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Experiment

Epithermal Neutron Activation Analysis at the IBR-2 Reactor of the Frank Laboratory of Neutron Physics at the Joint Institute for Nuclear Research (Dubna)

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Abstract—Experience of the Neutron Activation Analysis (NAA) Department in employing epithermal activation in life sciences and materials science is summarized. The potential of a combination of epithermal activation and the suppression of Compton scattering and contributions from cascade-photon-emitting elements for raising NAA-based analytical studies up to a new level are discussed.

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1. INTRODUCTION

Application of neutron activation analysis (NAA) was initiated at the Laboratory of Neutron Physics [now named after Frank and known as the Frank Laboratory of Neutron Physics (FLNP)] at the Joint Institute for Nuclear Research (JINR, Dubna) by I.M. Frank in the late 1960s, when this method aroused the keenest interest at reactor centers worldwide. Even at the IBR-30 reactor of the laboratory, a simple pneumatic transport facility was used to irradiate geological and biomedical samples for studying their element composition. When put into operation, the IBR-2 reactor with its high epithermal- and fast-neutron fluxes served to improve the multielement instrumental NAA and to promote epithermal neutron activation analysis (ENAA). Until the shutdown of the IBR-2 for conversion in 2007, both neutron activation techniques were widely used in life- and materials-science research. In the future, it is planned to enhance the experimental NAA basis by using a system for suppressing Compton scattering and contributions from elements emitting cascade photons. This would allow a higher level of analytical studies after commissioning the modified IBR-2M reactor.

2. RADIOANALYTICAL INVESTIGATIONS AT FLNP

Fission neutrons from the JINR FLNP IBR-2 pulsed fast reactor range in energy from ultracold (10^{-7} eV) to fast (20 MeV) ones. Thermal neutrons are most widely used in instrumental neutron activation analysis (INAA) owing to their copious production in the course of moderation of fission neutrons and larger interaction cross sections for most

isotopes. In many cases, reactions for host elements with large thermal-neutron activation cross sections lead to a great increase in the γ background. This is a particularly acute problem in element analysis of environmental and biological samples, where trace-element content has to be determined against the background of macroelements such as Na, Al, Sc, Mn, Fe, Co, and La emitting high-energy γ rays of Compton continuum, which often disguises peaks used to determine some elements. Therefore, selective activation by epithermal (resonance) neutrons with appropriate filters used to screen the thermal-neutron flux [1–4] is efficient in some cases. This NAA method, known as ENAA, is widely used for radioanalytical investigations at the IBR-2 reactor.

Thermal neutrons are efficiently screened by a filter of cadmium or any other material with a large thermal-neutron-absorption cross section, while the flux of epithermal (resonance + fast) neutrons is allowed to pass through it. In the IBR-2 cadmium channel with a filter 1 mm thick, epithermal neutrons have energies ranging from 0.55 eV to about 1 MeV. The thermal-to-epithermal-activation-probability ratio varies within a wide range for different nuclei of the irradiated target. This fact is usually illustrated by comparing ratios of the resonance-activation integral to the neutron cross section (I_0/σ_0) for the corresponding nuclei (Table 1) [5]. The gain in activity of the element to be determined, X , with respect to the interfering radionuclide D can be represented in the form

$$F = (I_0/\sigma_0)_X / (I_0/\sigma_0)_D.$$

This ratio is on the order of 0.5 for nuclei obeying the $1/v$ law in the epithermal region and may be as large as about 100 for other nuclei. Many trace elements—in particular, rare-earth ones—have large resonance integrals at specific epithermal energies. Herein lies an advantage to the NAA with epithermal activation used in addition to the activation by the entire spectrum of reactor neutrons. The method makes it possible to (i) improve INAA determination limits for elements such as As, Br, Rb, Sr, Cd, Sb, I, Tb, Hf, Ta, Th, and U; (ii) reduce the activity of host elements such as Al, Mn, Na, La, Sc, and Co; and (iii) reduce the interference of ^{235}U decay products when it is significant (e.g., in determination of molybdenum).

