APPLICATIONS OF NEUTRON ACTIVATION ANALYSIS TECHNIQUE IN ELEMENT DETERMINATION AT TRACE LEVEL

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Abstract: *Analytical applications of nuclear technique instrumental neutron activation analysis (INAA) for trace element characterization of solid materials are reviewed. Results for the analysis of samples from the technological flux of an integrated iron and steel plant and geological materials are presented and aspects concerning the limit of detection, matrix effects, spectral and reaction interferences and its complementarity with other micro-analytical nuclear techniques are highlighted.*

Keywords: *neutron activation analysis, trace elements, industrial and geological samples.*

1. Introduction

Knowledge of trace element content of industrial and environmental materials is important for company managers due to the fact that the presence of these elements influences the environment status and manufacture processes in industry [1-5] and information should be gathered in monitoring the environment changes, in identifying and analyzing problems, in designing and developing solutions, in testing and improving materials [6]. The determination of trace elemental levels in different matrices is a challenge for the entire spectrum of analytical methods, covering both nuclear and non-nuclear analytical methods. Many analytical techniques have been developed to investigate the chemical constituents of complex materials and great attention has been paid to nuclear (NATs) and related analytical techniques which have the advantage of being multi-elemental and very sensitive for a large range of chemical elements [6-13]. We define NATs [6] as those techniques that use nuclear reactions, radioactive decay, or nuclear instrumentation to investigate properties of matter. This definition extends from the well-established radiochemistry, neutron activation analysis (NAA) and prompt-gamma activation analysis (PGAA), to advanced methods at the limits of science and technology, and includes the applications of these techniques to the determination of composition and structure of matter for science and technology [6,12,14].

Among these techniques, instrumental neutron activation analysis (INAA) using both reactor (thermal, epithermal) and fast neutrons has been widely used in industrial and research laboratories for the analysis of a large variety of materials – raw and auxiliary materials, final products, by-products [1-5,10,12,15], environmental [7,13,14,16-19], biological [14,17,20-24], nano-materials [8,9,14], and reference materials for quality assurance/ quality control (QA/QC) [14], being a valuable tool in biomonitoring of atmospheric deposition of trace elements including heavy metals, lantanides and actinides, quality assessment of foodstuffs, biotechnology of new pharmaceuticals, monitoring of environment and human health [14,16,17,21,22].

Thermal instrumental neutron activation analysis (INAA) takes advantage of the high intensity of neutron beams available from the thermalization of fission neutrons and the large thermal neutron cross sections for most isotopes [14].

Analysis of biological and environmental samples is currently carried out using not only classical methods of analytical chemistry, but also nuclear and related physics techniques, such as:

atomic absorption spectrometry (AAS), X-ray fluorescence (XRF), inductively coupled plasma-atomic emission spectrometry (ICP-AES), inductively coupled plasma-mass spectrometry (ICP-MS), synchrotron radiation, neutron activation analysis (NAA), etc. Nuclear and related techniques are found to be most sensitive. The NAA technique is particularly effective in solving environmental problems where multielement analysis of a great deal of samples is required [16].

In this work are presented some applications of INAA in materials science, environment and geology for the determination of minor and trace elements in solid materials.

2. Experimental methods

INAA using reactor neutrons was applied in several irradiation steps at "Horia Hulubei" Institute of Physics and Nuclear Engineering (IFIN-HH) Bucharest-Magurele, Romania, and at Frank Laboratory of Neutron Physics (FLNP) of Joint Institute for Nuclear Research (JINR), Dubna, Russia. The experimental procedure is described in previous papers [1-4,14,16] and monographs [1,11-13].

Fast (14 MeV) neutron INAA using neutron beams from industrial neutron generators was employed at Nuclear Unit of Iron and Steel Works (ISW) of Galati, Romania [5,15,25].

