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k_0-Instrumental Neutron Activation Analysis Establishment at CDTN, Brazil: A successful story

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Since 1995 the k_0 instrumental neutron activation analysis has been applied at Radiochemical Laboratory, CDTN/CNEN, Brazil, by means of TRIGA MARK I IPR-R1 research reactor. At that time α, f, and T_n were determined and the most recent determination of these parameters confirmed the great stability of the reactor along these years. In order to verify the efficiency and accuracy of the method, several certified reference materials have been systematically analyzed. Participating in Intercomparison Exercises organized by IAEA has been an important, essential and useful procedure to quality control. CDTN is the only Brazilian Institute to apply the k_0-INAA to determine elements by means of their isotopes through short, medium and long half life using its own nuclear reactor.

Introduction

Since the starting up of the TRIGA MARK I IPR-R1 research reactor in 1960,[1] the Laboratório de Radioquímica (Radiochemical Laboratory) has developed its activities. This Laboratory is in CDTN, Centro de Desenvolvimento da Tecnologia Nuclear (Nuclear Technology Development Centre) sponsored by CNEN, Comissão Nacional de Energia Nuclear (National Commission of Nuclear Energy), located in Belo Horizonte, capital of Minas Gerais, a Brazilian state.

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) was implanted at CDTN.[2–5] It was possible due to the collaboration of Dr. Eduardo H. MONTOYA ROSSI (IPEN-Peru), ARCAL/IAEA Project.

The techniques available and in routine in the laboratory are: delayed neutron activation analysis, conventional (instrumental and radiochemical) and k_0 instrumental neutron activation analysis, fluorimetry, gross alpha and gross beta counting, alpha- and gamma-spectrometry, liquid scintillation and radiometry with radiochemical separations. Over the years the work carried out has been linked to the goals of the country and the institutions. 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. The k_0-INAA method has been responsible for 90% of the all neutron activation analysis demand.

Determination of the parameters

The reactor TRIGA MARK I IPR-RI is equipped with three facilities for irradiation: the rotary specimen rack outside the core, a central thimble and a pneumatic tube transfer system. The rotary rack facility at 100 kW is often used for irradiating routine samples because it is able to irradiate 40 samples in the lower layer and 40 more in the upper one simultaneously. At that time – 1995 – the parameters[2–5] were determined for the rotary rack, lower layer: α was calculated applying the "three bare monitor" method using ^{198}Au, ^{95}Zr and ^{97}Zr; f determination was done according to the bare bi-isotopic method; the neutron temperature was calculated through the direct method using ^{177}Lu, ^{95}Zr, ^{97}Zr, and ^{198}Au and the WESCOTT's $g(T_n)$ function for ^{177}Lu was calculated and the result was interpolated in the GRYNTAKIS[6] table determining the neutron temperature. It was concluded that the nuclear reactor presented characteristics in the rotary specimen rack, lower layer, adequate for the k_0 application. The parameters determined[2] were: $\alpha = 0.025 \pm 0.002$; $f = 24.2 \pm 0.2$; $T_n = (30.8 \pm 0.1)$ °C; $\Phi t = (6.0 \pm 0.5) \cdot 10^{11}$ n·cm^{-2}·s^{-1}; $\Phi e = (2.5 \pm 0.5) \cdot 10^{10}$ n·cm^{-2}·s^{-1}; and $r(T_n/T_0)^{1/2} = 0.0345 \pm 0.0002$.[2]

From time to time those parameters are checked to detect some variability. The most recent results confirmed all the values that have been determined along the years since 1995. The parameters were also determined in the upper layer in the carousel facility and in the central thimble.

The procedure to check the parameters consisted in using standard solutions of Au (metal ware high purity gold – SRM 685 National Bureau of Standards – NBS),

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Lu (LuO_2, Johnson Mattey Company – JMC) and Zr (metal, 98.14% pure, JMC). Aliquots from each standard solution was pipetted (Eppendorf Pipette, calibrated, 0.01% precision) into the irradiation tube and the solution was dried slowly. After adequate irradiation and decay time, the samples were counted. The counting time was statistically suitable in order to ensure reliable results, on a coaxial HPGe detector (efficiency of 15%, resolution of 1.85 keV for the 1332 keV peak of ^{60}Co) at 20 cm distance from the end cap. The spectra obtained were analyzed using the software MAESTRO, ORTEC.

