DEVELOPMENT OF NEW MULTIPURPOSE-REFERENCE MATERIALS OF RICE GRAINS AND RICE FLOUR

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Abstract – This paper details with the preparation of new *Multipurpose*-Reference Materials (RMs) of rice grains and rice flour performed as a pilot study within the PRO-METROFOOD-RI project. RMs will be characterized for different parameters including contaminants, biomarkers, stable isotopes and markers of origin/authenticity. These RMs will also have a major role in quality control and quality assurance of other food products.

Keywords: rice, reference material, characterization, pilot study, METROFOOD-RI

1. INTRODUCTION

Metrological support represents an essential part of METROFOOD-RI (*Infrastructure for Promoting Metrology in Food and Nutrition* – www.metrofood.eu) since many analytical methods and approaches are not yet standardised and Certified Reference Materials (CRM) are missing. One of the most important factors for improving method performance and lowering measurement uncertainty is the availability of suitable Reference Materials (RMs). Despite an increase in the production of new RMs in recent years, there is still a lack of fit-for-purpose RMs especially for what concerns the agrofood sector and there is a continuous need to develop new RMs with different matrix/analyte combinations to cover 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. In particular, within the PRO-METROFOOD project, pilot services have been organized to test the inter-operability and to demonstrate the actual capability of METROFOOD-RI to deliver services and to add value as an organized RI. One potential service offered by METROFOOD-RI is the development and provision of new (customized) RMs. A pilot study related to the development of new *Multipurpose*-RMs has been organized. The RMs for the pilot were selected based on: current availability, gaps in current RM production, and main analytical challenges related to the selected matrixes.

In this paper the development of new *Multipurpose*-Reference Materials (RMs) of rice (grains, flour) is presented. The development of new rice-RMs has been selected to promote, besides the characterization of "conventional" parameters, the use of stable isotopes and other biomarkers for origin/authenticity identification. The realization of *multipurpose*-RMs represents a major innovation that paves the way for the development of new RM having certified, reference or information values for several different parameters.

2. MATERIALS AND METHODS

Two batches of rice grains and rice flour RM were prepared from 80 kg lots of super-fine *Carnaroli* white rice grains and super-fine *Carnaroli* white rice flour (same variety and same origin) provided by Ente Nazionale Risi (IT). RM preparation was performed initially at the ENEA Agrofood RM Plant c/o Trisaia Research Centre (industrial scale production - Basilicata Region, IT) and then

completed at the RM Plant c/o Casaccia Research Centre (small scale production - Lazio Region, IT). Raw materials were first homogenized in a Vrieco Nauta ® Conical Mixer (V = 240 l – Fig. 1) equipped with a dosing valve, with the following order of processing: rice grains, rice flour. After homogenization of the flour, the dust remaining on the Nauta inner walls was removed using N_2.

Fig. 1. Vrieco Nauta ® Conical Mixer (V = 240 l)

In both cases, the Nauta was first rinsed with an aliquot (5 kg) of the material to be processed; rinsing was carried out by rotating the Nauta for 15 minutes and collecting and reloading multiple times the same material. This material was then discarded. The Nauta was then loaded with all the available material (75 kg) and allowed to rotate for 2 hours. Aliquots (3.2 kg) were then collected in plastic bags using the dosing valve, and always maintaining rotation. The bags were sealed under vacuum and then shipped to the Casaccia Research Centre. On arrival, the material contained in each bag was re-homogenised and partitioned using a Retsh sample divider (8 sub-aliquots) by applying a two-step procedure. Finally, 80 g and 50 g aliquots of rice grains and rice flour, respectively, where collected. Figure 2 shows an overview of the RM preparation procedure. Once prepared, the materials were shipped to different laboratories of the PRO-METROFOOD partners for characterization.

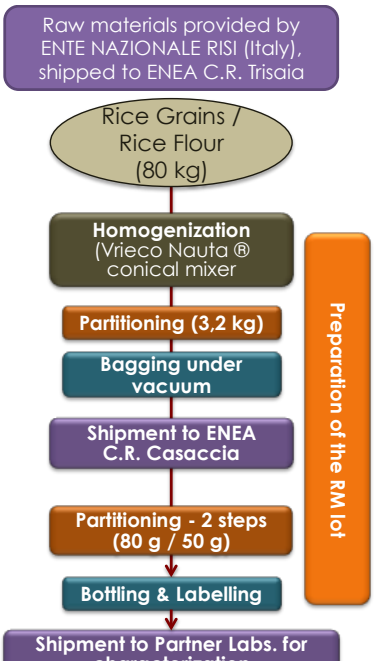

Fig. 2. RM preparation procedure

Each participant received five aliquots of the RM, together with a blank bottle. It was request to confirm the delivery of the samples by regular mail or e-mail using the sample receipt form on arrival. The information requested in the sample receipt included the following: ID numbers of the test-aliquots, arrival date, material conditions at the receipt (intact, damaged, notes), storage conditions, and the person receiving the materials. The shipment was accompanied by a document of "Guidance for characterization of Reference Materials" providing instructions to be followed for material storage and handling. It was recommended that the RMs be stored at room temperature (T max = 25 °C) in the dark in airtight containers in a low-humidity environment. Instructions were provided for determining moisture content, using the blank bottle, and for the analysis and submission of results.

