Food Irradiation by Electron Beam and X-Ray Technology (RAS5087), 3–4 September 2020

#### Carl Blackburn

The first meeting of this Asia and Pacific technical cooperation project on Promoting Food Irradiation by Electron Beam and X-Ray Technology to Enhance Food Safety, Security and Trade (RAS0587) could not be held in person in early 2020 due to COVID-19 related issues. A virtual meeting therefore took place from 3–4 September 2020 with a preparatory meeting held on 1 September.

The meeting aimed to provide detailed information on RAS5087 so that all participants could have a common understanding of the project objectives and finalize their national workplans. It also provided the opportunity to review current national capacities for food irradiation. This information is the baseline with which to measure the effectives of the project. The proposed regional and national workplans for 2020–2021 were discussed and reviewed.

There were 24 participants from 18 countries (Australia; Bangladesh; Fiji; India; Indonesia; Japan; Republic of Korea; Malaysia; Mongolia; Myanmar; Nepal; New Zealand; Pakistan; Philippines; Singapore; Sri Lanka; Thailand, and; Viet Nam). China has indicated its intention to participate in project RAS5087, unfortunately technical issues prevented their National Project Coordinator (NPC) from joining this virtual meeting. Details of the status of food irradiation in China have been provided in this report based on information received at recent international meetings and reviews.

It was concluded that approximately 1.2 million tonnes of food are irradiated in this region each year (Table 1).

Meeting participants also provided details of eighty-two irradiation facilities, with fifty irradiating food (41 gamma facilities, three electron beam and six X-ray facilities), there are also an additional 14 electron beam/X-ray research facilities. These figures do not include facilities in China.

Thirteen countries (those listed in Table 1 plus China) have facilities that are irradiating food commercially, but the volumes treated annually range from just a few tonnes to over 1 million tonnes. Five participating countries do not irradiate food commercially though one conducts research with a gamma facility (Myanmar) and another further country does not irradiate food domestically but does accept irradiated food imports for retail (New Zealand).

Project activities and outputs were agreed for the duration of the project. It was agreed that it would be important to engage with the food industry and equipment suppliers at both regional and national level. It was also envisaged that COVID-19 restrictions would remain a problem for future meetings and events, therefore some events might be changed to virtual events and that timing would have to remain flexible. Each country provided national workplans in draft form and these were revised and finalized by the end of the meeting. Participating countries requested the production of two technical documents for next year: a report on the comparison of the different radiation sources used in food irradiation and a technical document on the need to harmonize regional requirements, especially the maximum X-ray energy allowed for X-ray irradiation of food.

All the participants were thanked for their hard work which ensured that the meeting was a success, even though it spanned different time zones. Mr Roberts of New Zealand and Mr Hoang of Viet Nam were also thanked for working with the secretariat in advance of the meeting and helping to organize and implement this successful virtual event.

Country	Amount	Main food treated
	(tonnes/year)	
AUL	6,000	Fresh fruit and vegetables
BGD	145	Spices, pet foods
IND	12,500	Spices and condiments, grains and pulses, mangoes, dry fruit and pet
	(average of last 3	food
	years)	
INS	650	Herbs, spices, general foods
JPN	3,000	Potatoes
ROK	250	Dry vegetables, ginseng products, grains, seasonings, yeast products
MAL	1,100	Herbs, spices, processed foods
PAK	2,000	Herbs, spices, mangoes, fresh fruits and vegetables, pet food
PHI	470	Herbs, spices
SRL	1	Herbs, spices
THA	1,500	Herbs, spices, mangosteen, processed foods
VIE	120,000	Fresh and frozen seafood, herbs, spices, grain, fresh fruits, pet food
Sub-total	147,500*	_

TABLE 1. AMOUNTS OF FOOD IRRADIATED EACH YEAR IN THE ASIA AND PACIFIC REGION

\* In addition, China is estimated to produce more than one million tonnes of irradiated food each year.

## Nuclear and Isotopic Laboratory Capabilities Enhanced in Bangladesh to Help Address Food Safety Concerns

#### James Sasanya

A state-of-the-art food safety laboratory established at the Institute of Food and Radiation Biology (IFRB), Atomic Energy Research Establishment (AERE), Ganakbari, Savar, is now responding to food safety needs in Bangladesh thanks to IAEA's support in partnership with the Food and Agriculture Organization of the United Nations (FAO).