Experiment 1: aimed at confirming the thermal neutron flux homogeneity in the rotary rack. In this rack each five symmetrical positions, total of eight, an irradiation tube was inserted with Au, 25 μg and the irradiation time was 15 minutes. This experiment was performed on the rotating rack then on the motionless rack. Calculations based on Au specific activity pointed out that the neutron flux is homogenous presenting only 2% variability in the different positions on the rotating rack is and 3% when motionless.

Experiments 2, 3 and 4: aimed at determining the parameters. In the same positions of Experiment 1, lower layer, the irradiation tubes were inserted each one with Au, Lu and Zr varying the concentrations in each experiment. The irradiation time was 4 hours and the experiment was performed once with the rotating rack and the other stationary.

Experiment 5: the objective was to determine the parameters in the upper layer in the carousel facility. The experiments were carried out following the same procedure described above, but on a rotating carousel.

Experiments 6 and 7: the objectives were to determine the parameters in central thimble, irradiating also Au, Lu and Zr varying the concentrations and an irradiation time of 1 hour.

Experiments 8 and 9: the goals were to improve statistically the gamma-counting. They were performed the experiments as above, using the same Au, Lu and Zr concentrations but varying the irradiation time for 2.5 hours.

The parameters determined as well as the thermal and epithermal fluxes in the carousel facility and central thimble are in Table 1. Table 2 shows the concentrations calculated for GXR-2 Reference Material, irradiated in rotatory rack, lower layer, and in central thimble. The k_0-INAA was applied using the parameters recently determined.

Table 1. Parameters determined for the reactor TRIGA MARK I IPR-RI

Irradiation facility		α	f (ϕ_t/ϕ_e)	Cadmium ratio (R_{Cd})	Thermal neutron flux (ϕ_t), $n \cdot cm^{-2} \cdot s^{-1}$	Epithermal neutron flux (ϕ_e), $n \cdot cm^{-2} \cdot s^{-1}$	Spectral indices $r(T_n/T_0)^{1/2}$
Determination in 1995							
Rotary rack lower layer – R		0.03 ± 0.02	24.2 ± 0.2	2.6 ± 0.1	$(6.0 \pm 0.5) \cdot 10^{11}$	$(2.5 \pm 0.5) \cdot 10^{10}$	0.035 ± 0.002
Latest determination in 2001							
Rotary rack lower layer	R	0.04 ± 0.01	24 ± 2	2.6 ± 0.1	$(6 \pm 1) \cdot 10^{11}$	$(2.5 \pm 0.3) \cdot 10^{10}$	0.036 ± 0.002
	S	0.03 ± 0.02	23 ± 4	2.5 ± 0.3	$(6 \pm 1) \cdot 10^{11}$	$(2.6 \pm 0.4) \cdot 10^{10}$	0.04 ± 0.01
Rotary rack upper layer - R		0.100 ± 0.005	20 ± 1	2.6 ± 0 1	$(4.3 \pm 0.1) \cdot 10^{11}$	$(2.2 \pm 0.1) \cdot 10^{10}$	0.04 ± 0.01
Central Thimble		–0.02 ± 0.01	17 ± 4	2.1 ± 0.2	$(2.9 \pm 0.7) \cdot 10^{12}$	$(1.6 \pm 0.4) \cdot 10^{11}$	0.05 ± 0.01

R: Rotating.
S: Stationary.

Table 2. Experimental and certified elemental concentrations (in $\mu g \cdot g^{-1}$) determined for GXR-2 Reference Material

	Rotatory rack	Central thimble	Certified value
Al	190000 ± 13000	NA	185700 ± 600
Ba	2100 ± 200	NA	2240 ± 60
Br	3.7 ± 0.4	ND	3.20 i
Co	8 ± 1	8 ± 4	8.6 ± 0.3
Cr	38 ± 4	38 ± 4	36 ± 4
Cs	5.1 ± 0.4	6 ± 1	5.2 ± 0.3
Eu	0.9 ± 0.1	ND	0.81 ± 0.10
Fe	18100 ± 700	20500 ± 2100	18600 ± 600
Hf	8 ± 1	ND	8.3 ± 0.9
La	26 ± 1	32 ± 4	26.6 ± 1.8
Mn	1060 ± 70	NA	1010 ± 40
Na	5500 ± 400	6700 ± 700	5600 ± 110
Sb	44 ± 4	ND	49 ± 5
Sc	8 ± 1	8 ± 1	6.88 ± 0.09
Ta	0.7 ± 0.1	1.1 ± 0.2	0.90 ± 0.17
Ti	3500 ± 400	NA	3020 ± 240
Th	9 ± 1	ND	8.8 ± 0.3
V	53 ± 4	NA	52 ± 4
Yb	1.7 ± 0.2	2.2 ± 0.2	2.04 ± 0.24

NA: Not analyzed.
ND: Not detected.
i: Information values.