The REGATA radioanalytical complex at the IBR-2 reactor has four irradiation channels. Of these, two are connected to the pneumatic transport system. The parameters of the irradiation channels and experimental facility are described at length in [6]. The complex comprises an automated system for control, collection of spectrometric information, and its treatment. The system also allows a cyclic irradiation of samples when short-lived isotopes of few-second lifetime should be determined. A noteworthy feature is low temperature in irradiation channels (not higher than 60 to 70°C), this being of importance in dealing with biological and environmental samples. Spectrometric information is processed with software developed in the NAA Department of FLNP [7]. A regular participation in international interlaboratory comparisons of the results of analyses of various types of samples allow the conclusion that the analytical investigations carried out at the NAA Department comply with the currently adopted standards.

3. NAA APPLICATIONS AT THE IBR-2 REACTOR

3.1. Biomonitoring

In the 1990s, the NAA Department grew steadily interested in environmental research (analysis of environmental objects like soil, water, vegetation, and aerosol filters). In 1991, a new research direction—biomonitoring of the atmospheric deposition of heavy metals and other elements using mosses as biomonitors (passive biomonitoring)—began developing. Since 1995, the NAA Department has been taking part in the European program “Atmospheric Heavy-Metal Deposition in Europe: Assessments Based on Moss Analysis,” submitting the results of analysis of mosses taken in various European countries [8–15] to the European Atlas [16, 17]. In addition, the NAA Department carried out studies using transplanted mosses (active biomonitoring) environmentally exposed in areas where natural ground mosses disappeared because of intense technogenic pollution or

Table 1. Nuclear data for elements determined via (n , γ) reactions [5]

Element	Radionuclide	$T_{1/2}$	I_0/σ_0
ENAA preferred			
Ga	^{71}Ga	14.1 h	6.7
As	^{75}As	26.3 h	13.6
Se	^{74}Se	119.8 d	10.0
Br	^{81}Br	35.3 h	19.3
Rb	^{85}Rb	18.7 d	14.8
Sr	^{84}Sr	64.8 d	13.2
Zr	^{96}Zr	16.7 h	248
Mo	^{98}Mo	65.9 h	53.1
Ag	^{109}Ag	249.7 d	17.5
Cd	^{114}Cd	53.5 h	39.6
Sn	^{112}Sn	115.1 d	48.4
Sb	^{121}Sb	2.70 d	33.0
Sb	^{123}Sb	60.2 d	28.8
Cs	^{133}Cs	2.06 yr	12.7
Ba	^{130}Ba	11.8 d	24.8
Sm	^{152}Sm	46.7 h	14.4
Eu	^{153}Eu	8.56 h	5.7
Cd	^{158}Cd	18.6 h	31
Tb	^{159}Tb	72.1 h	17.9
Ho	^{165}Ho	26.8 h	10.9
Tm	^{169}Tm	128.6 d	14.5
Ta	^{181}Ta	114.4 d	32.3
W	^{186}W	23.9 h	13.7
Re	^{185}Re	90.6 h	15.4
Au	^{197}Au	2.7 d	15.7
Th	^{232}Th	27.4 d (^{233}Pa)	11.5
U	^{238}U	2.35 d (^{239}Np)	103.4
Ordinary NAA			
Na	^{23}Na	15.0 h	0.59
Mg	^{26}Mg	9.46 min	0.64
Al	^{27}Al	2.24 min	0.71
K	^{42}K	12.4 h	0.97
Ca	^{46}Ca	4.53 d	1.3
Ca	^{48}Ca	8.72 min	0.45
Sc	^{45}Sc	83.8 d	0.43
Cr	^{50}Cr	27.7 d	0.53
Mn	^{55}Mn	2.58 d	1.05
Fe	^{58}Fe	44.5 d	0.97
Co	^{59}Co	5.27 yr	1.99
Cu	^{63}Cu	12.7 h	1.14
Zn	^{64}Zn	243.9 h	1.91
La	^{139}La	40.3 h	0.88
Ce	^{140}Ce	32.5 d	0.83
Nd	^{146}Nd	11.0 d	2.00
Eu	^{151}Eu	12.4 yr	0.87
	^{235}U	Fission	0.48

anthropogenic effect and in arid areas, where mosses never grow at all [18–20].