Starting almost from scratch, this laboratory is now fully functional and serving the nation by testing a wide range of food hazards. The laboratory now uses 15 rapid screening and six confirmatory methods to determine antimicrobial residues in foods of animal origin without outsourcing these tests to other countries. Other hazards such as mycotoxins, pesticide residues and toxic metals can also be analyzed. Continuous operation of the laboratory will significantly reduce the outsourcing of analytical tests related to food safety.

The laboratory demonstrates reliability and competence by participating (successfully) in international proficiency testing schemes for a range of drug residues in egg, meat and fish by using radio receptor assay screening and isotopic confirmatory tools. Several food samples (above 2000) including eggs, milk, chicken and shrimp collected throughout the country can now be tested to generate residue data. Such data can enable regulatory institutions such as the Bangladesh Food Safety Authority (BFSA) to take necessary actions to protect public health and to improve the food safety control system in the country. The laboratory has started providing analytical services to non-technical stakeholders and is in the process of accreditation by Bangladesh Accreditation Board, which will enhance enduser confidence. The laboratory is also in the process of designation by the BFSA as a reference laboratory for food safety.



Analysis of food chemical hazards at IFRB.

The Ministry of Science and Technology has entrusted the laboratory with rapid screening of veterinary drug residues in milk in the country. Through the IAEA technical cooperation programme, 10 analysts have been trained in the testing of a range of food hazards. A new 600 square meter modern food safety laboratory has also been constructed recently. Furthermore, sustainability is now assured given government commitment to building on the foundation set through cooperation with IAEA in partnership with FAO. Government recently invested in procurement of three high-tech instrumentation worth over 700,000 Euros. The laboratory is now fully capable to provide analytical support to regulatory authorities.



Confirmatory isotopic analysis food chemical hazards.

## Agency Support Boosts Bahrain's Efforts to Safeguard Consumers from Potential Hazards in Food

#### James Sasanya

The Kingdom of Bahrain imports most food stuff and to better protect consumers, reliable testing of potential hazards such as mycotoxins, residues of pesticides and related chemicals, toxic metals and radionuclides that can pose human health concerns and should be analyzed. Thus, after receiving technical assistance from the IAEA in partnership with the Food and Agriculture Organization of the United Nations (FAO), the Public Health Laboratory, Ministry of Health can now more effectively conduct regular testing of such food and environmental hazards. For instance, there has been a 10-fold increase in samples analyzed for up to 160 pesticide residues a day, in a wide range of matrices including fruits, vegetables and seeds among others. Stableisotope labelled compounds are used in such methods and help improve precision and therefore performance of the techniques.

Following cooperation with the IAEA, the Public Health Laboratory now has adequate capability to monitor for radionuclides in foods including fish and set up plans to collaborate with the Ministry of Agriculture and Marine Resources to test fish and water samples.



Stakeholder awareness on food safety issues including sampling.

Another area that has benefited from the cooperation with the IAEA is the testing of toxic metals. Before the IAEA's support, the laboratory provided limited services and a very narrow scope (up to five) of toxic metal-analysis. This is now changing with new capabilities including availability of an ICP-MS. More toxic metals of concern (over ten) can be tested or monitored in both local and imported products. More samples can now be analysed per day at lower detection limits.

#### Towards export markets and helping regional needs

With the capabilities established/strengthened, foods such as spices, one of Bahrain's most important export commodities, are now also being tested for pesticides and mycotoxins among other hazards. Local production of spices as of 2018 to date exceeds 431 tons/year and the number of chemical tests is above 5000 tests annually. The sound laboratory capabilities enhanced in cooperation with the Kingdom of Bahrain can greatly benefit the region, especially the Gulf Cooperation Council Member Countries to whom food safety is a high priority.



Sampling for food safety testing in Bahrain.

