Implementation of the k_0-standardization method for analysis of geological samples at the Neutron Activation Analysis Laboratory, São Paulo, Brazil

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INTRODUCTION

Instrumental neutron activation analysis (INAA) is one of the most important applications of the IEA-R1 nuclear reactor. The Neutron Activation Analysis Laboratory (LAN-IPEN) has been analyzing geological samples for many years with the INAA comparative method, for geochemical and environmental studies. The k_0-NAA method, developed by the Institute of Nuclear Sciences, Gent, Belgium, has been increasingly used, as it requires only a single comparator such as ^{197}Au for multielement determination instead of the multielement standards required in the relative method. This study presents the results obtained in the implementation of the k_0 standardization method at LAN-IPEN, for geological samples analysis, by using the k_0-IAEA program, provided by The International Atomic Energy Agency (IAEA).

EXPERIMENTAL

The efficiency curve of the p-type coaxial HPGe detector (Canberra GX2020) was determined. The detector was 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 ^{137}Cs and ^{152}Eu point calibration sources.

The thermal to epithermal flux ratio f and the shape factor α of the epithermal flux distribution of the IEA-R1 nuclear reactor of IPEN were determined for the pneumatic irradiation facility and one selected irradiation position, for short and long irradiation, respectively. To obtain these factors, the "bare triple-monitor" method with ^{197}Au-^{96}Zr-^{94}Zr was used. The Certified Nuclear Reference Material IRMM-530R Al-0,1% Au alloy, high purity zirconium, Ni and Lu comparators were irradiated.

FLUX PARAMETERS

Irradiation flux parameters at the Pneumatic Station of IEA-R1

Paramaters	
Thermal neutron flux, Φ_{th} (m^{-2} s^{-1})	(8.6 ± 0.2) 10^{16}
Neutron Temperature, T_n, (K)	310.00 ± 0.01
Thermal to epithermal flux ratio, f	44 ± 6
Deviation of the epithermal neutron flux distribution from the ideal 1/E law, α	-0.08 ± 0.02

Irradiation flux parameters at the 24B/Position 2 in the IEA-R1

Parameters	
Thermal neutron flux, Φ_{th} (m^{-2} s^{-1})	(6.3 ± 0.09) ×10^{16}
Neutron Temperature, T_n, (K)	310 ± 5
Thermal to epithermal flux ratio, f	35 ± 1
Deviation of the epithermal neutron flux distribution from the ideal 1/E law, α	-0.049 ± 0.006

METHOD ACCURACY AND PRECISION EVALUATION

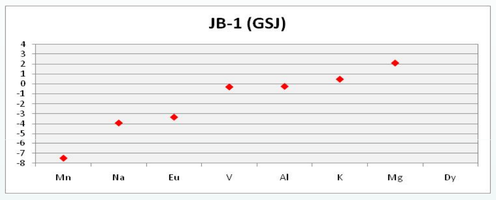
Data validation was done by analyzing (six replicates) of the reference materials: basalt BE-N (IWG-GIT) e JB-1 (USJ), andesite AGV-1 (USGS), granite GS-N (IWG-GIT), soil SOIL-7 (IAEA) and sediment Buffalo River Sediment (NIST-BRS-8704), which represent different geological matrices.

The preparation of the sample, irradiation and counting conditions were the same usually employed in relative INAA standardization.

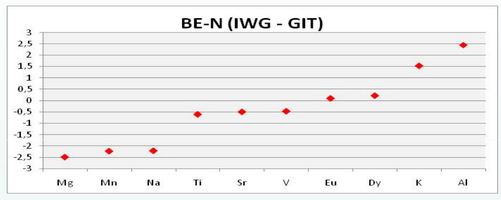
RESULTS AND DISCUSSIONS

The results obtained for the reference materials were analyzed by using the z-score criterion. The z-score were all within $|z|<3$, showing that the results are in a confidence level of 99% of the certified value. It is important to note that the results obtained were randomly above and below the certified values, showing that there is no systematic errors.