If a sample contains the stable nuclide ${}^{A}_{Z}X$, after the (n,γ) neutron capture reaction a radioactive nucleus ${}^{A+1}_{Z}X$ will result, which is β^- and γ -ray emitter [11-13], as following: ${}^{A}_{Z}X + {}^{1}_{0}n \rightarrow {}^{A+1}_{Z}X^* + \gamma$; ${}^{A+1}_{Z}X^* \rightarrow {}^{A+1}_{Z+1}Y + {}^{0}_{-1}\beta$.

After activation with a thermal neutron flux, the resulting radioactive nuclei emit β^- and γ radiations. The identification of radionuclides in a complex mixture is done using the decay properties of each nucleus, i.e. the gamma-ray energies (E_γ) and the intensities (I_γ) of the emitted radiations, and the half-life $T_{1/2}$ of the induced radionuclide [13].

The neutron activation cross-section as a function of energy shows great variation among different stable nuclides. In the thermal neutron region the activation cross-section of most nuclides follows the 1/*v* law (inversely proportional to the neutron velocity). Some nuclides continue to follow the 1/*v* law in the epithermal region, while other show strong resonances in their excitation function in that region [13,16]. Therefore, the ratio of thermal to epithermal activation shows large variation between different target nuclides, a fact which may be conveniently illustrated by looking at the ratio of resonance activation integral/thermal neutron cross-section (I/σ_0) for the nuclides concerned [16]. According to Westcott convention, the effective activation cross-section, σ, is expressed by [11-13]:

$$\sigma = \sigma_0 \left(g + K \frac{I - 0{,}45\,\sigma_0}{\sigma_0} \right)$$

where *g* is a factor dependent on the deviation of activation cross-section from *1/v* law (*g* = 1 for an activation cross-section following the *1/v* law); *K* – is the spectral index, defined as the ratio of epithermal and thermal neutron flux ($K = \phi_{epi}/\phi_{th}$), which is a constant for a defined irradiation position in a reactor operating steadily.

The sensitivity of analysis depends on the factor $\sigma.a/A$, where *a* is the natural abundance of the stable (target) nuclide and *A* the nucleus mass [1], as well as on the Compton background in the spectra [1,3,14,16]. Also, matrix effects – nuclear and spectral interferences – must be known and the results corrected for [3,14]. For the long - lived nuclides counting could be carried out at different decay times, in order to best detect radio nuclides of various half lives [2,3].

3. Results and discussion

The investigated metallurgical samples have been taken from the technological flux of ISW Galati, Romania, and irradiated in a thermal neutron flux at IFIN-HH Bucharest (*K* =31). Application of INAA using thermal neutrons for the investigation of micro-composition of metallurgical samples allowed the simultaneously determination of more than 30 chemical elements in industrial samples [1-4]. The minor and trace elements determined in the iron matrix of the steel samples were the following: Mn, Al, V, As, Cu, W, Ni, Mo, Cr, Sb, Co, Na, K, Ce, La, Sm, Sc, Zr, Zn, Au, Ga, Hf, Ta, Te, Se, Ba, Rb, Yb, Tb, Ca and Ir.

Knowing the concentrations of the residual elements in the final steel, the steelmaking process in LD converter can be conducted so that the recuperation of the elements with favorable influence upon steel characteristics (Mn, Al, V, Cr, Ni, Mo, W, Ta, Te, Zr, Co, La, Ce, Sm) should be accomplished [2,3].

As regards the undesirable elements for steel (As, Cu), they are found in the deoxidized steel, probably being transferred from the raw materials (iron ores) to final steels. Taking into account this fact, it is very important to choose the appropriate proportions of iron ores which contain these elements in high concentrations.