3.2. Determination of Halogens in Soils and Mosses

ENAA is known to be an unsurpassed method for a simultaneous determination of chlorine, bromine, and iodine. Therefore, it was used to assess the gradient of concentration of these elements in moss and soil samples collected within the range of 0–300 km down into the continent from the Norwegian seashore. The results showed that concentrations of all three elements in the soils and mosses decrease exponentially with increasing distance from the seashore, this being indicative of the marine origin of halogens [21, 22]. Similar behavior of halogens was also noticed in our transect studies of biomonitor mosses in South Korea [23].

3.3. Foodstuff Quality

The quality of local foodstuffs produced in technogenically affected areas and their effect on the health of the population was studied within the coordination programs and technical cooperation with the IAEA using the techniques developed at the NAA Department for foodstuff analysis involving nuclear-physics analytical methods [24, 25].

3.4. Monitoring at Work and Health of People

Combined use of ENAA at the IBR-2, atomic absorption spectrometry (AAS), and x-ray fluorescent analysis (XFA) allowed pollutant elements (Cr, Ni, Cu, Zn, As, Se, Sr, Mo, Ag, Cd, Sb, Pb, rare earths, Th, and U) to be determined in the products of the mineral-fertilizer factory in Voskresensk, Moscow oblast. The results of monitoring at work, the assessment of the effect on the health of the personnel involved in this production (analysis of human biosubstrates: hair, teeth, etc.), and analysis of food crops grown in the immediate neighborhood of the factory with its by-product (phosphogypsum) applied to the soil are reported in [26–28]. The developed techniques were successfully employed in the international project of the EU Fifth Framework Program (Copernicus), where the impact of similar factories on the environment and the health of the local population in Poland, Russia, Rumania, and Uzbekistan was studied [29–31]. At present, the experience of the FLNP NAA Department in this field is used in the joint project performed together with the Nuclear Energy Corporation of South Africa (NECSA) and aimed at studying the effect produced on children's health by food crops grown in some technogenically polluted areas of the Russian Federation and the Republic of South Africa.

3.5. NAA in Assessment of the Condition of Natural Ecosystems

The multielement NAA has shown itself a good method for determining scattered elements in soils of forest areas of European Russia [32, 33]. Profiles of trace elements in Al–Fe humus podsolc soils were studied for the first time [34–36] to assess the areal of pollution from the integrated copper-and-nickel works on the Kola peninsula under varying lithogenic background conditions. The distribution of pollutant elements around the integrated lead-and-zinc works near Skopje (Macedonia) was studied by ENAA and AAS [37]. Concentrations of scattered elements in underground waters was also studied in some central Russia regions subjected to anthropogenic effect [38]. To assess this effect on the environment, samples of water, bottom sediments, and biota taken near Klin (Moscow oblast) from the river Sestra, a tributary of the Volga, were analyzed [39]. Similar investigations were carried out in the Danube delta, and the NAA results obtained in Dubna and Bucharest were compared [40, 41]. ENAA was used to assess the condition of the water ecosystem of the Rybinsk reservoir, which supplies potable water to Moscow and Moscow oblast [42].

3.6. Biotechnologies

The potential of NAA in the blue-green microalga *Spirulina platensis* biotechnology was demonstrated in cooperation with biophysicists from the Adronikashvili Institute of Physics (Tbilisi, Georgia). An experimental substantiation was given to the possibility of making substances for Se-, I-, and Cr-containing spirulina-based pharmaceuticals retaining useful natural properties of spirulina, was reported in some papers (see, for example, [43, 44]), and was reflected in two patents of the Russian Federation [45]. It was shown that the spirulina biomass may also be used as a sorbent of mercury [46], which is a highly toxic metal. Another microorganism, *Arthrobacter oxidans*, separated from natural basalt rocks, was studied by NAA and the electron-paramagnetic-resonance (EPR) method as a reducer of toxic Cr(VI) to chemically stable nontoxic Cr(III) [47]. NAA was also efficiently used for developing the method for bacterial leaching of metals (including uranium and thorium) from low-grade ores, rocks, and wastes of factories in Georgia [48].

3.7. Materials Science

When the development of the REGATA radioanalytical complex at the IBR-2 reactor was at its initial stage, INAA was predominantly used to study particular materials-science problems, such as induced

radiation and physicochemical properties of shielding concrete and structural materials after nuclear plants shielded by them were taken out of service [49] and an analysis of semiconductor materials and high-purity aluminum produced by zone smelting [50]. Recently, the spectrum of IBR-2 fission neutrons was used to study physical properties of artificial small-crystal diamonds produced in Belarus. It was shown that nondestructive ENAA is best suitable for investigating the effect of impurities (Ni, Mn, Al, etc.) on the technological process of the synthesis of small-crystal diamonds [51, 52].

