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NEUTRON ACTIVATION ANALYSIS AT CDTN/CNEN USING THE IPR-RI TRIGA MARK I REACTOR

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ABSTRACT

This paper describes in summary the activities developed by the Laboratory for Neutron Activation Analysis since the starting up of the IPR-R1 TRIGA Mark I research reactor in 1960. This Laboratory is located at Centro de Desenvolvimento da Tecnologia Nuclear (Nuclear Technology Development Centre) / Comissão Nacional de Energia Nuclear (Brazilian Commission for Nuclear Energy), CDTN/CNEN. The activities of the Laboratory comprise the delayed fission neutron activation analysis, instrumental (comparative and parametric methods) and radiochemical / chemical methods. These methods are responsible for significant percentage of CDTN's analytical demand, meeting the clients' analytical needs and researches developed by the Laboratory, by CDTN and by other institutions. Over the years the work has been linked to the goals of the country and the institutions. Nowadays the neutron activation analysis is responsible for 70% of the analytical demand and the k_0-Instrumental method for 80% of this demand answering clients' request and researches. In Brazil, CDTN is the only Institute that fully masters the Instrumental Neutron Activation Analysis k_0-method using its own nuclear reactor.

1. INTRODUCTION

The Centro de Desenvolvimento da Tecnologia Nuclear (Nuclear Technology Development Centre) sponsored by Comissão Nacional de Energia Nuclear (Brazilian Commission for Nuclear Energy), CDTN/CNEN is in Belo Horizonte, capital of the Brazilian state of Minas Gerais. At the CDTN, several nuclear analytical techniques are carried out including the neutron activation analysis. The Laboratory for Neutron Activation Analysis has developed its activities since the starting up of the IPR-R1 TRIGA Mark I research reactor in 1960 [1].

At that time the need for the determination of natural uranium had just began in pyrochlore, a niobium ore that would be exported. The x-ray fluorescence and the instrumental neutron activation techniques were then applied to determine the uranium content. The neutron activation technique was applied by means of the delayed fission neutron analysis [2] that was the first method to use the reactor as an analytical tool. At this time started the mineral prospecting of uranium ore in Brazil and practically all the samples, thousand of samples, collected during almost 15 years were analysed by delayed fission neutrons method. In 1989 almost 500,000 uranium determinations were performed. The mineral uranium prospecting associated with thorium occurrence increased the need to determine thorium and the

application of neutron activation analysis by comparative method started. It was opened several opportunities to apply the neutron activation analysis technique.

Nevertheless in the seventies it was needed to determine thorium at low levels, ranging from 0.05 to 20 mg L^{-1} in water and leaching, and 0.05 to 20 μg g^{-1} in residues. The instrumental neutron activation analysis that had been applied to determine other elements, started to be applied [3,4] to ^{232}Th analysis by measuring ^{233}Th, short half-life, and ^{233}Pa, long half-life. Radiochemical procedures were introduced afterwards, in order to reduce the matrix interference. At the same time, the interest in determining thorium in environmental samples increased. The establishment of the Monitoring Environmental Programs co-ordinated by CDTN in nuclear facilities and mining industries diversified the matrixes to be analysed: pasture, air filters, biomaterial and food [5, 6]. In parallel other procedures to determine other elements were also developed and applied as routine methods.

