METROFOOD-RI: a new reality to develop Reference Materials for the agrifood sector

G Zappa[1], L Anorga[2], N Belc[3], I Castanheira[4], O F X Donard[5], L Kourimska[6], A Kukovecz[7], I Iatco[8], A Najdenkoska[9], J Nieminen[10], N Ogrinc[11], H Ozer[12], M Rychlik[13], M Z Tsimidou[14], J Van Loco[15], and C Zoani[1]

[1] Italian National Agency for New Technologies, Energy and Sustainable Economic Development, Dept. for Sustainability - Biotechnologies and AgroIndustry Division (SSPT-BIOAG), C.R. Casaccia –V.Anguillarese 301, 00123 Roma, Italy
[2] CIDETEC, Parque Científico y Tecnológico de Gipuzkoa, Paseo Miramón 196, 20014 Donostia-San Sebastián, Spain
[3] National R&D Institute for Food Bioresources – IBA Bucharest, 5 Baneasa Ancuta, Bucharest 2, Romania
[4] Department of Food and Nutrition, National Health Institute Doutor Ricardo Jorge Av. Padre Cruz 1649-016, Lisbon, Portugal
[5] Université de Pau et des Pays de l'Adour, 64012 PAU cedex, France
[6] Department of Microbiology, Nutrition and Dietetics, Faculty of Agrobiology, Food and Natural Resources, Czech University of Life Sciences Prague, Kamycka 129, 165 00 Praha 6 – Suchdol, Czech Republic
[7] Dept. of Applied and Environmental Chemistry, University of Szeged, Rerrich Bela ter 1., H-6720 Szeged, Hungary
[8] DAS Foundation - 1/7 Str. Studentilor, Chisinau MD-2045, MOLDOVA
[9] IJZRM, Institute of Public Health, 50 Divisia 6, Skopje, 1000, FYROM
[10] Finnish Food Safety Authority (Evira), Chemistry Researcher Unit, Mustialankatu 3, FI-00790, Helsinki, Finland
[11] Dept. of Environmental Sciences, Jožef Stefan Institute, Jamova 39,1000 Ljubljana, Slovenia
[12] TÜBİTAK MAM, Food Institute - Barış Mh. Dr. Zeki Acar Cd. No:1,PK:21; Gebze,Kocaeli, Turkey
[13] Chair of Analytical Food Chemistry, Technical University of Munich, Maximus-von-Imhof-Forum 2, D-85354 Freising, Germany
[14] Lab. of Food Chemistry and Technology, School of Chemistry, Aristotle University of Thessaloniki, 54124, Thessaloniki, Greece
[15] Scientific Institute of Public Health (WIV-ISP), Food, medicines and consumer safety – Juliette Wytsmanstraat 14, 1050 Brussels (Belgium)

E-mail: giovanna.zappa@enea.it

Abstract. METROFOOD-RI is a new Research Infrastructure (ESFRI Domain "Health & Food") aimed at promoting metrology in food and nutrition constituted by an organised network of physical and electronic facilities allowing to perform cross-cutting research activities and deliver advanced services addressed to different categories of users: research/academy, food inspection and control agencies/policy makers, food business operators, consumers/citizens. Among the core-services of METROFOOD-RI, there is the development and production of new (certified) Reference Materials (RMs). The RI includes in fact - among its physical facilities – an integrated network of plants and laboratories specialised in the production and characterisation of new RMs for the agrifood sector. As further added value, thanks to its network of experimental fields, farms and pilot production plants, METROFOOD-RI is also able to produce the raw materials to be used for preparing the RMs themselves and therefore, so being able to realize customized RM perfectly meeting

the user's needs. The capability of producing food matrix RMs has been recently tested during the "Early Phase" of METROFOOD-RI (H2020 INFRADEV-02-2016 PRO-METROFOOD, G.A. 739568), by implementing a specific pilot service dedicated to the development and characterization of new RMs of rice grains, rice flour and oyster tissue.