4. ASSESSMENT OF THE POTENTIAL OF USING A COMBINATION OF THE EPITHERMAL ACTIVATION AND COMPTON SUPPRESSION

Compton suppression in γ -spectrometric measurements in the INAA was proposed by Cooper and Brownell as far back as 1967 [53] but was not applied widely until the 1990s. In 1996, Landsberger and Peshev published a review [54] of applications of Compton suppression in INAA. The idea is to decrease substantially the Compton continuum from high-energy γ rays, which often seriously interfere in measurements of low-energy γ rays. The fact that the contributions to the spectrum from radionuclides whose decays are accompanied by two or more γ rays in coincidence will decrease, especially when the sample is located close to the detector surface, is an additional advantage of the method. The decay of some nuclides induced by neutron activation in large amounts in samples of various types involves two or more γ coincidences. The Compton suppression method may thus appreciably reduce interference of these radionuclides in determining other elements when the radionuclide to be determined decays without coinciding γ rays (without γ coincidences).

A combination of the Compton suppression method and the epithermal activation proved itself to be good for analyzing aerosol filters [55], natural waters [56], and biological standards [57]. Below, we consider possible combinations of these two methods as applied to a wide range of samples, including soils, rocks, and so-called new materials (ultrapure semiconductors and nanoparticles).

5. ANALYSIS OF SOME NUCLEAR DATA

Tables 2 and 3 present nuclear data allowing one to estimate the efficiency of the combination of the epithermal activation and Compton suppression in INAA.

The ratios of the resonance integral to the cross section for thermal-neutron-induced reaction, I_0/σ_0 ,

were taken from the studies of Moens et al. [58] and Steinnes [4].

Table 2 presents data for elements determined with the aid of short-lived (half-lives below 15 h) products of (n, γ) reactions, while Table 3 lists data for elements determined with the aid of long-lived products from these reactions. This separation arises from the fact that ^{24}Na , with its half-life of 15.0 h, is the main interfering element for both short- and long-lived radionuclides. Tables 2 and 3 list radionuclides, their γ lines most often used to determine the corresponding element, and fractions of decays where each γ ray is emitted in coincidence with one or more γ rays [59].

According to Landsberger and Wu [57], the Compton suppression system is usually capable of reducing the background by a factor of 5 to 10. Bacci et al. [60] estimated the suppression factor of their system for ^{137}Cs at about five and found that it varies with the distance from the detector. The suppression system shows its advantage when the radionuclide X decays without γ coincidences and when interfering γ rays from D coincide. Obviously, the suppression of coinciding γ rays depends on the configuration of the spectrometer and, probably even to a larger extent, on the distance between the sample and the detector. It should be borne in mind that the location of the sample close to the detector substantially changes the probability for coinciding γ rays to hit the semiconductor detector, this affecting the Compton suppression efficiency.

Taking into account the properties of the major interfering radionuclides given in Tables 2 and 3, we can in general predict when the Compton suppression system is efficient and when it is not. In environmental, geological, and biological samples, ^{28}Al (2.3 min), ^{56}Mn (2.58 h), and ^{24}Na (15.0 h) are most frequently the major interfering radionuclides in the INAA of short-lived isotopes. In the case of ^{24}Na , γ rays of energy 1368 and 2754 keV coincide, and one can appreciably suppress them. In the case of ^{56}Mn , the γ ray of energy 1811 keV can be similarly suppressed and the γ ray of energy 847 keV can also be partly suppressed. However, the γ ray of energy 1780 keV in the case of ^{28}Al is monoenergetic and is not suppressed.

Thus, the advantage of the Compton scattering system in the ENAA of short-lived isotopes can manifest itself only for arising nuclides of half-lives 5 to 10 min and longer.

