

## APPLICATION OF NEUTRON ACTIVATION ANALYSIS TO STUDIES OF GEOLOGICAL MATERIALS

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Trace elements, including U, Th, Ba, Sc, Rb, Ta, Cs, Co, Hf and rare earth elements (REE), have been extensively used in petrogenetic studies of igneous rocks since they allow the evaluation of the main processes involved in the generation and differentiation of melts. The Neutron Activation Laboratory of IPEN has several collaborative research programs with different Universities (USP, UNICAMP, UFRGS, UnB, UNESP) aiming the determination of trace elements in different kinds of geological matrices by INAA. As part of these collaborations, two gamma-ray spectrometers, one from UNICAMP and the other from USP, were set up in Radiochemistry Division for common utilization. For the determination of REE, U, Th, and other trace elements, the analytical procedure consists of weighing aliquots of approximately 100 mg of the powdered rock sample, and of geological reference materials used as standards, in pre-cleaned polyethylene vials. Samples and reference materials are subjected to a reactor neutron flux of  $10^{12}$  n cm<sup>-2</sup> s<sup>-1</sup> for 8 hours at the nuclear reactor IEA-R1. The measurements of the induced gamma-ray activity are carried out in a GX20190 hyperpure Ge detector, with a resolution of 1.90 keV for the 1332 keV gamma-ray of <sup>60</sup>Co. The gamma-ray spectra are processed by using the program VISPECT, which locates peak positions and calculates gamma-ray energies and net areas.

The Neutron Activation Laboratory of IPEN is participating in the program denominated Quality Assurance of Analytical Laboratories, sponsored by the International Agency of Atomic Energy. As part of this program, the Neutron Activation Laboratory has been participating in a Proficiency Test Program, GEOPT, an international proficiency test for analytical geochemistry laboratories, sponsored by the Department of Earth Sciences, The Open University, UK, which consisted of analysis of different kinds of geological materials. Proficiency testing is becoming increasingly accepted as one of the standard quality control procedures used to help laboratories to highlight unsuspected errors and deficiencies in their analytical methodology and, therefore, improve the quality of their analytical results. The elements Ba, Ce, Co, Cr, Cs, Eu, Hf, La, Lu, Nd, Rb, Sc, Sm, Ta, Tb, Th, U and Yb were analysed by INAA.

The results obtained presented z-score in the range of  $-2 < Z < +2$ , showing the good quality of the data for research in geochemistry. The results demonstrate that the Neutron Activation Laboratory is proficient to perform these analyzes.

An ungraduated student (PIBIC-CNPq) is analyzing Mezozoic dykes from Santa Catarina Island and of the neighboring coastal zone, in collaboration with the Astronomic and Geophysics Institute of USP. The aim of this study is to characterize these dykes from the geochemical point of view and to investigate the mantelic sources involved in the igneous activity as well as crustal contamination processes and in situ differentiation.

## NEUTRON ACTIVATION ANALYSIS APPLIED TO THE CHEMICAL COMPOSITION OF METALLIC MATERIALS

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The chemical composition of metallic materials plays an important role in the technological properties of these materials. Mechanical properties, corrosion resistance, temperability and other properties may be enhanced by the addition of suitable amounts of different elements to the materials. It is also important to control impurities from the manufacturing process, which could influence negatively the characteristics of the material [1]. Many analytical techniques have been used in the study of the chemical composition of metals and their alloys, such as atomic absorption spectrometry, x-ray fluorescence spectroscopy, ICP spectroscopy, ionic chromatography and activation analysis. The instrumental neutron activation analysis technique, INAA, has the advantage, over some of these techniques, to provide precise and accurate results, not only for the major and minor constituents but also for trace elements, without the difficulties arising in the process of sample dissolution and/or element separation prior to analysis.

Contribution to the metallic materials composition determination by INAA was accomplished in the following studies:

1 - Analysis of iron and steel samples by INAA The purpose of this work is the optimization of the INAA technique in the study of the chemical composition of iron and steel samples in the Activation Analysis Laboratory, LAN, at IPEN. To attain this intent, iron and steel certified reference materials, CRM, were analyzed to assess the suitability of the method. Possible interferences in the INAA technique were also investigated, suggesting that there is no need for interference corrections for the elements analyzed. Afterwards, the method was applied to the analysis of industrial iron and steel samples. Applying the comparative method of INAA, As, Co, Cr, Cu, Mn, Mo, Ni, V and W were determined in reference materials and samples after short and long irradiations, according to element half-lives. Induced radioactivities were measured by gamma ray spectrometry.

2 - Analysis of silicon and ferrosilicon reference materials and silicon candidate reference material by INAA Chemical silicon has many industrial applications; for instance, it is used as a precursor in the silicone industry. Control of the chemical and structural characteristics of silicon must be attained as it influences the process of silicone production [2].

IPT Chemical Division set out a new silicon reference material development program as the existing reference materials are old and do not accomplish Brazilian producers present specifications. INAA was used at IPEN Neutron Activation Analysis Laboratory, LAN, in the development of this new material, evaluating possible sample contamination from the grinding machines; and the variation in sample composition due to granulometry. After the new reference material was packed, its intra-bottle and between bottles degrees of homogeneity were accessed by means of INAA of Mn for a suitable amount of bottles, assuring that the CRM is homogenous for its intended use.

The comparative method of INAA was also used in the determination of 21 elements in silicon and ferrosilicon reference materials and in the silicon candidate reference material.