Due to the growing need to determine several elements in a unique sample meeting the clients' analytical needs and researches and following the general analytical tendency, in 1995 the k_0 instrumental neutron activation analysis, k_0-INAA [7], was implanted at CDTN [8,9] after verifying that the TRIGA MARK I IPR-RI reactor presents the suitable characteristics for applying the method, mainly due to stable and homogenous neutron fluxes. The establishment of the method was possible due to the collaboration of Dr. Eduardo H. Montoya Rossi (IPEN-Peru), ARCAL/IAEA Project. This method is called in house k_0-monostandard because instead of using flux monitors uses a comparator. At that time - 1995 - the average thermal and epithermal fluxes were determined for the rotating carousel facility (CF) of the TRIGA reactor as well as an average α (the parameter which measures the epithermal flux deviation from the ideal (1/E) distribution) and an average f (the thermal to epithermal flux ratio) were also determined [8, 9]. Any variations in neutron flux distribution in different channels were not taken into account due to the symmetry of the core configuration and the rotary rack, until the reactor core configuration was changed in 2001 to enable a future increase of the reactor power from 100 to 250 kW. It was decided to rotate the CF only when inserting samples in the irradiation channels. This decision and the need to update the reactor flux distribution in typical irradiation channels and consequently the values of f and α, led the re-establishing of the k_0-method.

In 2003 the k_0-method was completely re-established and improved due to the collaboration of Dr. Radojko Jaćimović through the Expert Mission IAEA (International Atomic Energy Agency) BRA/0/018-01, Project Human Resource Development and Nuclear Technology Support. The acquisition of more suitable programs for spectral evaluation and for element concentration resulted in the improvement of the method and the k_0-standardization method was introduced successfully [10].

Nowadays the techniques available and in routine in the laboratory are: instrumental neutron activation analysis comprising fission delayed neutron analysis, instrumental (comparative and parametric methods - k_0-monostandard and standardisation methods) and radiochemical and/or chemical methods. The matrixes analysed are surface, drinking and groundwater, aqueous and organic effluents, soil, sediment, ore, biomaterial – human tissues and fluids (hair, nails, urine), animal tissues and fluids (muscle, liver, blood, urine) plant (vegetable, pasture, fruit, etc.) - air filters, mud, aqueous and organic leaching, solid and liquid waste,

industrial residue, petroleum, stainless steel, several precipitates, archaeological ceramic, and others.

2. EXPERIMENTAL

The neutron activation analysis (NAA) is a analytical technique well known [11,12]. It consists of exposing a sample to a neutron flux in which the isotopes of chemical elements with suitable cross section to given neutron flux energy is transmuted to a radionuclide through the reaction (n,γ). The irradiation is carried out for a period of time long enough to activate the elements. After suitable decay time, the characteristic radiations emitted from radionuclides formed in this reaction are measured by gamma-spectrometry identifying the chemical elements. It is a multi-elemental technique suitable for the determination of trace elements in several matrices, covering a large range of concentration, with low detection limit, with accuracy and precision. At CDTN/CNEN the irradiation is performed in the TRIGA MARK I IPR-R1 reactor, that is equipped with four facilities for irradiations: the carousel facility, a central thimble and two fast pneumatic transfer systems, one inside the core for determination of elements with radionuclides with short half-lives and the other outside of the carousel to determine uranium by delayed neutron fission analysis. The rotary rack facility (at 100 kW the average thermal flux is 6.6×10^{11} n cm^{-2} s^{-1}, is often used for irradiating the routine samples because it is able to irradiate 40 samples in the lower layer and 40 more in the upper one simultaneously.

At the Laboratory for neutron Activation Analysis, the available methods applying the technique are:

2.1 Instrumental Neutron Activation Analysis

This method is the application of the neutron activation technique without requiring any chemical process neither during preparation nor during the analysis. Due to this characteristic it is called instrumental method. Combining the fact of being a non-destructive process and being a multi-elemental analysis these attributes confer the characteristic of a quasi-ideal analytical technique.

This instrumental method is applied using comparative and parametric procedures comprising gamma-spectrometry - k_0-monostandard and k_0-standardization methods – besides delayed fission neutrons analysis. The application of the methods depend on the kind of matrix, the element to be determined, the range of concentration, matrix interferences and detection limits.