1. Introduction

The agrifood sector is a strategic asset of all European Countries and one of the largest and most important economic activities in Europe, with particular socio-economic relevance, since it is vital to maintain employment, preserve rural public goods, supply foodstuffs of quality, and facilitate the integration of small and medium-sized enterprises into the international food chain. In particular, Europe's food and drink industry is a key pillar of the European Union economy and represents the largest manufacturing sector in terms of turnover, added value and employment [1]. The challenges for the agro-industrial sector is to succeed in producing quality food, in sufficient quantity to meet the nutritional needs, effectiveness in consumption of natural resources and reducing the impact on the environment, respecting the ethics of production, meeting the demands of consumers and keeping costs low. Measurements play a key role in every aspect of process control and the evaluation of quality and safety of products: from the determination of nutritional value, bioavailability and biological value of nutrients, to the evaluation of freshness, nutraceutical properties and sensory characteristics, up to chemical and microbiological safety checks, detection of food adulteration and frauds, food authentication and control of raw material and product traceability. In the food sector, measurement reliability is a key factor to effectively address technological innovation, sustainability, food safety and security actions. While in the past metrological aspects related to food trade essentially concerned the "amount" of products (in terms of mass, length, etc.), nowadays, with the spread of EU Regulations on the movement of goods and the need to make objective and comparable (measurable) the "quality" of products, the field of measurements has increasingly involved the fields of chemistry and biology. The enlargement of Metrology to the emerging fields of Chemistry and Biology resulted in a major effort to standardize the terminology and transfer concepts and methodologies originally developed for measurements of physical quantities, to the new application fields. While for the traditional sectors of Metrology, the National Calibration System and the traceability chain are well established and are able to ensure measurement traceability and comparability, the emerging fields of Chemical and Biological Measurements need the enforcement of the Metrological Infrastructure, overcoming intrinsic difficulties and filling the gap between the *"World of Metrology"* and the "*World of Real Measurements"* (related to different conceptual approaches). In physical measurements, metrological traceability is achieved and demonstrated through an unbroken chain of comparisons all having stated uncertainties, up to the National Reference Samples. For chemical measurements, this traceability chain is not always easily achievable. In fact, for biological and natural samples most of the analytical techniques are "destructive" and samples are "unique" with chemical and chemical-physical characteristics often complex and variable in an unpredictable way, because they are subject to the influence of factors not always known and/or parameterized. The application of the metrological rules to chemical and biological measurements requires a wide availability of fit-for-purpose Reference Materials (RMs), which very often constitute the only way to establish traceability of the measurement results and/or to quantify measurement uncertainty. Despite an increase in the production of new RMs in recent years, there is still a lack of fit-for-purpose RMs especially concerning the agrifood sector and there is a continuous need to develop new RMs with different matrix/analyte combinations to cover the different analytical requirements. This need is related to many factors, such as the increasing innovation in analytical techniques, the development of new analytical methods able to detect/determine new parameters of emerging interest, the development of new methods related to food profiling (for quality, authenticity, traceability), and the need to study contaminants of emerging concern [2]. The new Research Infrastructure METROFOOD-RI (www.metrofood.eu) is focused on supporting metrology in food and nutrition; one of its core services

is the development and production of new Reference Materials. In this paper, after a brief introduction to METROFOOD-RI, the main facilities for producing RMs are described and their distinctive features are discussed highlighting the different types of RMs that can be provided.

2. METROFOOD-RI

METROFOOD-RI is a new Pan-European distributed Research Infrastructure of Global interest (ESFRI Domain "Heath & Food"), which allows to carry out different activities supporting data collection and measurement reliability, as well as basic and frontier research in food and nutrition. It aims at providing high quality metrology services in food and nutrition, comprising an important cross-section of highly inter-disciplinary and inter-connected fields throughout the food value chain, including agro-food, sustainable development, food safety, quality, traceability and authenticity, environmental safety, and human health. The general objective is to enhance scientific cooperation and encourage interaction between the various stakeholders, as well as the creation of a common and shared base of data, information and knowledge. METROFOOD-RI mission is to enhance quality and reliability of measurement results and make available and share data, information and metrological tools, in order to enhance scientific excellence in the field of food quality & safety and strengthen scientific knowledge, promoting scientific cooperation and integration. METROFOOD-RI addresses a multidisciplinary field requiring expertise in e.g. chemistry, biology, biochemistry, nutrition, dietetics, toxicology, food technology, general metrology, environmental sciences, agro-ecology, agronomy and environmental chemistry. Dealing with Metrology, it embraces both experimental and theoretical determinations in science & technology and is characterised by an holistic approach to the agro-food sector, which implies measurements being carried out from primary production until final consumption (all along the supply chain, *from farm – to fork*). METROFOOD-RI is constituted by both a *Physical*-RI (P-RI) and an electronic-RI (e-RI) working in close synergy and cooperation, which will allow to carry out different activities supporting data collection and measurement reliability, as well as basic and frontier research in food and nutrition. In particular, the Physical-RI includes a network of laboratories, plants for development/production and characterisation of Reference Materials ("Metro" side), and experimental fields/farms/plants ("Food" side). The e-RI, networking a number of high-level e-resources, will provide a new useful, free access web platform to share and integrate information and data on availability of metrological tools for food analysis. It deals with integration of existing database on food, focusing on emerging needs and collection of data on food composition, nutritional contents and levels of contaminants in foods produced in different geographic regions by applying different technologies. METROFOOD-RI will deliver different services addressed to different user categories (Research/Academic, Policy Makers/Food Inspection and Control Agencies, Food Business Operators, Consumers/Citizens), such as: RM supplying; Proficiency Testing management; development of new methods and devices; food analysis and agro-ecosystem characterization; optimization of food production along the food chain; courses and training services; access to facilities; information services; database querying; data link; data collection, sharing, analysis and display; development and provision of best practices and guidelines.

