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PRELIMINARY RESULTS FOR THE K_0-INAA METHODOLOGY IMPLEMENTATION AT THE NEUTRON ACTIVATION ANALYSIS LABORATORY, LAN-IPEN, USING K_0-IAEA SOFTWARE

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ABSTRACT

The present paper presents the preliminary results obtained in the implementation of the k_0 standardization method at the Neutron Activation Laboratory (LAN) at IPEN, São Paulo, Brazil, using the program k_0-IAEA, provided by The International Atomic Energy Agency (IAEA). This method is an important alternative for the comparative neutron activation analysis, which has been used for several years at LAN-IPEN. This quasi-absolute standardization method presents a great advantage with relation to the comparative method, since it does not require the preparation of accurate individual standards for each analysed element, which is very laborious and time-consuming. The k_0 method allows the determination of almost all elements whose gamma-ray peaks are present in the gamma spectrum. The analysis of gamma-ray spectra and the calculation of concentration are performed by the k_0 software, thus the analysis time is shortened: the time spent to calculate, for instance, the concentration of 25 elements in 10 samples takes about 5 minutes.The efficiency curve of one of the gamma-ray spectrometers used at LAN was determined by measuring calibrated radioactive sources at the usually utilised counting geometries. The parameters α and f were determined by irradiating a Certified Nuclear Reference Material IRMM-530R Al-0,1% Au alloys and high purity zirconium comparators at the IEA-R1 nuclear reactor of IPEN. In order to evaluate the efficiency of the methodology, the geological reference material basalt JB-1 (GSJ) was analysed. The preliminary results obtained showed promising results in spite of some discrepancies of the data in comparison to certified values. These preliminary results indicate that some improvements in the parameters required for the use of the k_0-IAEA software should be made so that the k_0-NAA software can be completely successful.

1. INTRODUCTION

The neutron activation analysis laboratory at IPEN (LAN-IPEN) has analysed geological, biological and archaeological matrices samples for more than 30 years, using the comparative method of analysis by neutron activation with the IEA-R1 reactor of IPEN [1]. In this method, samples and standards are irradiated simultaneously with neutrons under the same conditions. Elemental concentrations are calculated by comparison of the activities of the gamma rays from the sample and standard. This procedure requires the preparation of element standards, which is very laborious and time consuming. Furthermore, some elements present in the sample can not be analyzed due to the absence of a corresponding element standard.

To solve this problem, the k_0 standardization method was developed by the Institute of Nuclear Sciences, Gent, Belgium[2]. This method consists in determining the flux parameters, such as thermal to epithermal neutron flux ratio *f* and *α* by the irradiation of flux monitors. The concentrations of the elements are calculated in relation to one element, usually gold, using k_0 and Q_0 literature values, eliminating the necessity of a standard. The k_0-NAA method has been used in several neutron activation laboratories in Brazil and in other countries all over the world [4, 5, 6, 7].

With the k_0-NAA method, significant advances have been made in determining the physical constants involved, due to the improvement of computer software for carrying out the calculations required for analysis. In this study, the results of the implementation of the k_0-NAA with the k_0-IAEA software[3, 8] at LAN-IPEN are presented.

Obtaining an accurate gamma-ray detection efficiency curve is essential in the k_0 standardization method. The hyperpure germanium detector was calibrated for photopeak efficiency.

The flux parameters *f* and *α* were determined for the short irradiation facility of the IAEA-R1 nuclear reactor of IPEN. To obtain these factors, the "bare-triple-monitor" method with ^{197}Au-^{96}Zr-^{94}Zr was used.

To validate the methodology, the geological reference material JB-1 (GSJ) was analyzed.

2. EXPERIMENTAL

The parameters *f* and α for the short irradiation facility of the IEA-R1 nuclear reactor were determined by irradiating about 5 mg of Al-0,1% Au wire (Certified Reference Material IRMM-530R) and 40 mg of Zr foil 99.9%. The iron monitor (~ 25 mg of Fe) was irradiated to calculate the epicadmium-to-fission neutron flux ratio using the nuclides ^{54}Mn and ^{59}Fe. This ratio was used to quantify the elements using the nuclides which are produced by threshold reactions (Ni by ^{58}Co, for example). The values obtained were: $f = 34.95 \pm 0.48$ and $\alpha = 0.05 \pm 0.01$.

Methodology validation was done by analyzing the reference material JB-1 (GSJ) which was irradiated with all monitors. The preparation of the sample, irradiation and counting conditions were the same usually employed in laboratory comparative INAA. 100 mg of the sample was weighed in a polyethylene envelope previously cleaned with diluted nitric acid solution. Samples were placed in polyethylene containers (*rabbit*) and irradiated at the reactor IEA-R1 of IPEN-CNEN/SP at a thermal neutron flux of approximately $10^{13}\,ncm^{-2}s^{-1}$ for 1 min.

