

CERTIFICATION OF BIOLOGICAL REFERENCE MATERIALS: PARTICIPATION OF THE NEUTRON ACTIVATION LABORATORY (LAN-IPEN/CNEN-SP)

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ABSTRACT

Analytical laboratories have as one of their important goals to demonstrate their competence allowing international acceptance and comparison of analytical data. The IPEN Neutron Activation Laboratory (LAN-IPEN) has implemented its Quality Assurance Program which comprises, among other activities, the participation in intercomparison runs. As a part of this Quality Assurance Program, LAN-IPEN has participated in interlaboratorial trials to analyze two biological candidate reference materials: INCT-CF-3 Corn Flour and INCT-SBF-4 Soya Bean Flour from the Institute of Nuclear Chemistry And Technology (Warszawa, Poland). The elements Br, Ca, Co, Cs, Fe, K, Na, Rb and Zn were analyzed in the candidate reference materials by instrumental neutron activation analysis (INAA). The performance of the laboratory was statistically evaluated in relation to the consensus values for these materials using the Z-Score test. This laboratory evaluation method has been accepted as a standard by ISO/IUPAC. In the present study, adequate Z-Score values ($|Z| < 2$) were observed for all of the analyzed elements, confirming the accuracy of the nuclear methodology employed. The contribution of LAN-IPEN in the certification of the reference materials analyzed was very important, since the results provided were used in the statistical evaluation of the certified value.

1. INTRODUCTION

Lately, analytical laboratories have been very much concerned with analytical quality control. One of the main goals of all laboratories around the world is to assure the quality and credibility of their results, thus allowing international acceptance and comparison of analytical data. The IPEN Neutron Activation Analysis Laboratory (LAN-IPEN) has implemented a Quality Assurance Program based on ISO/IEC 17025:2001, which comprises among other activities the participation in intercomparison runs. These intercomparison runs are considered one of the standard procedures in quality assurance programs, since they enable the laboratories to find out deficiencies in their analytical methods, allowing them to improve their data. The results from each laboratory, in general a mean of six replicates, are sent to the organizers of the interlaboratory comparison and are statistically evaluated with all contributed data. For each element analyzed, a certified or recommended or information value is assigned, depending on the consensus of the different laboratories data. The performance of each laboratory is verified by comparing the obtained results with the certified/recommended values. In the present paper, this comparison was performed by using the Z-score criterion [1], which can be used to judge the accuracy of the laboratory's routine determinations, with results with $|Z| < 2$ considered satisfactory.

This work is part of a collaborative study intended to certify two candidate reference materials developed by the Institute of Nuclear Chemistry and Technology of Warsaw, Poland – INCT-SBF-4 *Soya Bean Flour* and INCT-CF-3 *Corn Flour*.

1.1. INCT-SBF-4 *Soya Bean Flour*

The candidate reference material was procured from POLGRUNT (Poland) through Pro-Vita (Poland). Flour was prepared from non-genetically modified soya bean grown in India [2].

1.2. INCT-CF-3 *Corn Flour*

The other candidate reference material was produced from corn grown in Poland by ViVi-Tak sc. (Poland) according to Polish standards [3].

2. EXPERIMENTAL PROCEDURE

Samples with about 150 mg were accurately weighed in polyethylene envelopes, previously cleaned with diluted nitric acid solution. The certified reference materials (CRMs) used for elemental composition determination in this study were SRM 1547 *Peach Leaves* (NIST/USA) and *DR-N diorite* (ARNT/France), and they were prepared the same way as the samples. Samples and CRMs were inserted in aluminum containers specially developed for the use in the IEA-R1 nuclear reactor of Instituto de Pesquisas Energéticas e Nucleares and irradiated for 8 hours in a thermal neutron flux of $\sim 5 \times 10^{12} \text{ n.cm}^{-2}.\text{s}^{-1}$.

Measurements of the induced gamma-ray activity were carried out by using a Canberra GX20190 hyperpure Ge detector. The multichannel analyzer was a 8192 channel Canberra S-100 plug-in-card in a PC computer. The resolution (FWHM) of the system was 1.90 keV for the 1332 keV gamma-ray of ^{60}Co , and the nominal efficiency of the detector is 20%.

Two series of measurements were performed; the first was done from 5 to 7 days after irradiation and the second one after 15-20 days of decay; all measurements were performed with a sample-detector distance of 3 cm. Counting times ranged from 1 to 3 hours. Gamma-ray spectra were processed by using an in-house gamma-ray software, VISPECT [4], which locates peak position and calculates the energies and net areas. The elements Br, Ca, Co, Cs, Fe, K, Na, Rb and Zn were studied in all samples.

The final results from our analysis were calculated from the average of the six replicate samples, and the uncertainty was assigned using the standard deviation of the mean.

3. RESULTS AND DISCUSSION

Table 1 shows the results from our analysis compared to the certified values for the two reference materials studied.

Table 1. Results of the analysis of the two reference materials compared to the certified values.

