HOMOGENEITY STUDY FOR CERTIFICATION OF A BRAZILIAN FISH REFERENCE MATERIAL *(Micropogonias furnieri)*

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To assure the quality and comparability of analytical results, analytical laboratories need to demonstrate the validity and reliability of produced analytical results. Usually this is done through participation in proficiency testing and the use of certified reference materials.

Certified reference materials (CRMs) are still not widely used in Brazil and other Latin American countries. The main reason is the high cost of these materials, since most of them are imported, and due to unavailability of CRMs which fulfill the national needs.

The International Atomic Energy Agency (IAEA) has been supporting several projects aiming at improving capabilities of Latin American laboratories. In this context, an IAEA technical project (RLA 2/014) "Improvement of analytical quality through proficiency testing and certification of matrix reference materials using nuclear and related analytical techniques in the Latin American nuclear analytical techniques network"- ARCAL XCVII) has started in 2009. In this project a new Brazilian fish reference material was prepared to be used in interlaboratory programs and/or proficiency tests, as well as for validation of methods for the determination of trace elements of environmental and nutritional concern.

The Whitemouth croaker fish (*Micropogonias furnieri*) was chosen as it is the second most produced fish in Brazil and it is widely distributed and consumed in the Latin American countries. For preparation of the candidate CRM, about 300 kg of fish was collected at the Brazilian southern coast. Each fish weighed about 900 g and was transported to the laboratory without head, and gutted. Only the edible parts were used. About 80% mass was lost in the freeze drying process. After the lyophilization, the material was milled in a planetary ball mill and sieved in a polyester sieve with sieve opening of 105 µm. The moisture content of the bulk material was determined as 6% (w/w). Thus, the powdered material was again submitted to the freeze drying process until the moisture content level was at 3% (w/w). After that, the material was homogenized in a 30L-Y-Homogeneizer for 72 hours and was bottled in 534 bottles with approximately 25 g each. The bottles were gamma-ray irradiated at the ^{60}Co irradiation facility at IPEN-CNEN/SP in order to enhance the material stability.

For the homogeneity study ten bottles of the fish material were randomly selected covering the whole range of bottles for the determination of the mass fractions of As, K and Na by Instrumental Neutron Activation (INAA), Cd by Graphite Furnace Absorption Atomic Spectrometry (GF AAS), and Hg by Cold Vapor Atomic Absorption Spectrometry (CV AAS).

Six subsamples of each bottle were analyzed in repeatability conditions and potential outliers identified with the Grubbs' test. One-way analysis of variance test, ANOVA (ISO Guide 35, 2006) [1] was applied to assess the between and within bottle variability. Table 1 shows the ANOVA outputs.

Table 1. ANOVA OUTPUT FOR THE BETWEEN BOTTLE HOMOGENEITY STUDY FOR As, Cd, Hg, K, Na IN THE FISH REFERENCE MATERIAL

Element	Variation source	Mean square	F	P value	F crit
As	between	0.0875	0.422	0.917	2.073
	within	0.207			
Cd	between	1.473	1.12	0.367	2.073
	within	1.315			
Hg	between	287	0.781	0.634	2.073
	within	368			
K	between	1657302	1.10	0.378	2.073
	within	1503158			
Na	between	47733	0.916	0.519	2.073
	within	52099			

Table 2 presents the range of mass fractions for the analyzed elements on a wet mass basis (no correction for residual moisture was performed) and the values of the between bottle homogeneity uncertainty, u_{bb}. The uncertainty values were estimated according to ISO Guide 35 [1].

Table 2. RANGE MASS FRACTION VALUES ($\mu g\ g^{-1}$) WITH THE UNCERTAINTIES OF BETWEEN-BOTTLE HOMOGENEITY STUDY

Element	Range ($\mu\ g^{-1}$)	u_{bb}	u_{bb} %
As	4.5 – 6.5	0.0832	1.58
Cd	0.010 – 0.020	0.000162	1.06
Hg	0.20 – 0.30	0.00350	1.52
K	9500 – 15000	160	1.33
Na	3500 – 5000	41.7	0.941

u_{bb} between bottle homogeneity uncertainty; u_{bb} %: Contribution of the inhomogeneity

As shown in Table 2, u_{bb} estimates for all elements were below 2%, which is acceptable for biological matrix reference materials at these levels of mass fractions. The u_{bb} values will be used in the next step of the certification process of the fish reference material as a contribution of the between bottle homogeneity to the mass fraction combined standard uncertainty (u_{MRC}).