All countings were performed by a p-type coaxial HPGe detector (Canberra GX2020) which has 20% relative efficiency and 1.90 FWHM at 1332 keV of ^{60}Co. The detector is connected to a multichannel analyser Canberra 8192 S-100 available in a microcomputer at LAN-IPEN. Full energy peak efficiency calibration of the detector was carried out in the range of 59 keV to 1408 keV, in the geometry usually used in the gamma-ray measurements at LAN-IPEN (about 10 cm from the crystal), using ^{241}Am, ^{57}Co, ^{60}Co, ^{137}Cs and ^{152}Eu point calibration sources.

3. RESULTS AND DISCUSSIONS

The results for the reference material basalt JB-1 as well as certificates and recommended values, are shown in Table 1. The errors associated with results are related to counting statistics. Although the data obtained are preliminary, we obtained satisfactory agreement to the reference values as shown in Table 1. Some errors are of about 40% which demonstrate that the irradiation and counting must be improved. Figure 1 show the normalized results in relation to certified values.

The study will continue characterizing the flux parameters of other irradiation facilities at IEA-R1 reactor and analysing other reference materials, representatives of the various types of matrices commonly analysed at the LAN-IPEN as soil, rocks, sediments, plants and biological samples.

Table 1. Results obtained for the reference material JB-1 (GSJ).

Element	Obtained mg/kg	Certified mg/kg
Na	212±19	206
K	121±17	118
Mn	1174±129	1239
Sr	431±202	435
Ba	480±101	490
La	37±7	36
Sm	3.50±1.40	5.16
Eu	1.5±0.2	1.5
Dy	4.1±0.7	4.1
W	20.3±8.1	19.4

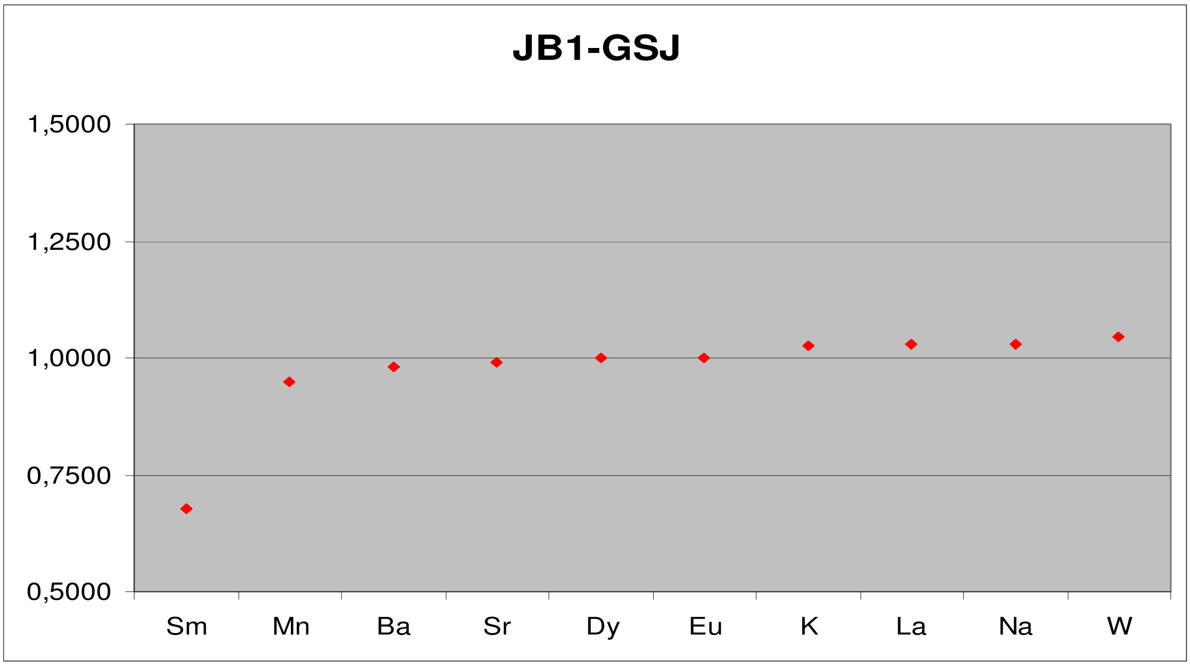

Figure 1. Ratios between obtained values and recommended values for JB1-GSJ

4. CONCLUSIONS

The k_0-NAA method with the computer program k_0-IAEA provided results for some elements in geological reference material JB-1 (GSJ) with good precision. Thus, these seem to be of great potential for the use of the parametric method for neutron activation analysis at LAN-IPEN. The execution time of the analysis is shorter than the benchmark, since there is no need for standards. The analysis of gamma-ray spectra and calculations of the concentrations are all executed by the k_0-IAEA program. In conclusion, the results indicate that the implementation of the k_0-NAA at the Neutron Activation Laboratory LAN-IPEN should increase the analytical potential of the laboratory, assuring and maintaining the quality of the analytical results.

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