2.1.1 Comparative instrumental neutron activation analysis

In order to apply the comparative instrumental procedure [13], several standards, at least five, of the element or elements to be analysed are included in each group of samples to be studied. The samples and standards are distributed in the carousel facility (CF) and irradiated simultaneously for a length of time needed to activated the elements of interest. The standards and samples gamma-spectrometry is carried out at the same conditions of geometry and the elemental concentrations are calculated by linear regression, based on standards

measurements. The use of this procedure is advantageous when the objective of the analysis is to determine few elements.

2.1.2 Parametric methods - k_0-instrumental neutron activation analysis

The k_0-Instrumental Neutron Activation Method [7] is a "quasi" absolute technique in which instead of standards neutron flux monitors are used and the nuclear data which are unknown are replaced by a compound nuclear constants characterising the nuclides, the so-called k_0 factors. These factors can be experimentally determined with small uncertainties. In addition, k_0-method also requires good knowledge of spectral parameters of the neutron flux in irradiation channels of the reactor. It is applied the k_0 fundamental equation, the Högdahl convention. In this method instead of using standards of the interested element it is used neutron flux monitors.

k_0-monostandard method
At the Laboratory for Neutron Activation Analysis, two versions of the k_0-method is used, one is called in house k_0-monostandard method [8, 9] and the other is the standardisation method [7, 10]. The first one, k_0-monostandard method, is an alternative method and used in some specific situations, for instance, when one is introduced to use the method because it is more didactic for beginners or when the geometry of the samples is not punctual. This method uses sodium as comparator, the values for f and α are average values for the CF, each sample is irradiated in one irradiation channel and all samples and comparators are irradiated simultaneously. For the spectra analysis software as Genie 2K from CANBERRA and Maestro from ORTEC are used and calculations are carried out with Excel. It is because the method uses the basic equation of the k_0-method. Due to several approximations – average values for f and α, comparator standard instead of neutron flux monitor, one simple equation that does not consider other influences like true-coincidence effects, becomes this method not so accurate as the k_0-standardisation.

k_0-standardisation method
Concerning the k_0-standardisation method [7, 10], it is applied irradiating the samples pile in the irradiation vial and intercalated by neutron flux monitor Al-Au (0.1%) IRMM-530RA foil cut into 5 mm diameter and 0.1 mm thick. The irradiation is performed in a irradiation channel in which the values for f and α are determined in this specific channel. For the spectra analysis - peak area evaluation - the HyperLab program [14, 15] is used. For the calculation of elemental concentrations a software package called KAYZERO/SOLCOI [16] is applied when applying the k_0-standardisation. This program takes in account burn-up effects, spectral interferences, correction for reaction interferences, etc. All these corrections become the procedure much more accurate.

For both of them, the usual neutron activation analysis including the gamma spectroscopy, comprises two schemes: 5 min of irradiation time and suitable decay and measurement time to determine elements which radionuclides present short half-lives and 8 hours to determine elements which radionuclides present medium and long half-lives. The gamma-spectroscopy is performed on HPGe detectors with 15% and 50% efficiencies.

2.1.3 Delayed fission neutrons analysis

The Delayed Fission Neutron Activation method [2] applied is a specific method for uranium analysis when the samples are irradiated with thermal neutrons. It is based on a short irradiation followed by measurement of delayed neutrons. Usually an irradiation time of 60 seconds, decay time of 30 seconds and measurement time of 60 seconds are used. A pneumatic transfer system is used for irradiation is located outside the core where the contribution of the thermal neutron flux to the total neutron flux is higher than in carousel facility. After irradiation – fission reaction - the delayed neutrons are counted in a device comprising $^{10}BF_3$ detectors in a moderator of paraffin. The whole procedure is completely automatic analyser with sample changer, pneumatic transfer system, measurement electronics and computer. The system irradiates, measures and calculates the results automatically. The final result is calculated by linear regression.