The amount of material for analysis and analysis itself followed the usual protocols of the participating laboratories.

Table 1 provides information regarding the participating laboratories and the parameters used to characterize the RMs. To provide a wider set of parameters, each participant identified their own analytical capability and expertise for RM

characterization including vitamins, fibres, other nutritional and bioactive compounds, mycotoxins, antibiotics residues, other organic contaminants, toxic elements, speciation, contaminants of emerging concern, origin/authenticity/isotopes and the presence of genetically modified organisms (GSO).

Table 1. Participating laboratories and selected parameters for RM characterization

Institution/Abbreviation/ Country	Parameters for RM characterization				
	1	2	3	4	5
Agenzia Nazionale per le Nuove Tecnologie, l'Energia e lo Sviluppo Economico Sostenibile/ENEA/IT (6 labs)			x	x	x
Italian National Research Council/CNR/IT (6 labs)	x	x			x
Italian National Metrology Institute/INRIM/IT				x	x
Italian National Institute of Health/ISS/IT (2 labs)		x	x		
Council for Agricultural Research and Economics /CREA/IT (3 labs)	x	x			x
Brescia University/UniBS/IT			x		
Instituto Nacional de Saúde Dr. Ricardo Jorge/INSA/PT	x		x	X	
National Research & Development Institute for Food Bioresources/ IBA/RO	x	x	x		
Fundación CIDETEC/CIDETEC/ES		x	x		
Université de Pau et des Pays de l'Andour/UPPA/FR		x	x		x
Laboratoire national de métrologie et d'essais/LNE/FR		x	x		
Agence nationale de sécurité sanitaire de l'alimentation, de l'environnement et du travail/ANSES/FR		x	x		
Association pour le Développement de l'Enseignement et des Recherches auprès des universités, des centres de recherche et des entreprises d'Aquitaine/ADERA/UT2A/FR			x	x	x
Aristotelio Panepistimio Thessalonikis/AUTH/GR	x				
Czech University of Life Sciences Prague/CULS/CZ	x		x		
University of Szeged/USZ/HU		x	x	x	
Technical University of Munich/TUM/DE		x			x
Jozef Stefan Institute/JSI/SI (2 labs)			x		x
National Institute of Biology/NIB/SI		x			
Science and Research Centre Koper/ZRC Koper/SI	x	x			
Institute of Public Health of the Republic of Macedonia/IJZHP/MK	x		x		
Faculty of Agricultural Sciences and Food – Univ. "Ss Cyril and Methodius/FASF/MK	x				
Institute Scientifique de Santé Publique/WIV-ISP/BE		x	x		
TUBITAK National Metrology Institute/TUBITAK/TR		x	x		
DAS Foundation "For a Moldova Based on Knowledge"/DAS/MD	x	x	x		

1 – nutritional and bioactive compounds
2 – organic contaminants and genetically modified organisms (GSO)
3 – inorganic contaminants
4 – contaminants of emerging concerns
5 – origin/authenticity/isotope

Further, specific spreadsheets were prepared and circulated in order to collect all necessary information about the analytical techniques and methods used by each laboratory, as well as for collecting the data (measurement results for each replicate; mean value; standard deviation; moisture content, also indicating if the results are corrected for the moisture content or not; correction on the basis of a recovery factor; correction for blank levels; Limit of Quantification). The participants were free to choose any suitable analytical method, but must provide a full description of the methodology. It was recommended to carry out the analytical measurements in parallel. Furthermore, metrological traceability of measurement results should be obtained using calibrated devices and appropriate measurement standards and validated methods. If not, attention must be paid to the application of appropriate calibration procedures and evaluation of matrix interferences and blank levels. It was also recommended that each laboratory provide a full uncertainty budget. Contributions to the overall uncertainty that arise from sample preparation, instrumental

determination, reference materials used and other parameter specific to the method of analysis chosen by the participant should be taken into account. Each laboratory was assigned an ID number and the data collection was organized to guarantee the anonymity of the participating laboratories.
All results will be statistically evaluated. To date, homogeneity studies are underway for evaluating the homogeneity within-bottle and between-bottles. Isochronous stability tests under thermal (at different temperatures) and luminous stress on both materials (grains and flour) have also begun.

The plan is to obtain all necessary data by the end of September 2017, and for the official presentation and discussion of the results to be given at a special meeting in November 2017 in Ljubljana, Slovenia.

3. FURTHER DEVELOPMENTS AND CONCLUSIONS

At the end of the pilot service, well-characterized multipurpose-RMs of rice grains and rice flour will be available. Furthermore, it is planned to promote the RMs in order to obtain certification.

The expertise of the participants will assure the preparation of high quality RMs that will have a major impact on quality control and quality assurance for rice and other food products. The RMs presented in this study will be of interest from a market and trade perspective, for rice producers wishing to demonstrate the origin and authenticity of their products, and in regards to analytical issues relating to the EU market. In addition, the analytical capacity will support the metrology system providing traceable and comparable measurement data, e.g. in the framework of European Metrology System (EURAMET).

5. ACKNOWLEDGEMENT

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