#### <sup>12</sup> https://opendatacharter.net

## Updates on the IAEA TCP RLA5080, 'Strengthening the Regional Collaboration of Official Laboratories to Address Emerging Challenges for Food Safety' (ARCAL CLXV)

#### Britt Maestroni

The IAEA technical cooperation project RLA/5/080 "Strengthening the Regional Collaboration of Official Laboratories to Address Emerging Challenges for Food Safety (ARCAL CLXV)" officially started in January 2020. The goals of this regional project are to contribute to improving food safety through risk-based policies to ensure public health and environmental protection; promote cooperation between reference laboratories in the region; harmonize the risk monitoring and evaluation methodology; facilitate the generation of analytical data through collaborations between reference laboratories and establish a data network as part of the regional infrastructure in food safety. The expected outcome of the project is the availability in the region of consolidated data, in the form of a database, that represents the starting point for evidencebased risk assessment and subsequent decision-making by the competent authorities.

Despite a difficult first year marked by the COVID-19 pandemic, the project managed to create functional synergies among the counterparts, brought together different partners in the region and, most importantly, paved the way to create sustainability in the region through the creation of a RALACA Data Sharing Committee (RALACA-DSC), the members of which will be officially appointed by the participating countries most likely by the end of 2020. A regional meeting involving 19 counterparts will prepare the framework within which the RALACA-DSC will operate, in the absence of harmonized regional policies and a legal framework for data sharing and transparency laws. The principles of data sharing can be found at the Open Data Charter  $(ODC)^{12}$ , which is a collaboration between over 100 governments and organisations working to improve data availability and sharing based on a shared set of principles. The ODC contains six principles that are: data open by default; timely and comprehensive; accessible and useable; comparable and interoperable; for improved governance and citizen engagement; and for inclusive development and innovation. Those principles were brought to the RLA/5/080 project by Dr Jane Richardson from the European Food Safety Authority (EFSA), during a virtual event on the future of data sharing, organized in the framework of the project on 27 and 29 October 2020. Together with other colleagues from EFSA, Dr Richardson discussed the benefits of data sharing, the modalities implemented at the EU level and the

challenges and lessons learned from the European process of establishing a data portal for evidence-based risk assessments. In the current project, data will not be open to the public, but will be available in an aggregated manner to comply with privacy and data protection requirements set by the participating counterparts and at the same time to be of use for initial data exploration and investigation exercises. On the national level, project counterparts liaise with national stakeholders and decision-makers to establish the collaborative networks required for data sharing activities and to pave the way to long-term collaborations among regional partners and ultimately ensure food safety and quality in the region.

The project provides a platform to discuss with regional and interregional partners the required institutional framework and processes related to the collection, integration and interpretation of food safety and quality data/information available at the country-level. Participating counterpart institutions will also benefit from the procurement of digital services to facilitate the management of relevant data and risk assessment.



The RLA5080 project flyer used by the counterparts for promoting the project with national stakeholders.

## Developments at the Food and Environmental Protection Laboratory

### Transition of the Food and Environmental Protection Laboratory to the Yukiya Amano Laboratories

#### Andrew Cannavan

The construction and fit-out of the new Yukiya Amano Laboratories (YAL) building, which will house the Food and Environmental Protection Laboratory (FEPL), The Soil and Water Management and Crop Nutrition Laboratory and the Animal Production and Health Laboratory was largely completed in the latter half of 2020. As well as the laboratories, the building offers a meeting room/lecture theatre suite, suitable for both internal meetings and external training events.

Many of the operations of all three laboratories have been transferred to the new building. Unfortunately, the COVID-19 pandemic and the consequent ongoing travel and working restrictions have had knock-on effects, causing delays to the completion of some of the final installations of infrastructure and systems in the new building. At the time of writing, therefore, some of FEPL's main instrumentation has not yet been relocated to YAL. This includes the isotope ratio mass spectrometric systems and the high-resolution quadrupoletime of flight mass spectrometer, meaning that research on food authenticity and geographical origin verification requiring stable isotope measurements or high-resolution mass spectrometric metabolomics is currently on hold, or being performed to a limited degree in the old laboratory. It is expected that all systems will be commissioned in YAL and active in early 2021.