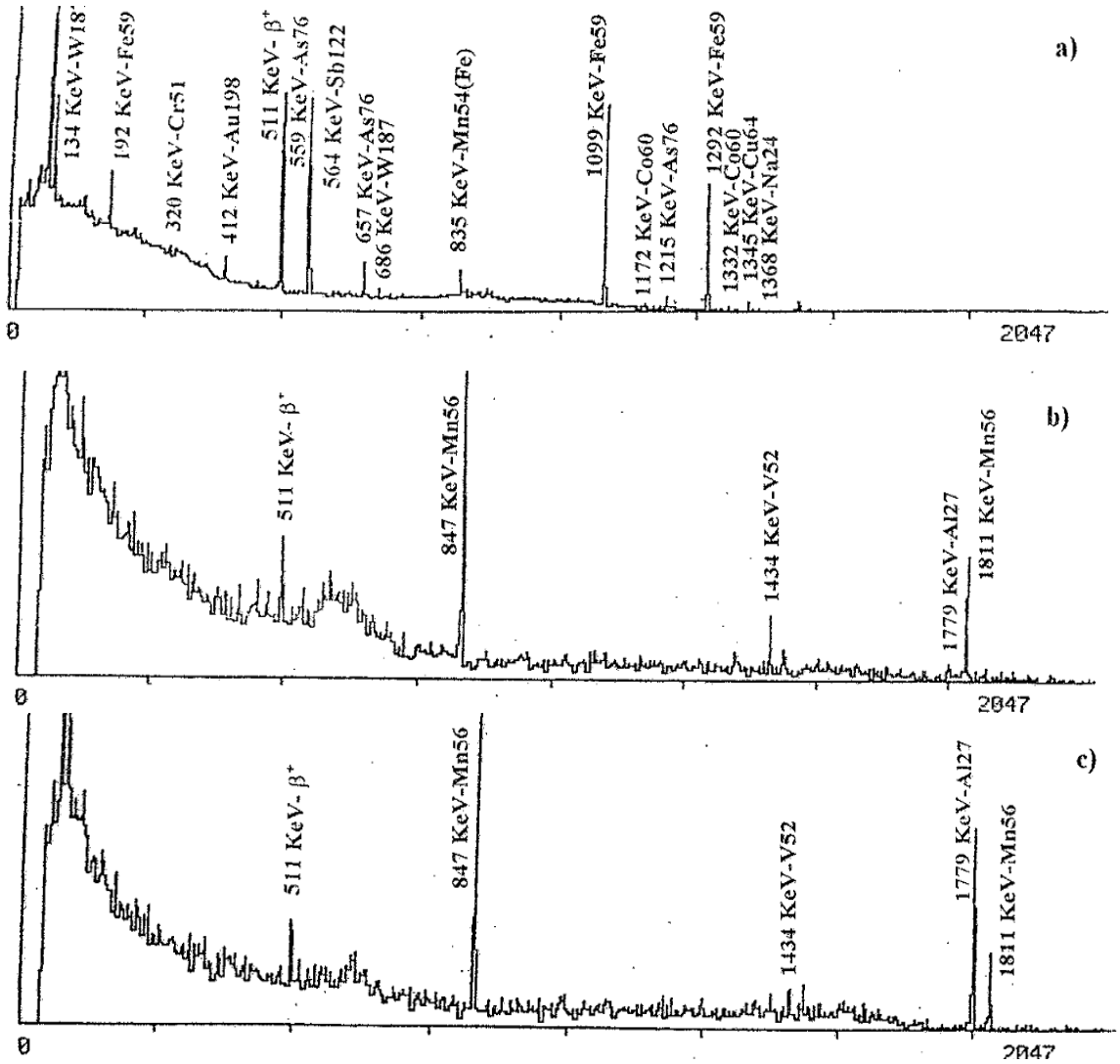

Figure 1: *Gamma rays NAA spectra of a steel sample obtained at IFIN-HH for long (a) and short (b) irradiation and spectrum of an iron ore for short irradiation (c).*

Gamma-ray spectra obtained for some steel and iron ore samples by the application of reactor INAA are presented in **Fig. 1**. The identification of chemical elements in the samples is based on the energies corresponding to the gamma-ray peaks in the spectra presented in **Table 1**.