References

[1] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, ISO Guide 35, Reference materials – General and statistical principles for certification, ISO, Geneva (2006).

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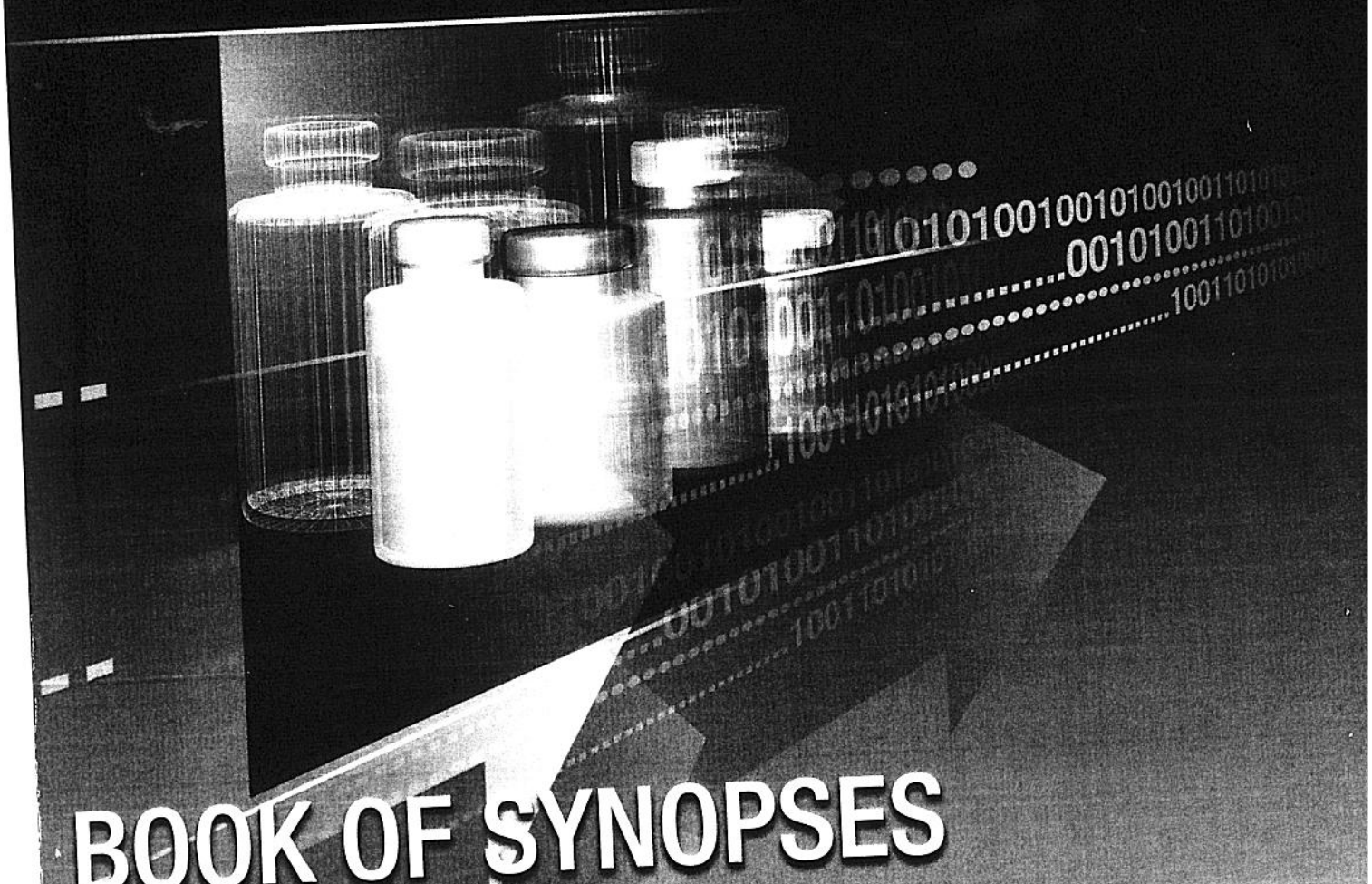

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