The new FEPL is spacious and designed for purpose and offers much greater opportunities for both research and technology transfer through training activities. Various research threads are already active in the new laboratory. Research and method development is ongoing using benchtop and portable instruments (FTIR, multispectral imaging, SCIO hand-held IR molecular sensor) for authentication and detection of adulteration of various commodities including coffee and rice. A study is under way using liquid and gas chromatography coupled to tandem mass spectrometry (LCand GC-MS) to investigate the pesticide metabolites that have toxicological relevance in lyophilized biomixtures from biopurification systems (biobeds) used to degrade pesticide-containing waste of agricultural origin, such as wastewaters. Method validation has recently been completed for the LC-MS amenable pesticide residues in orange juice and validation is under way for the GC-MS amenable pesticides. A new benchtop nuclear magnetic resonance (NMR) instrument is being applied to develop methods for the differentiation of mandarin orange varieties,

and Robusta and Arabica coffee beans. FEPL is also participating in an interlaboratory study on testing the authenticity of olive oils using GC-ion mobility spectroscopy (GC-IMS).

We look forward to having the FEPL fully functional in the very near future and to the opportunity to better serve our Member States with the much-improved facilities.



The spectroscopy lab in the new FEPL.



The LCMS lab in the new FEPL.



The wet chemistry lab in the new FEPL.

### Development of Analytical Methods to Support Research on Degradation of Pesticides in Food Commodities and the Environment

Britt Maestroni and Sofia Rezende

The FEPL, through the work of the RALACA network, continues to support research studies on agricultural pollution sources, as it is important to ensure the safety of food products and a sustainable agricultural environment. To control field pests, conventional agricultural production makes use of pesticides, which may lead to pollution of the environment not only through application of the pesticides to the crop, but also due to accidental spillages that may arise from the different manipulations performed before the application of pesticides in the field, such as preparation of formulations, rinsing of the formulation tanks, the triple washes of the pesticide plastic containers, and after application, from the cleaning of the sprayers and other apparatus. These are all significant sources of contamination at agricultural sites and might affect the quality and safety of food produce as well as negatively affect the environment. To mitigate this contamination, in the 1990s a Swedish research team developed an eco-friendly, low cost biotechnological tool, known as biopurification systems, or simply biobeds, for treating point-source pesticidecontaining waste of agricultural origin, such as wastewaters. These are simple but effective bioreactors made of easy-toget and cheap farm materials such as peat, straw and soil. The degrading capacity of these systems relies on the biological matrix, called biomixture, which can remove pesticides faster than possible in other environments (e.g., soil).

Biobeds are known to be versatile and scalable bioreactors that can be adjusted to the producer needs. As the composition of the biomixtures depends on the availability of local agricultural by-products of no commercial value, biomixture design should be adapted to such regional availability. The efficacy of biomixtures should be tested in each situation, since other local variables such as weather conditions and temperature will define the microbial diversity and activity of the biomixture and therefore its degrading potential.

Under the framework of IAEA technical cooperation projects, the Research Center of Environmental Pollution (CICA) in Costa Rica has adapted different biomixtures for use in biobed systems, implemented in small farms in Costa Rica for the disposal and treatment of pesticide-containing wastewaters. In CICA nuclear techniques are used to evaluate the degradation of pesticides, using radiotracers and measuring the mineralization rate and the formation of CO<sub>2</sub> marked with carbon-14. CICA has published this method in the book "Analytical methods for agricultural contaminants" (Academic Press - Elsevier, edited by FEPL).

In Uruguay biobeds have been adapted to work at very small farm scale up to the degradation of wastes from postharvest citrus and horticultural production.

In this context, and in the framework of a PhD consultancy from Uruguay, the FEPL is working towards the development of analytical methods using liquid and gas chromatography coupled to tandem mass spectrometry to study the pesticide metabolites that have toxicological relevance in lyophilized biomixtures from biobed systems installed in Uruguay. Once established, the methods could also be applied to study the degradation of pesticides in orange samples after spraying pesticide formulations on orange trees in Mexican fields. This will complement a study on the establishment of analytical and isotopic methods to distinguish between conventional and organic production of fruit juices.

It is the hope of FEPL to develop collaborations with additional partners to promote the use of advanced chromatographic techniques for studying metabolite profiles in a range of different commodities that have socioeconomic importance in Member States. The work through the RALACA network has so far helped to establish research collaborations in Uruguay, Costa Rica and Mexico.