Long-lived nuclides occurring in soils, silicate rocks, and other environmental samples irradiated with neutrons include ^{140}La (40.2 h), ^{59}Fe (44.6 d), ^{46}Sc (83.9 d), and ^{60}Co (5.26 yr). All these nuclides, with the exception of ^{59}Fe , emit coinciding γ rays,

Table 2. Selected nuclear data for some short-lived nuclides ($T_{1/2} < 15$ h)

Element	Radionuclide	$T_{1/2}$	I_0/σ_0^*	γ -line energy, keV	Fraction of decays involving γ coincidences, %
Na	^{24}Na	15.0 h	0.59	1368	100
			2754	100	
Mg	^{27}Mg	9.5 min	0.68	842	2
			1013	0	
Al	^{28}Al	2.3 min	0.74	1780	0
S	^{37}S	5.1 min	1.1	3090	0
Cl	^{38}Cl	37.3 min	0.69	1600	100
				2170	100
K	^{42}K	12.4 h	0.97	1524	0
Ca	^{49}Ca	8.8 min	—	3100	0
Ti	^{51}Ti	5.8 min	0.66	928	1
V	^{52}V	3.8 min	0.55	1434	1
Mn	^{56}Mn	2.58 h	1.05	847	48
Ni	^{65}Ni	2.56 h	0.64	1115	40
				1481	0
Cu	^{64}Cu	12.8 h	1.1	511	100
	^{66}Cu	5.1 min	1.01	1039	3
Zn	^{69m}Zn	13.8 h	2.9	439	0
Ga	^{72}Ga	14.1 h	6.6	835	90
Sr	^{87m}Sr	2.83 h	5.7	388	0
Zr	^{97}Zr	17.0 h	231	747	7
	^{97}Nb	72.0 min		665	0
Mo	^{101}Mo	14.6 min	18.8	2080	0
	^{101}Tc	14.0 min		307	10
In	^{116}In	54.0 min	16.3	417	100
				1097	100
				1293	100
I	^{128}I	25.0 min	23.7	441	15
Cs	^{134m}Cs	2.90 h	13.2**	127	0
Ba	^{139}Ba	82.9 min	0.89	1430	0
Sm	^{155}Sm	22.4 min	6	104	0
Eu	^{152}Eu	9.3 h	3.9**	122	100
				963	0
Dy	^{165}Dy	139.2 min	<0.5**	94	0
Er	^{171}Er	7.5 h	5.3**	124	100
				308	100
U	^{239}U	23.5 min	100	75	0

* Data of Moens et al. [58].

** Data of Steinnes [4].

Table 3. Selected nuclear data for some long-lived nuclides ($T_{1/2} > 15$ h)

Element	Radionuclide	$T_{1/2}$	I_0/σ_0^*	γ -line energy, keV	Fraction of decays involving γ coincidences, %	
Na	^{24}Na	15.0 h	0.59	1368	100	
				2754	100	
Ca	^{47}Ca	4.53 d	1.28**	1300	0	
	^{47}Sc	3.43 d		160	0	
Sc	^{46}Sc	83.9 d	0.44	889	100	
				1121	100	
Cr	^{51}Cr	27.8 d	0.49	320	0	
Fe	^{59}Fe	44.6 d	1.3	1095	0	
				1292	5	
Co	^{60}Co	5.26 yr	1.99	1173	100	
				1332	100	
Zn	^{65}Zn	244 d	1.91	1115	0	
Ga	^{72}Ga	14.1 d	6.62	630	100	
				835	100	
As	^{76}As	26.5 d	13.6	559	20	
Se	^{75}Se	120.4 d	10	122	100	
				136	100	
				265	100	
				280	100	
				401	87	
Br	^{82}Br	35.3 h	18.5	554	100	
				777	100	
				1044	100	
				1475	61	
Rb	^{86}Rb	18.7 d	11.3	1078	0	
Sr	^{85}Sr	64.9 d	13.3**	514	0	
Zr	^{95}Zr	64.0 d	4.61	724	0	
				756	0	
Mo	^{95}Nb	35.2 d	53.1	765	0	
	^{99}Mo	66.0 h		181	33	
				740	100	
Ag	^{99m}Tc	6.0 h	15.4	143	0	
	^{110m}Ag	250 d		657	100	
				764	100	
				884	100	
				937	100	
				1387	100	

Table 3. (Contd.)