Applications

The k_0-INAA in lower layer rotatory rack has been applied using Na as comparator. Besides, each irradiation is followed by at least one certified reference material analyzed in replicate in order to verify the accuracy. The neutron activation and gamma-spectroscopy comprise, in general, three schemes for irradiations performed in the rotary rack. For upper layer the irradiation time should be 30% more. The schemes are: 5 minutes of irradiation time, 2 to 15 minutes of decay time and 10 minutes of measuring time to determine Al, Ba, Cl, Cu, I, Mn, Ti and V; 4 hours of irradiation time; 12 hours of decay time and 3 hours of measuring time to determine As, Au, Br, Ga, K, La, Na and Sn; 20 hours of irradiation time, 10 days of decay time and 4 hours of measuring time to determine Ag, Co, Cr, Cs, Fe, Hg, Hf, Rb, Sb, Sc, Se, Th, U, Zn and rare-earths.

Several certified reference materials have been analyzed by means of k_0-INAA in order to verify the efficiency of the method using the TRIGA MARK I IPR-R1' s parameters. Some of the certified reference materials analyzed were: IAEA/Soil-7 (International Atomic Energy Agency), GBW 08303 - Polluted Farmland Soil (Beijing Municipal Environmental Monitoring Centre), BCR-176-Trace Element in a City Waste Incineration Ash (Community Bureau of Reference), GXR-3, GXR-6 (United States Geological Survey) and GBW 09101-Human Hair (Shanghai Institute of Nuclear Research). Table 3 summarizes some results for the certified reference materials. The overall accuracy was (1–5)%.[7]

The application of the k_0-INAA has been successfully used in several projects, analyzing biomaterials as bovine blood, liver and urine,[8] food[9] and others as well samples as soil, sediment, pasture, from Environmental Monitoring Programmes developed by CDTN.

The most recent projects that are being carried out are:

IAEA's CRP The Use of ^{99m}Tc as an Absorbable Tracer for Studying the Dynamics of Fine Sediments in Suspension, BRA-10891. This project studies the suspended sediment behavior, emphasising the study of individual discharges of contaminants associated to such sediments. The project focuses on the Pampulha Lake, reservoir in Belo Horizonte city, contaminated by heavy metals. The main matrixes analyzed are water and sediment.[10,11]

IAEA's CRP Nuclear Analytical Techniques in *Archaeological Investigations – Tribes and Chiefdoms: An Analytical Study of Some Brazilian Ceramics, BRA 9395*. This project has been conducted together with Museu Emílio Goeldi, Pará, Museu de Arqueologia da UFMG (Archaeological Museum of Federal University of Minas Gerais) and UFGO Federal University of Goiás). It aims at determining the elemental concentration in ceramics and clay and then investigate the sources of the material and confirm or not the archaeological hypotheses. The matrixes analyzed are clay and ceramic.[12]

Table 3. Experimental and certified elemental concentrations (in $\mu g \cdot g^{-1}$) for several reference materials