*PhD consultant Sofia Rezende working on method development for pesticide metabolites in FEPL.* 



A biobed in Colonia Valdense, south western Uruguay.

## Nuclear Magnetic Resonance (NMR) Spectroscopy—A New Technique for Food Safety and Authenticity Screening at FEPL

Alina Mihailova, Ignacio Migues, Beatrix Liebisch and Simon Kelly

A new Spinsolve 60 benchtop nuclear magnetic resonance (NMR) spectroscopy system was commissioned in the Food and Environmental Protection Laboratory (FEPL) in September 2020.

NMR spectroscopy is a non-destructive analytical technique which provides detailed information about the molecular structure of a sample, enables quantification of molecules even in complex mixtures and allows the direct observation of chemical reactions. NMR is often used as an analytical tool for untargeted metabolomic studies. NMR spectra are unique, analytically tractable and often highly predictable for small molecules.



Spinsolve 60 NMR spectrometer at FEPL.

NMR is based on the principle that nuclei of atoms have magnetic properties that can be utilized to vield chemical information. All nuclei are electrically charged. In many atoms (e. g. 12C), the nucleus has no overall spin. However, in some atoms (e. g. 1H, 13C, 15N, 19F, 31P) the nucleus does possess spin. Any nucleus with an odd atomic number or an odd atomic mass has nuclear spin and can be analysed by NMR equipment. Spinning charged nuclei generate a magnetic field and possess a magnetic moment. When placed in a magnetic field, nuclei with spin align themselves according to their energy states. When an external timedependent magnetic field is applied, transfer of energy is possible from the base energy to higher energy levels. This transfer of energy occurs at a wavelength that corresponds to a radio frequency. When the spin comes back to its base level, energy is emitted at the same frequency. By measuring and processing the signal from this transfer, an NMR spectrum can be obtained. The most common types of NMR are proton (1H) and carbon (13C) NMR spectroscopy; however, the technique is applicable to any sample that contains nuclei possessing spin.

The resolution of an NMR spectrometer depends directly on the strength of the magnetic field used. High-resolution NMR spectrometers (300–1200 MHz), which have a large superconducting magnet, can enable structural elucidation. However, the main drawback of these instruments is their very high cost in terms of initial investment, consumables (liquid helium), maintenance (hardware) and operation (skilled personnel). This led to the introduction of benchtop NMR spectrometers with permanent magnets (42–100 MHz). They combine a small footprint, a  $5-20 \times 1000$  MHz). They combine a small footprint, a  $5-20 \times 1000$  mHz). They combine a small footprint, a  $5-20 \times 1000$  mHz). They combine a small footprint, a  $5-20 \times 1000$  mHz) and the set of each state of eac



Remote training on the use of the Spinsolve 60 benchtop NMR system at FEPL.

NMR spectroscopy has been used for a wide range of applications in the areas of chemistry, biology, medicine, pharma, food and feed, forensics and process control. In the area of food safety and authenticity, NMR has been applied for the quality control, authentication and traceability of various commodities, e.g. fruit juices, honey, coffee, spices, edible oils, wine, meat, fish etc. Some examples of the food safety and authenticity applications using benchtop NMR systems include the detection of cheap refined edible oils in extra virgin olive oil or cold pressed rapeseed oil, pork or horse meat in beef, cheaper Robusta coffee in Arabica coffee, and synthetic chemical compounds or cheap edible oils in Patchouli and other essential oils.

Benchtop NMR spectroscopy offers an untargeted multianalyte screening capability as well as low operational costs. This makes the technique suitable for authenticity screening, complementing the other analytical approaches that are being developed and used at FEPL under CRP D52042 "Implementation of Nuclear Techniques for Authentication of Foods with High-Value Labelling Claims" and transferred to the Member State laboratories.

FEPL is currently in the process of using the Spinsolve 60 NMR system to optimize methods for the differentiation of mandarin varieties, and Robusta and Arabica coffee beans.