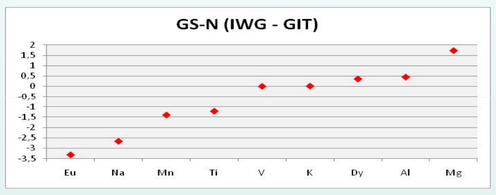
Short Irradiation



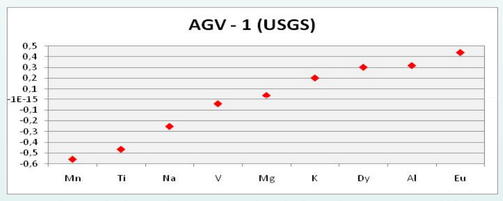
Control chart (z-score values) for elements in JB-1



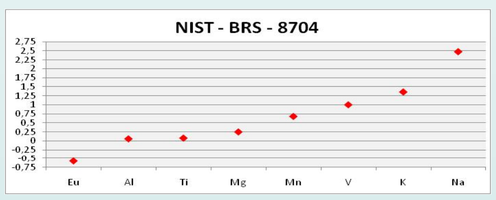
Control chart (z-score values) for elements in BE-N



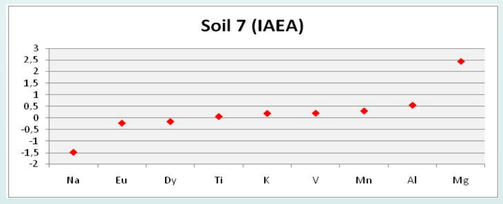
Control chart (z-score values) for elements in GS-N



Control chart (z-score values) for elements in AGV-1

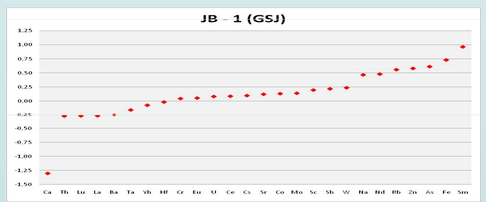


Control chart (z-score values) for elements in Buffalo River Sediment

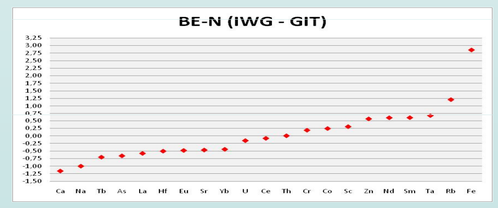


Control chart (z-score values) for elements in Soil-7)

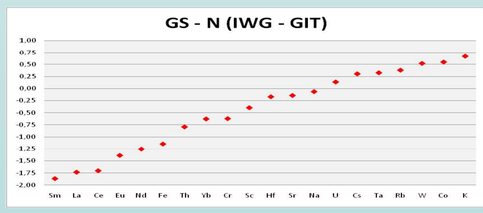
Long irradiation



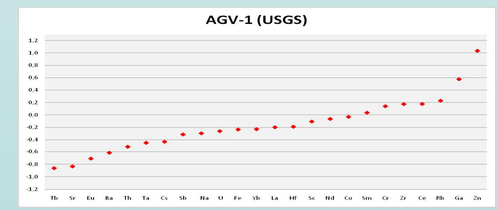
Control chart (z-score values) for elements in JB-1



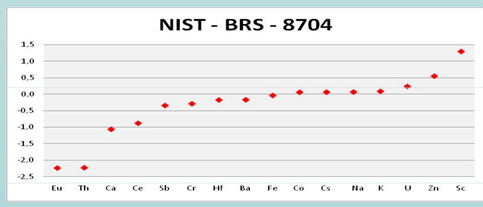
Control chart (z-score values) for elements in BE-N



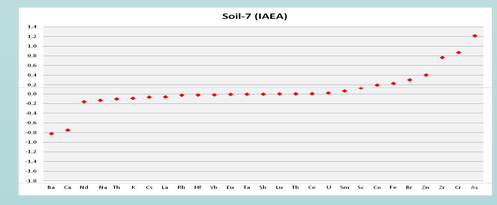
Control chart (z-score values) for elements in GS-N



Control chart (z-score values) for elements in AGV-1



Control chart (z-score values) for elements in Buffalo River Sediment



Control chart (z-score values) for elements in Soil-7

CONCLUSIONS

The k_0-NAA method with the k_0-IAEA software provided results for more than 30 elements in the geological reference materials analyzed. These results show that the implementation of the k_0-NAA at the Neutron Activation Laboratory (LAN-IPEN) will increase the analytical potential of the laboratory, maintaining the standard of quality.

The authors wish to thank FAPESP for financial support