Element	INCT-SBF-4 <i>Soya Bean Flour</i>		INCT-CF-3 <i>Corn Flour</i>	
	This Study	Certified Value	This Study	Certified Value
Br (mg kg ⁻¹)	2.317±0.074	2.40±0.17	0.390±0.028	0.388±0.046
Ca (%)	2667±267	2467±170		
Co (ng g ⁻¹)	93.2±13.8	95.6±5.8		
Cs (ng g ⁻¹)	130±10	129.1±4.6		
Fe (mg kg ⁻¹)	99.0±8.3	90.8±4.0	34.3±4.1	32.0±1.4
K (mg kg ⁻¹)	26080±1617	24230±830	3467±198	3157±119
Na (mg kg ⁻¹)	2.84±0.44	5.5 *		
Rb (mg kg ⁻¹)	33.2±0.8	31.7±1.7	0.840±0.096	0.912±0.042
Zn (mg kg ⁻¹)	54.8±3.9	52.3±1.3	20.83±0.75	20.09±0.76

* This is an information value and has no uncertainty assigned to it.

The Z-Score control chart for the analysis of the INCT-SBF-4 *Soya Bean Flour* reference material, calculated from the values in Table 1, is presented in Fig. 1, showing that our results agree with the values assigned for this material after the certification process was accomplished [2]; the element Na was also detected and quantified in this analysis, but as the certification process did not reach a consensus value, this element has only an “information” value and the Z-Score could not be calculated.

The Z-Score control chart for the INCT-CF-3 *Corn Flour* reference material is presented in Fig. 2; these results also show that our laboratory’s analyses were consistent with the certified values [3]. For this material, our methodology was not able to assign values for the elements Ca, Co, Cs and Na as their concentration was below the quantification limit for the INAA method used.

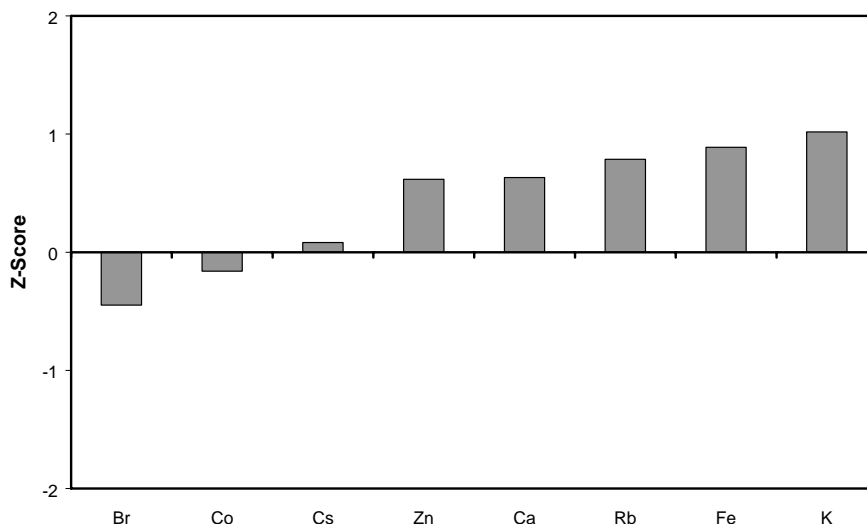


Figure 1. Control chart for the INCT-SBF-4 *Soya Bean Flour* reference material.

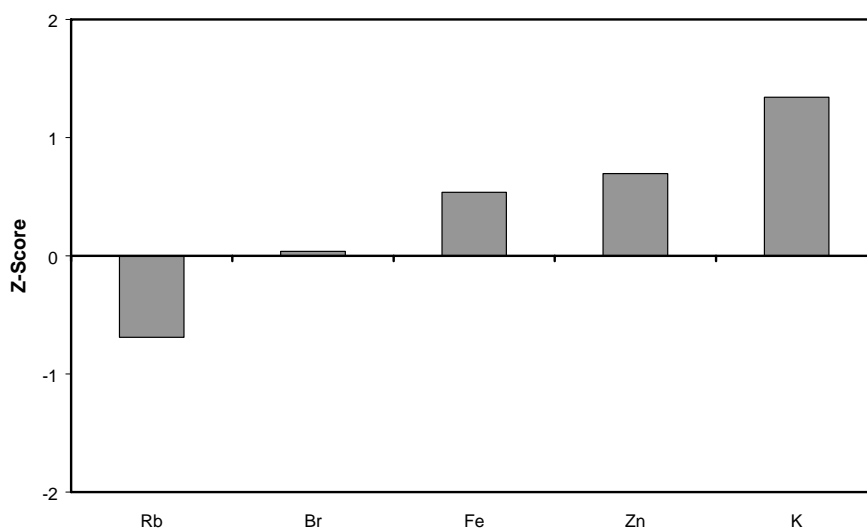


Figure 2. Control chart for the INCT-CF-3 *Corn Flour* reference material.

3. CONCLUSIONS

The comparison of the results obtained in LAN-IPEN with the final results of the certification process of the two candidate reference materials studied show that our results agree with the certified values for all the detected elements. The contribution of LAN to the certification process of both reference materials was important, as the results from the laboratory were used in the assignment and statistical evaluation of the certified values.

REFERENCES

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