2.2 Chemical Neutron Activation Analysis

The application of this method involves the chemical separation of the interested element from the original matrix or the separation of the interference elements. The chemical separation may be done by precipitation (separation of thorium as oxalate of thorium from uranium[5]), solvent extraction [17], electrodepositon [18], evolution a gas [19, 20] . After the destructive step of the sample, the interested element is submitted to neutron activation analysis through parametric or comparative methods. In the comparative method the standards of the element under study is submitted to the same steps during chemical procedures.

2.3 Radiochemical Neutron Activation Analysis

In this case, the separation of the interested element from the original matrix or the separation of the interference elements is carried out after the sample be irradiated. In this case only comparative method is used [5]. After the irradiation and the radiochemical steps, the radionuclide is measured by gamma-spectrometry and the linear regression is applied to determine the correspondent elemental concentration.

2.4 Radiochemical and Chemical Neutron Activation Analysis

This analysis [5] involves the chemical separation of the interested element or the interferences, irradiation of the phase where the interested element is and radiochemical separation of the interested radionuclide or interferences.

2.5 Quality Control

In general the quality control is made by using certified reference materials. Standards of the elements under analysis and spike samples as well as replicate of the samples are usual procedure to ensure the results.

Participating in intercomparison programs organized by IRD/CNEN, Radioprotection and Dosimetry Institute, Brazil (Instituto de Dosimetria e Radioproteção) determining thorium and uranium, intercomparison rounds organised by the IAEA [21] during the development of co-ordinated research projects determining several elements as well as other institutions has

been an essential procedure to quality control. The successful results obtained during the Intercomparison Exercises organised by IAEA, ARCAL XXVI (RLA/4/013), IAEA – Quality Assurance in Analytical Laboratories, 1997-2000, were responsible for the level promotion in conformity with the requirements of the ISO/IEC 17025:1999 norms.

3. DISCUSSION

Several Projects have been developed since 70's determining chemical elements by neutron activation analysis in diversified fields of science as medicine, geology, environment, health and nutrition, chemistry, biology, etc.

Some examples are given with more relevant references: Nuclear method applied on studies of foodstuff [22, 23, 24, 25]; Study about medicinal plants used on treatment of diarrhoea of children [26]; Studying of biochemical composition of milk from diabetic mothers (in Portuguese) [27]; Evaluation of nutrition status of children and adolescents [28, 29]; Workplace and occupational health: the first metal evaluation using nuclear and analytical techniques in the State of Minas Gerais – Brazil" IAEA BRA-11920 [30, 31, 32, 33]; Supporting other analytical techniques [34, 35]; Determination of chemical contaminants in medicines [36, 37]; On tribes and chiefdoms: research upon socio-economic organisation in South American lowlands [37, 38, 39]; Determination of several elements in environmental samples [40, 41]; Study about absorption of arsenic by plants aiming at decontaminating of soil in the Iron Quadrangle, Minas Gerais, Brazil [42], etc.

4. CONCLUSIONS

Over the years the work carried out has been linked to the goals of the country and the institutions, answering the clients' analytical needs and researches developed by the Laboratory, by CDTN and by other institutions, offering results with quality control [42]. At the beginning the major application of nuclear analytical techniques was related to the field of geochemistry and mineral exploration. All the uranium determinations related to Brazilian mineral prospecting were executed in the Laboratory for Neutron Activation Analysis mainly through delayed neutron fission analysis.

Nowadays the major application is in the field of medical and environmental measurements, determining several elements in a large range of concentration in several matrixes such as biomaterials (food, plants, yeast, human and animal tissues, medicines, etc.) environmental samples (soil, sediment, water, liquid effluent, airborne particulate matter, etc.) industrial products (alloys plastic, etc.).

The newest method established, the k_0-Instrumental Neutron Activation Analysis has been responsible for 90% of the analytical demand of the Laboratory for neutron activation analysis. In 2007 over 1000 samples were analysed producing around 30000 determinations, answering clients' request and researches. In Brazil, CDTN is the only Institute that fully masters the Instrumental Neutron Activation Analysis k_0-method using its own nuclear reactor.

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