Element	Radionuclide	$T_{1/2}$	I_0/σ_0^*	γ -line energy, keV	Fraction of decays involving γ coincidences, %
Cd	^{115}Cd	53.5 h	78**	530	2
	^{115m}In	4.50 h		335	0
In	^{114m}In	50.0 d	131**	192	
Sn	^{113}Sn	115 d	30	393	2
	^{117m}Sn	14.0 d	82	158	100
Sb	^{122}Sb	2.80 d	33.9	564	5
	^{124}Sb	60.3 d	30.2	603	78
Cs	^{134}Cs	2.05 yr	12.4	1692	100
				605	100
				796	100
Ba	^{131}Ba	11.5 d	25**	123	100
				216	15
				373	35
				493	100
La	^{140}La	40.2 h	1.32	326	100
				487	100
				815	100
				1596	73
Ce	^{141}Ce	32.5 d	0.82	145	0
Nd	^{147}Nd	11.1 d	2.3**	91	6
				531	0
Sm	^{153}Sm	46.8 h	14.4	103	40
Eu	^{152}Eu	12.4 yr	1.15**	122	100
				965	100
				1087	0
				1408	100
	^{154}Eu	8.5 yr	4.21	123	100
Gd	^{159}Gd	18.0 h	24**	724	100
				1278	100
				363	0
				87	100
Tb	^{160}Tb	72.1 d	17	298	100
				879	100
				966	50
				1178	100
				81	0
Ho	^{166}Ho	26.9 h	10.3	84	0
Tm	^{170}Tm	128.6 d	13.5**		

Table 3. (Contd.)

Element	Radionuclide	$T_{1/2}$	I_0/σ_0^*	γ -line energy, keV	Fraction of decays involving γ coincidences, %
Yb	^{169}Yb	30.7 d	4.6**	63	100
				94	100
	^{175}Yb	4.2 d	0.46	283	100
				396	0
Lu	^{177}Lu	6.7 d	<0.5**	113	50
				208	100
Hf	^{181}Hf	42.4 d	2.62	133	100
				482	100
Ta	^{182}Ta	115.0 d	33.5	100	100
				179	100
				222	100
				264	100
				1189	100
W	^{187}W	23.9 h	13.2	134	100
				478	100
				686	0
Ir	^{192}Ir	74.2 d	3.8**	308	84
				316	100
				468	100
Au	^{198}Au	2.70 d	15.7	412	0
Hg	^{197}Hg	64.1 h	0.49	77	0
	^{203}Hg	46.9 d	0.98	279	0
Th	^{233}Pa	27.0 d	11.5	300	100
				312	0
				416	0
U	^{239}Np	2.35 d	100	106	100
				228	100
				278	0***

* Data of Moens et al. [58];

** Data of Steinnes [4].

*** When the coinciding 7.9-keV γ ray remains undetermined.

which can be considerably suppressed by the Compton suppression system during measurements. Thus, the use of the Compton suppression system in measuring the spectra of induced activity of long-lived radionuclides upon epithermal neutron activation will allow a substantially better detection limit owing to a

reduced interference from host components in the γ spectra.

With allowance for these factors, the values of I_0/σ_0 , and the fractions of $\gamma\gamma$ coincidences in Tables 2 and 3, the following elements of those determinable by ENAA can be much better determined in measurements with Compton suppression.

These include the short-lived elements Sr (^{87m}Sr), Zr ($^{97}\text{Zr}/^{97}\text{Nb}$), I, Cs (^{134m}Cs), Eu (^{152}Eu , 9.3 h, 963 keV), and U (^{239}U) and the long-lived elements As, Rb, Sr (^{85}Sr), Mo (^{99m}Tc), In (^{114m}In), Sn (^{113}Sn), Sb (^{122}Sb), Ba (^{131}Ba , 216 keV), Gd, Ho, Tm, W (^{187}W , 686 keV), Au, Th, and U (^{239}Np).

For the other elements well determined after epithermal neutron activation (Ga, Se, Ag, In, Cs, Tb, Yb, Hf, Ta, and W), it is unreasonable to use spectrometry with Compton suppression.

6. CONCLUSIONS

As alternative analysis methods, in particular, ICP-MS (inductively coupled plasma mass spectrometry), the main rival of NAA, have been rapidly developing in the recent decades, NAA, remaining a reference analytical method, needs further improvement for retaining its leading position. To give a new impetus to the use of this method, its capabilities should be increased by methodological improvements. The combined use of ENAA and Compton suppression is one of the ways to increase competitiveness of NAA.

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