Element		BCR 176	GBW 08303	GBW 09101 human hair	GXR-1	GXR-6	Soil-7
Al	E	ND	73,000 ± 800	14.7 ± 7	ND	ND	52,000 ± 700
	C	NI	68,600 ± 3,400	13.3 ± 2.3	NI	NI	NI
As	E	ND	ND	ND	400 ± 40	290 ± 30	16 ± 4
	C	NI	NI	NI	427 ± 45	330 ± 25	13.4 ± 0.8
Au	E	ND	ND	ND	5 ± 1	0.12 ± 0.01	ND
	C	2.30 ± 0.02	NI	NI	3.3 ± 0.3	0.095 ± 0.014	–
Co	E	29.5 ± 0.4	11 ± 1	ND	7.3 ± 0.5	14.8 ± 0.5	8.6 ± 0.5
	C	30.9 ± 1.3 i	13.0 ± 1.2	0.135 ± 0.008	8.2 ± 1.5	13.8 ± 1	8.9 ±
Cr	E	ND	110 ± 1	4.6 ± 0.6	ND	NI	70 ± 1
	C	840 ± 4	112 ± 12	4.77 ± 0.38	NI	NI	60 ± 14
Cs	E	ND	6.5 ± 0.1	ND	2.4 ± 0.2	4.4 ± 0.4	6.1 ± 0.6
	C	8 ± 0.2	NI	NI	3.0 ± 0.6	4.2 ± 0.21	5.4 ± 1
Cu	E	ND	ND	19 ± 2	1390 ± 150	ND	<10
	C	NI	NI	23 ± 1.4	1110 ± 115	66 ± 8	11 ± 2
Fe	E	20,800 ± 300	27,700 ± 100	ND	270,000 ± 20,000	60,000 ± 5,000	29,000 ± 3,000
	C	21,300 ± 1100	29,700 ± 2,000	71.2 ± 6.6	250,000 ± 12,000	55,800 ± 4,100	25,700 ± 5,000
Ga	E	ND	ND	ND	16 ± 1	42 ± 4	7.6 ± 0.8
	C	NI	NI	NI	13.9 ± 1.6	35 ± 3	10 ± 3
La	E	ND	40 ± 10	ND	10 ± 1	13 ± 1	26 ± 2
	C	NI	40 i	0.014 i	7.5 ± 0.8	13.9 ± 0.9	28 ± 1
Mn	E	ND	590 ± 10	4 ± 2	860 ± 70	1020 ± 40	680 ± 60
	C	NI	519 ± 36	2.94 ± 0.20	880 ± 70	1040 ± 50	631 ± 30
Na	E	29,600 ± 300	11,800 ± 200	270 ± 50	ND	NI	2,300 ± 20
	C	NI	11,000 ± 1,2000	266 ± 12	NI	NI	NI
Rb	E	ND	80 ± 2	ND	25 ± 3	100 ± 10	61 ± 6
	C	151 ± 1	68 i	NI	14 ± 9	90 ± 4	51 ± 5
Sb	E	430 ± 4	40 ± 1	ND	110 ± 10	6 ± 2	1.9 ± 0.1
	C	412 ± 18	NI	0.21 i	122 ± 18	3.6 ± 1	1.6 ± 0.2
Sc	E	ND	10.2 ± 0.02	ND	1.8 ± 0.2	24.8 ± 0.4	9.4 ± 0.9
	C	2.7 ± 0.03	10 i	NI	1.58 ± 0.2	27.6 ± 2.6	8.3 ± 0.7
Ti	E	ND	2,700 ± 300	ND	ND	5,100 ± 200	2,600 ± 300
	C	NI	3,600 ± 200	NI	380 ± 190	5,000 ± 100	3,000 ± 400
V	E	ND	80 ± 10	ND	95 ± 9	200 ± 20	60 ± 6
	C	NI	NI	0.069 i	80± 10	186 ± 11	66 ± 7
Zn	E	ND	ND	160 ± 10	ND	ND	ND
	C	NI	NI	189± 8	NI	NI	NI

E: Experimental values.
C: Certified values.
ND: Not detected.
i: Information values.
NI: Not informed.

IAEA's CRP Assessment of Levels and Health-Effects of Airborne Particulate Matter in Mining, Metal Refining and Metal Working Industries Using Nuclear and Related Analytical Techniques, BRA 9473 – Workplace and Occupational Health: The First Metal Evaluation Using Nuclear and Analytical Techniques in the State of Minas Gerais – Brazil. This project has been conducted together with the physicians of the Secretaria Municipal de Saúde (Municipal Department of Health) and researchers from Fundação Ezequiel Dias, an official institute involved with public health. It is inserted in a Worker's Health Awareness Program. The goals were to assess metal levels in a galvanizing industry by means of biomonitors and airborne particulate matter collected in air filters.[7,13] The matrixes analyzed are hair, toenails and air filters.

Ethnobotanic, morphoanatomic and chemical study of medicinal plants popularly used as diuretic, project conducted together CDTN and Pontifícia Universidade Católica (Catholic University). The objectives involve the study of the efficiency of medicinal plants used as diuretic and the publication of a guide on use, preparation and dosage. The samples analyzed are plants most commonly used as diuretic.

Characterization of trace elements in topaz by means of neutron activation technique. This project is conducted by CDTN and UFOP, Ouro Preto University. The goal is to study geochemically those elements. The samples analyzed are semi-precious stones.