Several papers [6,14,16,17] reviewed the contribution of reactor neutron activation analysis at FLNP-JINR, Dubna, Russia, to solution of the major problems in the studies of the chemical composition of objects in the biosphere with a view to understanding the role of various elements in the functioning of living organisms and ecosystems under anthropogenic effects on nature, often resulting in irreversible changes in the environment and human health. In spite of competing non-nuclear analytical techniques (AAS, ICP-AES, ICP-MS, etc.), the reactor NAA as a primary (ratio) method continues to be the most powerful multi-element analytical technique providing quantification of trace elements at ultra low levels. Combined with modern statistical data treatment of large arrays of data, GIS (geographic information system) technologies, electron scanning microscopy, tomography, and others, NAA serves to obtain practical results [16].

Table 1: *Nuclear data of isotopes which could be present in metallurgical materials and energies, in keV, for the emitted gamma rays, resulted from (n, γ) reactions (completion of data presented in [1])*

TARGET NUCLEUS			Z - X - A+1 RADIONUCLIDE				
Z-**X**-A	a (%)	A	$T_{1/2}$	σ_0 (b)	I (b)	γ-ray energy (E_γ), in keV (intensity I_γ,%)	σ.a/A
25- Mn-55	100	54.938	2.58 h	13.3	13.8	847(99); 1811(27)	0.242
13-Al-27	100	26.9815	2.246 h	0.232	0.175	1779(100)	0.0086
23-V-51	99.75	50.9414	3.755 m	4.93	2.6	1434.2(100)	0.096
22-Ti-50	5.3	47.9	5.76 m	0.179	0.12	320(93); 609(1); 929(7)	0.0002
28-Ni-64	0.95	58.71	2.52 h	1.58	1.19	366.5(4.8); 1115.5(15.2); 1481.7(23)	0.00025
42-Mo-100	9.63	95.84	14.6 m	0.199	4.2	191.9(18.1); 505.9(14.35); 590.8(19.3)	0.002
12-Mg-26	11.01	24.305	9.46 m	0.035	0.027	843.8(72); 1014.4(28)	0.00016
26-Fe-58	0.31	55.847	44.6 d	1.28	1.4	142.4(0.81); 192(2.8); 1099.3(56); 1291.6(44)	0.00007
11-Na-23	100	22.9898	15.03 h	0.53	0.32	1368.6(100); 2754.1(100)	0.023
19-K -41	6.7	39.102	12.36 h	1.46	1.4	1527.7(18)	0.0025
24-Cr-50	4.35	51.996	27.7 d	15.9	8.1	320(10)	0.0133
29-Cu-63	69.1	63.546	12.74 h	4.5	4.94	511(37); 1345.8(0.5)	0.049
33-As-75	100	74.9216	26.4 h	4.48	61	559.1(44.6); 657(6.4); 1216.3(3.7)	0.059
74-W -186	28.4	183.85	23.9 h	37	490	134.2(10.1); 478.5(26.6); 685.7(32)	0.057
27-Co-59	100	58.9332	5.272 y	37.45	71.1	1173.2(99.9); 1332.5(100)	0.635
58-Ce-140	88.48	140.12	32.51 d	0.57	0.48	145.5(48)	0.0036
57-La-139	99.91	138.9065	40.23 h	8.93	12.2	487(46.1); 1596.2(95.5)	0.064
30-Zn-64	48.9	65.37	265 d	0.76	1.4	1115.5(51)	0.0057
30-Zn-68	18.6	65.37	13.9 h	0.072	0.24	574(100)	0.0002
21-Sc-45	100	44.9559	83.8 d	27.2	11.5	889.3(100); 1120(100)	0.605
62-Sm-152	26.63	150.4	46.8 h	206	2960	103.2(28.3)	0.365
51-Sb-121	57.25	121.75	2.7 d	5.9	202	564.1(63)	0.0277
51-Sb-123	42.75	121.75	60.3 d	4.28	50	602.7(98.1); 722.8(11.8); 1691(50)	0.015
79-Au-197	100	196.9665	2.695 d	98.8	1551	411.8(95.5)	0.502
40-Zr-94	17.4	91.22	65.5 d	0.056	0.38	724.2(43); 756.7(54.6)	0.0001
42-Mo-98	23.78	95.84	66.02 h	0.13	1.3	140.5(5); 181.1(7);739.7(14); 779(4)	0.0003
73-Ta-181	99.98	180.918	115.1 d	21.5	717	1121(35); 1189(16); 1221(27)	0.1188
31-Ga-71	40	69.72	14.1 h	4.86	31.2	630(25.5); 834(100); 894(10.3)	0.026
72-Hf-180	35.22	178.49	42.5 d	12.6	33.6	133(43); 346(14); 482(86);	0.025
77-Ir-191	38.5	192.2	74.2 d	624	35.35	206(70); 308(31); 316(87); 468(50); 485(67)	1.25
34-Se-74	0.9	78.96	120 d	51.8	424	121.1(16.5);135.9(58);264.5(58.5);279.5(25)	0.006
37-Rb-85	72.17	85.47	18.7 d	0.43	3.76	1077(9)	0.0036