3. Facilities for RM development

METROFOOD-RI takes advantage of numerous facilities distributed in 18 European Countries that can provide scientific services in an integrated, collaborative and distributed way on the territory. Particularly concerning RM development/preparation, the Physical Infrastructure includes 16 different specialised facilities distributed in the following countries: Italy (ENEA, CREA, ISS), Belgium (WIV-ISP), Czech Republic (CZU/CULS), France (UPPA, UT2A, LNE), Greece (IAPR), Hungary (USZ), Portugal (INSA) and Turkey (TUBITAK). These plants are specialised for the development of agrifood RMs and, thanks to some distinctive features, allow to realise customized RMs and materials with high performances in terms of both homogeneity and stability, as well as to conduct R&D activities on the development of innovative RMs (such as double phase-RMs, multipurpose-RMs, single use-RMs, or driven-RMs) [3,4,5]. Different technologies can be applied for dehydration, such

as lyophilisation or spry-drying, and the application of industrial rotary evaporator systems and conical mixers at different volume scales permit to apply different homogenisation steps at both the wet and the dry stage. The halls hosting the plants allow the control of the environmental process conditions (T, RH) and – where necessary – it's possible to work under inert atmosphere (N2) or under vacuum. Radiosterilisation is possible both at the bulk and bottled stage so to enhance RM shelf life. In addition, anti-contamination inert contact materials are always used. Furthermore, the facilities include not only equipment for RM preparation (considering all the steps of homogenization both at a wet and at a dry stage, milling and grinding, sieving, dehydration, partitioning, dosing), but also for bottling, sealing, labelling, and packaging, as well as dedicated areas for raw material and RM storage at different temperatures and lighting conditions. On the whole, the METROFOOD-RI RM plants cover different production scales, from feasibility studies and small-scale production up to the industrial-scale, and are organised with different production lines for the different types of RMs to be prepared: solid dry materials (e.g. soils and sediments), lyophilised, and liquid materials. They include Matrix-RMs, Process Intermediate-RMs, pure substances and calibrated solutions, and spiked-RMs. Particularly concerning Matrix-RMs, it is possible to realise RMs for any type of matrix that could be of interest for the agrifood sector, including: environmental matrixes, foods of animal origin, foods of plant origin, beverages and vinegar, preparations and total diet, feeds, additives and supplements, non-food agricultural products, packaging and food contact materials. METROFOOD-RI can count also on a wide series of advanced analytical laboratories to be employed for characterising the RM for a very wide set of parameters, so being able to obtain pure substances for calibration or Matrix-RMs accompanied by certified, reference or information values for a very wide set of different chemical, physical-chemical, (micro)biological parameters considering, as an example, nutrients and bioactive compounds, bioactivity and reactivity, organic contaminants and residues, inorganic contaminants (both toxic elements and speciation, and organometals), isotopes, microbiological analysis, physical characterization, allergens profile, and genetic analyses. Furthermore, thanks to the "Food" side of the Infrastructure, with its network of experimental fields, farms, production plants, and kitchen labs, it is possible to produce in-house the materials needed to realise the food matrix-RMs, so having the possibility to prepare matrix-RMs of well known origin (geographic, genetic, of production process, etc.), or to obtain "driven-RMs". Finally, these physical facilities are supported by the e-facilities dedicated to statistical analysis and data management and Proficiency Testing provision, thus providing a further contribution and improvement for the characterisation and certification. The actual capability of METROFOOD-RI to produce food matrix RMs has been recently tested during the "Early Phase" of the Infrastructure (H2020 INFRADEV-02-2016 PRO-METROFOOD, G.A. 739568), by implementing a specific pilot service dedicated to the development and characterization of new RMs of rice grains, rice flour and oyster tissue [6].

4. References

[1] ETP Food for Life SRIA 2016 "Food for tomorrow's consumer" - http://etp.fooddrinkeurope.eu
[2] M. Rychlik, et al. (2018) Ensuring Food Integrity by Metrology and FAIR Data Principles, Front. Chem. 6:49. doi: 10.3389/fchem.2018.00049
[3] C Zoani et al. Development of innovative Reference Materials for the agrofood sector. Int.Conf. 1st IMEKOFOODS Rome,12-15/10/ 2014
[4] G Zappa et al. Feasibility study for the development of a Multiparameter-Reference Material for food contact material testing and characterization. XXI IMEKO World Congress, Prague 2015
[5] G Zappa, C Zoani. Double Phase-RM: a New Opportunity in Food Analysis. Annual Workshop of the EU RL CEFAO. Rome, 28/09/2016. ISTISAN Congressi 16/C5 [ISSN: 0393-5620; 2384-857X]
[6] C Zoani at al. Feasibility studies for new food matrix-Reference Materials. Submitted for presentation XXII IMEKO World Congress, Belfast 2018ù

Acknowledgments - PRO-METROFOOD project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 739568.