Future applications may include the authenticity screening of edible oils, meat and spices as well as monitoring chemical reactions to determine optimal reaction parameters. Some of this research work will be presented in future editions of the FEP newsletter. Visiting scientists, fellows and interns will have the opportunity to be trained in the operation of the Spinsolve benchtop NMR system, and to learn the analytical methods that can be applied, thereby supporting Member State training efforts and raising awareness of this highly accessible and novel rapid screening technology.



An example of an OPLS-DA model of Robusta and Arabica coffee generated using NMR spectral data obtained at FEPL.

## Method Validation for Pesticide Residues in Orange Juice Using LC-MS/MS

#### Sofia Rezende and Britt Maestroni

Based on Member State needs, the FEPL initiated a study on the optimization and validation of a multiresidue method for the detection of pesticide residues in orange juice using chromatography coupled liquid to tandem mass spectrometry (LC-MS/MS). During the second part of 2020 the method was validated according to Codex guidelines at the new Yukiya Amano Laboratories (YAL) premises of the FEPL. The Swedish ethyl acetate sample preparation method (SweET method) was adopted (and validated) for its simplicity, speed, and the selectivity afforded by the low solubility of sugars and proteins in ethyl acetate (EtOAc). The validation was designed to ensure that the method is fit for purpose; that the analytes can be accurately quantified at trace levels (µg/kg) and that unequivocal evidence can be generated to confirm the identity of any pesticide residue detected.

Ethyl acetate extracts were directly injected into the LC system and chromatography was effected using an optimized gradient elution profile. The LC-MS/MS was operated in scheduled multiple reaction monitoring (MRM) to achieve the required sensitivity and selectivity. The experimental design included the analysis of reagent blanks, matrix blanks, fortified samples at three different concentration levels (0.010, 0.020 and 0.050 mg/kg) each in five replicate samples, with repeated analysis on three different days to establish the within laboratory reproducibility.

The key method performance parameters investigated were specificity. linearity. trueness, within laboratory repeatability and reproducibility. Others were limit of detection, limit of quantitation, matrix effects and qualitative criteria for confirmation. From an initial proposed scope of 55 pesticides, only 52 analytes could be validated at all three validation levels. The method was validated for these 52 pesticides with acceptable performance criteria according to the Codex Alimentarius Guidelines on Good Laboratory Practice in Pesticide Residue Analysis (Codex guideline CAC/GL 40-1993. Rev.1-2003). Recoveries for the 52 pesticides validated ranged from 60% to 110%, and the relative standard deviations were lower than 20% for all the pesticides. Figure 1 shows the analytes included in the scope of the method and the average recoveries obtained at the three fortification levels. Figure 2 shows the elution profile of the method in a sample fortified at each of the validation concentrations; in a matrix matched calibrator at 0.05 mg/kg; and in a blank sample. The blank sample contains some interferences. One spurious peak occurs at a retention time of about 2 mins and does not interfere with the analytes. However, the large interference present at 6 minutes, as observed in figure 2 (E), is responsible for the signal saturation of a pesticide that elutes at this retention time (malathion). In addition, fenthion and phosmet did not comply with the qualitative criteria for ion ratios and therefore could not be validated. Fenthion had a very high value for matrix effects (about 300%) and could not be precisely quantified even using matrix matched calibration strategies.

Due to delays caused by the move of the FEPL laboratory to the new YAL premises, validation of the method using gas chromatography coupled to tandem mass spectrometry (GC-MS/MS) is still ongoing



FIG. 1. The average recoveries obtained at three fortification levels (0.01, 0.02, 0.05 mg/kg) for the 52 pesticides included in the scope of the method by LC-MS/MS.



FIG. 2. The elution pattern of the method in a fortified sample at (A), 0.01 mg/kg, (B), 0.02 mg/kg and (C), 0.05 mg/kg, (D) in a matrix matched calibrator at 0.05 mg/kg, and (E) a blank sample.