Intercomparison exercises

Participating in intercomparison studies is a well known way to verify the quality of all procedures involved in the determination of elemental concentration. To participate in relevant Intercomparison Exercises organized by IAEA has been an important, essential and useful procedure to quality control. The k_0-INAA installed at Laboratório de Radioquímica has been applied in such studies in diversified matrixes. For instance, an intercomparison study in urban dust was carried out as an activity planned during the IAEA's CRP Assessment of levels and health-effects of airborne particulate matter in mining, metal refining and metal working industries using nuclear and related analytical techniques in 1999.[14] The intercomparison samples were analyzed by 39 laboratories in 31 countries by means of several analytical techniques such as neutron activation analysis (relative and k_0-standardization), particle induced X-ray emission analysis (PIXE), X-ray fluorescence analysis (XRF) and atomic absorption spectrometry (AAS). The results are in Table 4. During the ARCAL XXVI – Acuerdos Regionales do Cooperación para a America Latina – (RLA/4/013), IAEA – Quality Assurance in Analytical Laboratories, 1997–2000, intercomparison exercises were carried out and the results of one of them are in Table 5. This exercise involved 8 laboratories from 7 countries.

Table 4. Intercomparison results: elemental concentrations (in mg·kg^{-1})

Element	Urban dust	
	Laboratório de Radioquímica results	Intercomparison results*
Al	39600 ± 400	36290 ± 2755
Au	0.52 ± 0.01	0.412 ± 0.002
Cl	2700 ± 200	2874 ± 598
Co	14.50 ± 0.30	14.52 ± 1.25
Cr	258 ± 3	236 ± 21
Cs	2.1 ± 0.2	3.75 ± 0.78
Cu	720 ± 200	768 ± 134
Fe	41800 ± 300	40698 ± 2804
K	12900 ± 200	12318 ± 1034
La	21 ± 10	18.15 ± 1.76
Mn	900 ± 10	808 ± 84
Na	6890 ± 90	6460 ± 316
Rb	57 ± 6	56 ± 6
Sc	6.07 ± 0.04	5.42 ± 0.48
Ta	0.6 ± 0.2	0.445 ± 0.00
V	94 ± 5	94.27 ± 13.5

* Mean of 39 laboratories results.

Table 5. ARCAL XXVI – Intercomparison results: elemental concentration (in μg·g^{-1}) river sediment

Element	CDTN' s Results*	Intercomparison results (8 laboratories)
Al	61000 ± 5000	44800 ± 19800
As	5 ± 1	7.3 ± 2.6
Co	16 ± 2	15.9 ± 1.6
Cr	653 ± 50	622 ± 53
Cs	5.9 ± 0.5	5.04 ± 1.03
Cu	63 ± 5	65.8 ± 10.8
Fe	33 ± 3	34.0 ± 4.9
K	18.6 ± 0.2	18.6 ± 0.2
La	30 ± 2	27.4 ± 1.8
Mn	854 ± 75	801 ± 58
Na	12.1 ± 0.3	10.9 ± 0.6
Rb	110 ± 15	99 ± 20
Sc	11.4 ± 0.1	11.9 ± 0.1
Ta	0.9 ± 0.2	0.91 ± 0.17
V	84 ± 8	75 ± 22

* Mean of 5 determinations.

Discussion and conclusions

The reactor TRIGA MARK I IPR-RI presents the suitable characteristics for applying the k_0-INAA, mainly due to stable and homogenous neutron fluxes. These characteristics are reflected in the parameters used in k_0 method. These parameters values have been confirmed since 1995 according to the most recent experiments.

In spite of being not significant the difference between the fluxed determined during rotatory rack rotating and stationary, it is recommended to perform the irradiation rotating the carousel facility to minimize the flux fluctuations influence on the positions in the rack.

The concentrations determined for several elements in the GXR-2 Reference Material irradiated in the rotatory rack and in the central thimble, present good

agreement with certified results. Some elements were not determined in the central tube. These elements were those that should be determined through short half life. Al, Ba, Mn, Ti and V, but due to inadequate facility and high activity it was not possible. Others, Br, Eu, Hf, Th, Rb and Sb, were not determined either because of inherent interference.

The Intercomparison Exercises results confirm not only how powerful the neutron activation technique is as well as the potential of the k_0-method as an analytical multi-elemental determination tool. Participating in relevant Intercomparison Exercises organised by IAEA has been an essential procedure to quality control. The successful results obtained during the 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.

Up till now more than 3,000 samples were analyzed applying the k_0-INAA. Nowadays the k_0-standardization method has been responsible for 80% of the analytical demand answering clients' request and researches.

In Brazil, the CDTN is the only Institute that fully master the use of the k_0-INAA and the nuclear reactor.

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