Most elements associated with various industries belong to group I toxicants and are potentially hazardous for the environment and human beings. Biological research is currently being carried out in many areas with the concentrated efforts of scientists from many countries. The most important of these areas are the investigation of environmental objects (soil, water, air, vegetation, *etc.*); analysis of trace elements in human and animal tissues; and investigation of the elemental composition of foodstuffs [16].

The biomonitoring technique is widely used in various variants in this research. Depending on the character of the particular investigation, the biomonitors are mosses and lichens, bacteria, fungi, animals, and human and animal tissues and substrates, *etc.*[8,16,19,21,24]. The statistical approach to solution of environmental problems using biomonitors requires a multielement analysis of a great deal of samples, and in this respect non-destructive instrumental neutron activation analysis (INAA) surpasses other techniques [16].

The detection limits that can be achieved by INAA strongly depend on the chemical composition of the sample in question. Most often one or more of the matrix elements give rise to activities strongly interfering with the analysis by producing a high background in the gamma-spectrometry measurements. In the cases where the major interfering radionuclides have shorter half-lives than those of interest in the analysis, the

problem can partly be overcome by appropriate delay of the measurements [14].

In the period 2009-2013, at JINR Dubna, INAA has been applied by Romanian research teams (a consortium consisting of "Valahia" University of Targoviste (UVT), "Dunarea de Jos" University of Galati, "Al.I. Cuza" University of Iasi and University of Baia Mare) in collaboration with NAA group from FNLP-JINR in conjunction of moss biomonitoring technique for the study of atmospheric pollution in Romania [26,27] and the results have been synthesized in the recently published atlas of heavy metals deposition in Europe [28] in the frame of European moss survey 2010/11, conducted under the auspices of the UNECE ICP Vegetation. Within the bilateral project JINR-Romania, "Nuclear and related analytical techniques for Environmental and Life Sciences", moss samples were collected during the summer/autumn of 2010 at 332 sites in Romania: in the Carpathian Mountains, Transylvanian plateau, and Moldavia province, following internationally accepted guidelines. A total of 42 elements (Na, Mg, Al, Cl, K, Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Zn, As, Se, Br, Rb, Sr, Zr, Mo, Ag, Cd, In, Sb, I, Cs, Ba, La, Ce, Sm, Eu, Tb, Hf, Ta, W, Au, Hg, Pb, Th, and U) were determined by epithermal instrumental neutron activation analysis at the pulsed fast reactor IBR-2, FLNP, JINR, complemented by AAS at UVT.