## Determination of C4-Plant Sugar Addition to Pineapple Juice by GC-CrAg-IRMS Measurement of δ2H of Carbon-bound-non Exchangeable (CBNE) Hydrogen in Sucrose

#### Aiman Abrahim, Simon Kelly and Andrew Cannavan

Differentiating between sugars produced by crassulacean acid metabolism (CAM) plants, in particular pineapple, and those derived from  $C_4$  metabolism used to produce cheap adulterant sugar syrups (e.g. cane, maize) is challenging. This is because the overall molecular stable carbon isotope ratios of the sugars and site-specific deuterium/hydrogen

ratios of the methyl group of ethanol  $(D/H)_1$ , measured by deuterium site-specific natural isotope fractionation nuclear magnetic resonance (<sup>2</sup>H SNIF-NMR), are very similar. Currently, the only reported successful method for detecting the addition of exogenous C<sub>4</sub> sugars to pineapple juice is <sup>13</sup>C SNIF-NMR analysis of the methyl and methylene sites of ethanol fermented from pineapple juice sugars, with a detection limit of approximately 15% w/w of the total sugars present. However, this method is time consuming and expensive to implement due to the need to ferment the pineapple juice sugars to ethanol and the use of a high resolution, deuterium enabled, NMR.

A novel procedure for the rapid isotope analysis of the carbon-bound nonexchangeable (CBNE) hydrogen in mono

and disaccharides was recently developed in FEPL. The method was targeted at the detection of the undeclared addition of exogenous sugar products in foods and beverages susceptible to economically motivated adulteration. The procedure utilizes a simple one-step reaction to substitute the exchangeable hydroxyl-hydrogens with trifluoroacetate derivatives that are sufficiently volatile to be separated by gas chromatography and measured by an isotope ratio mass spectrometer coupled to the gas-chromatograph. The derivatised sugars are converted into hydrogen gas using a high temperature chromium-silver reactor that retains carbon, oxygen and fluorine whilst releasing hydrogen for stable isotope measurement. This new procedure has advantages over existing methods in terms of ease of use, analysis time and compound-specific information. The method was initially applied in FEPL for the analysis of sugars from fruit juice and honey to demonstrate its feasibility and has now been applied to evaluate its feasibility for detecting C4-sugar addition to pineapple juice.

A crude extract of the sugars present in pineapple juice is isolated by removing pulp by centrifugation and the other major soluble-solid, citric acid, by precipitation of its insoluble calcium salt. The supernatant is lyophilised, and sugars are derivatised with N-methyl-bis the (trifluoroacetamide) (MBTFA). This process removes exchangeable hydroxyl-hydrogen atoms and replaces them with trifluoroacetatate (TFA) groups, which also makes the sugars sufficiently volatile for gas chromatography. The major sugars in pineapple juice (sucrose, glucose and fructose) are separated as their TFA derivatives by gas chromatography and then passed into a capillary furnace containing chromium metal particles and silver wool maintained at 1200 °C. The furnace retains carbon, oxygen and fluorine releasing hydrogen gas for determination of the mass distribution of the isotopologues (<sup>2</sup>H<sup>1</sup>H and <sup>1</sup>H<sup>1</sup>H) by isotope ratio mass spectrometry and calculation of  $\delta^2 H$  with respect to relevant certified reference materials.

Authentic production samples of single strength pineapple juice and pineapple juice concentrate were obtained from the SGF (Schutzgemeinschaft der Fruchtsaft- Industrie e.V, a non-profit industrial association financed by more than 650 fruit juice companies from nearly 60 countries worldwide). Pineapple concentrates were diluted to 12 °Bx (approximately 12 % w/w of sugar) with Millipore water prior to sugar extraction and analysis (Figure 1). Figure 2 shows the 95% prediction intervals calculated from the bulk  $\delta^{13}$ C analysis of lyophilised sugars extracted from pineapple juice and the corresponding  $\delta^2$ H values of the sucrose-TFA derivative. Sucrose was selected because it is generally the highest concentration sugar present in pineapple juice and provides the highest intensity peaks for repeatable isotopic measurement. The prediction intervals may be used to assess the authenticity of retail samples with respect to the addition of exogenous C<sub>4</sub> and C<sub>3</sub> sugars e.g. cane and beet sugar (syrups) respectively. The method has been successfully demonstrated to be effective as a potential tool for detecting economically motivated C<sub>4</sub>-sugar adulteration of pineapple juice.



FIG. 1. Sugar extract from pineapple analysed by GC-CrAg-IRMS as TFA derivative.



FIG. 2. Plot of authentic pineapple juice sucrose-TFA derivative  $\delta^2 H$  versus bulk sugar  $\delta^{13}C$ .

#### FEPL Staff

FEPL is very happy to welcome several new members to our team in the latter half on 2020.

Mr Ignacio Migues Borghini, from the University of the Republic, Uruguay, joined FEPL as a PhD Consultant in September 2020. Ignacio is working on the development and transfer to Member States of analytical methods using bench-top NMR to authenticate food products, under the PUI project "Enhancing Capacity in Member States for Rapid Response to Food Safety Incidents and Emergencies", which is funded by Japan. Initial work using Ignacio's expertise in NMR has focused on the adaptation and transfer of high-field NMR methodology for the authentication of coffee to the newly commissioned bench-top NMR instrument in FEPL.

Ms Beatrix Liebisch commenced a one-year internship in FEPL in September 2020. Beatrix is currently studying for a Master of Food Chemistry and Toxicology degree at the University of Vienna, and is contributing to the FEPL's work on the development and transfer to Member States of analytical methods to detect contaminants and authenticate food products. During her initial period in FEPL Beatrix has been working with Ignacio and FEPL staff on the application of the bench-top NMR for the authentication of high-value coffee, and on spectroscopic methods for authentication of various commodities.

Mr Shuichi Nakaya joined the FEPL team in October 2020 for a two-year position as a cost-free expert under the PUI project "Enhancing Capacity in Member States for Rapid Response to Food Safety Incidents and Emergencies". Shuichi has a Masters' Degree in Life Sciences from the University of Tokyo, Japan, and research and development experience using mass spectrometric analytical instrumentation in the public and private sectors in Japan. Shuichi will work mainly on the development of instrumental methods of analysis for the detection and control of food contaminants, and the transfer of those methods to developing country counterparts through training workshops and courses held under the PUI project.

Ms Florence Maxwell joined the FEPL team as a laboratory technician in November 2020. Florence transferred from the Insect Pest Control Laboratory and will contribute to all aspects of the FEPL research and development. She has quickly integrated with the team and has made a good start, working on the development of screening methods for authenticity testing and detection of adulterants in foods, as well as providing general laboratory support.

## Announcements

### Rescheduling of the FAO/IAEA Training Course on Detection and Control of Organic Contaminants in Food-targeted Testing

#### Britt Maestroni and Andrew Cannavan

The FAO/IAEA 'Training Course on the Detection and Control of Organic Contaminants in Food – Targeted Testing' initially planned to be held at the Joint FAO/IAEA Division's laboratories at Seibersdorf, Austria, 20–30 April 2020 had to be postponed because of travel restrictions and national responses to the COVID-19 pandemic. The training course has now been rescheduled and a new format has been developed.

A virtual component of the training course focusing on the theoretical and background aspects of the detection and control of organic residues and contaminants in food took place from 1–4 December 2020 using the IAEA virtual platform Nucleus. Participants were those previously selected for the training course in April. Candidates who successfully completed the virtual training course are eligible for a one-week follow-up practical training course to be held when travel restrictions are relaxed, hopefully in 2021 (date yet to be finalised) in the new Yukiya Amano Laboratories at Seibersdorf, Austria. The dates of the practical training course will be announced, and candidates informed, as soon as possible.

### International Food Irradiation Symposium, Online 9–11 March 2021

Ion Beam Applications s.a. (IBA), Aerial and Bühler with the support of the Joint FAO/IAEA Joint Programme for Nuclear Techniques in Food and Agriculture and the International Irradiation Association are delighted to announce the organization of an online symposium fully dedicated to food irradiation. The symposium will cover food irradiation, legislation, food quality, dosimetry and technical solutions. Register online<sup>13</sup>.



International Food Irradiation Symposium.

<sup>&</sup>lt;sup>13</sup> <u>https://www.ifis2021.com/</u>

## Publications

### 2020

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Sasanya, J.J. (2019). 51st Session of the Codex Committee on Pesticide Residues of Joint FAO/WHO Food Standards Programme, Macao SAR, P.R. China, 8–13 April 2019. http://www.fao.org/fao-who-codexalimentarius/sh-

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