Using fast neutrons obtained from industrial neutron generators (**Fig. 2**) in geochemical research and mineral exploration, a sensitive determination of gold in auriferous sands, rocks and concentrates [5,25] and of alkali metals [15] in iron ores used in metallurgical industry is possible. In geochemical exploration, where a large number of auriferous samples have to be analyzed at a low cost, the use of the neutron generators, short irradiation and NaI(Tl) spectrometry is justified for gold analysis at trace level [5,25]; care must be taken in this case to small corrections for the interferences due to Rb, V and Ti. In the case of ^{197}Au(n,n')^{197m}Au reaction, if available, a Ge(Li) detector can be used for the detection of 279 keV gamma rays of the gold short-lived radionuclides without any interference. When only a few number of samples are to be evaluated or gold must be determined more accurate in a sample, the long irradiation and Ge(Li) spectrometry of the 355.7 keV most intense γ-ray of ^{196}Au from the reaction ^{197}Au(n,2n)^{196}Au is clearly the method of choice, taking into account only the small contribution of Se to the gold concentration. The contribution of the nuclear interfering elements – Hg and Pt – to the concentration of gold in the samples has been calculated and from the obtained data it can be deduced that the nuclear reactions ^{197}Au (n, 2n)^{196}Au, ^{197}Au (n, 2n)^{196m}Au and ^{197}Au (n, n')^{197m}Au can be used for gold determination, with minimal errors. In conclusion, two methods for rapid determination of gold in auriferous geological materials in the range 20-2500 ppm were proposed [25], using optimum calculated experimental times, so that the systematic errors of analysis due to the gold accompanying elements should be considerably diminished: a method using short irradiation (25 s) and a NaI(Tl) detector for measuring the induced gamma radioactivity in the samples and a method using long irradiation (3000 s) and a Ge(Li) detector.

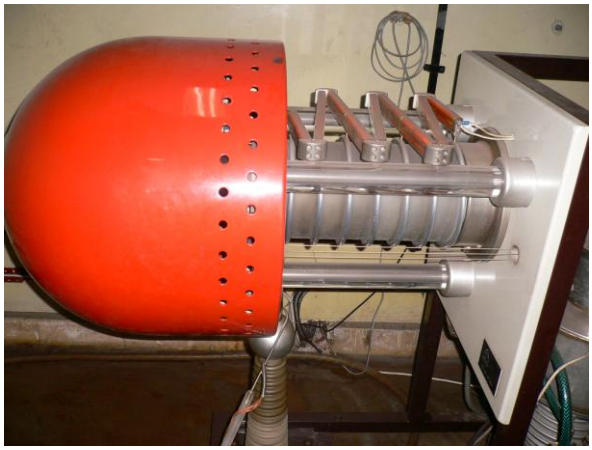

Figure 2: *Neutron generator used for fast (14 MeV) NAA in metallurgical industry (ISW Galati)*

From this work it results that by applying INAA technique, a very good overall picture of the elemental composition of a multi-elemental sample may be obtained. For light elements determination, INAA could be coupled with other nuclear and ion beam analysis techniques [29].

Extended work will be carried out in the future using INAA at JINR Dubna, Russia, for the investigation of micro-composition of high purity advanced materials.

4. Conclusions

Retrospective review and future prospects of Russian-Romanian studies in Materials and Life Sciences using INAA at IBR-2 reactor in Dubna are presented in the frame of collaboration between "Dunarea de Jos" University of Galati (UDJG), Romania, and Frank Laboratory of Neutron Physics (FLNP) of Joint Institute of Nuclear

Research (JINR) at Dubna, Russia, on metallurgical materials and environmental samples. A large number of microelements could be determined in metallurgical and environmental samples at part-per-million (ppm) level.

The perspectives of applying INAA for the characterization of high purity advanced materials in combination with their microstructure characterization are described.

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