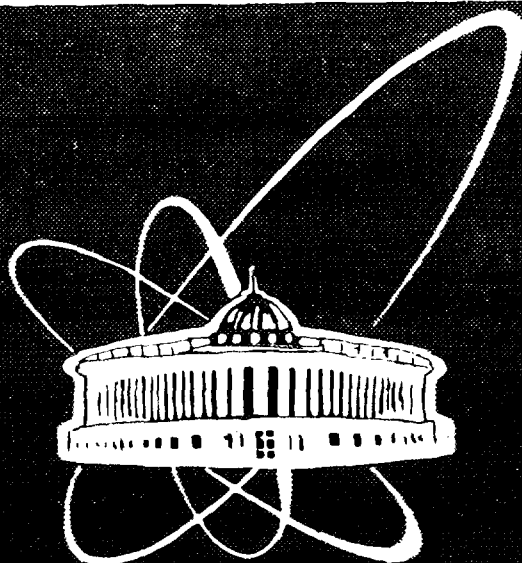




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EPITHERMAL NEUTRON ACTIVATION ANALYSIS  
FOR STUDYING THE ENVIRONMENT

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## 1. INTRODUCTION

The advent of analytical techniques for the simultaneous determination of a great number of elements has created an enormous interest for multi-element studies in environmental sciences. Since the introduction of emission spectrometry more than sixty years ago, a variety of optical, X-ray; mass spectrometric, and nuclear techniques have been introduced and applied to studies of rocks and minerals, soils, plant and animal tissue, aerosols, and many other sample categories of environmental interest. At the present time the most popular multi-element techniques for studies of environmental problems seem to be induction-coupled plasma emission spectrometry (ICP-ES), particle-induced X-ray emission (PIXE), instrumental neutron activation analysis (INAA), and most recently, induction-coupled plasma mass spectrometry (ICP-MS). Each of these techniques have their own merits and problems.

INAA as a multi-element technique depended on the development of stable solid-state detector systems for gamma-spectrometry. First introduced for the analysis of silicate rocks [1], INAA soon found applications to other sample types of environmental interest such as aerosols [2], fly ash [3], water [4], and plant tissue [5]. The strongest merits of INAA was relatively few sources of systematic errors, lower detection limits in many cases than what could be achieved by other multi-element techniques, and the fact that no dissolution of the sample was necessary. Moreover it was in some cases possible to analyze very small samples for a considerable number of elements. On the other hand the precision and detection limit that could be obtained often depended strongly on the element composition of the sample, and a combination of different irradiation and counting times was necessary to achieve an optimal result. Last, but not the least, INAA showed poor performance for some of the elements of greatest interest in environmental pollution studies, *i.e.* lead, nickel, copper, cadmium and tin, and had to be supplemented by other techniques in cases where these elements were of importance [6]. Therefore, although still having a strong position in specific cases such as the analysis of aerosols, INAA has lost considerable ground since its strongest period in the 1970's. In particular the introduction of ICP-MS, which offers low detection limits for most elements in the Periodic table, has made INAA less attractive than before for a number of multi-element studies.

The stronger competition from analytical techniques has made it more necessary than before for the activation analyst to look for approaches to improve the selectivity and detection power of his methods. In many cases that can be achieved by means of epithermal neutron activation analysis (ENAA) where the thermal neutrons are excluded by surrounding the samples with a

suitable filter material, enabling only the resonance and fast reactor neutrons to penetrate into the sample. In the present paper the performance of ENAA to environmental studies is discussed, and based on the experience of the authors, some examples are given where it has been possible by ENAA to determine trace elements which could not be done as well by non-nuclear techniques.

## 2. ACTIVATION WITH EPITHERMAL NEUTRONS

The neutron energy spectrum in a nuclear reactor is conventionally divided into the following three parts:

- a) Reactor fast neutrons, exhibiting an energy range up to at least 15 MeV, and with the most probable energy at about 1 MeV;
- b) Neutrons of intermediate energies, which are in the process of slowing down in the reactor. This component is usually named resonance neutrons, and includes neutrons with energies from below 1 eV to about 1 MeV;
- c) Thermal neutrons, which are in thermal equilibrium with the moderator, with a most probable energy of about 0.03 eV depending on the neutron temperature.

The thermal neutrons can be effectively removed by means of a filter consisting of some material with very high thermal neutron absorption cross-section, such as boron, cadmium, or gadolinium. This facilitates selective activation with the epithermal neutron flux in the reactor (resonance + fast neutrons). The practical «cut-off» value of the thermal neutron filter depends on the character and thickness of the filter material. For the commonly used 0.7 mm cadmium foil it is about 0.4 eV.

Most radioactive nuclides originating from activation with thermal or resonance neutrons are produced by (n, $\gamma$ ) reactions. In the thermal neutron region the (n, $\gamma$ ) activation cross-section of most nuclides follows the  $1/v$  law (is inversely proportional to the neutron velocity). Some nuclides mainly follow the  $1/v$  law also in the epithermal region, whereas others show strong resonances in their cross-section function in that region. Therefore the ratio of thermal to epithermal activation shows large variation between different target nuclides, as conveniently illustrated by the ratio of resonance activation integral/thermal neutron cross-section ( $I_r/\sigma_0$ ) of the nuclides concerned. Whereas this ratio is of the order of 0.5 for nuclides following the  $1/v$  law in the resonance region, it may be as high as 100 in other cases. This means that the radionuclide distribution originating from epithermal activation may deviate strongly from that apparent when the whole reactor spectrum is employed, and that forms the basis of ENAA.

In Table I relevant data for long-lived nuclides ( $T_{1/2} > 15$  h) shown to be suitable for determination by ENAA are listed along with the most important interfering nuclides in environmental samples, and in Table II a similar list is presented for short-lived nuclides. Normally the advantage of ENAA is significant if the  $I_r/\sigma_0$  ratio corresponding to the radionuclide of interest is 10 times higher or more than that of the interfering nuclide, provided that the total counting rate of the epithermally irradiated sample is sufficiently high to ensure good counting statistics.

In addition to improving detection limits, ENAA sometimes leads to reduction in fission product interference from uranium, such as in the determination of molybdenum [7]. Moreover fast-neutron induced reaction products play a much more prominent role than in ordinary INAA. This means that detection limits for elements such as Ni and Si which can be determined via (n,p) reactions are strongly improved. On the other hand interference from (n,p) and (n, $\alpha$ )

Table I. Feasibility of ENAA for some elements yielding long-lived isotopes ( $T_{1/2} > 10$  h) by (n, $\gamma$ ) reactions. A: Favourable by ENAA, B: Favourable by thermal NAA

Element	Target nuclide	$T_{1/2}$ of product nuclide	$I_0/\sigma_0$ [21]
A:			
Ga	$^{71}\text{Ga}$	14.1 h	6.7
As	$^{75}\text{As}$	26.3 h	13.6
Se	$^{74}\text{Se}$	119.8 d	10.0
Br	$^{81}\text{Br}$	35.3 h	19.3
Rb	$^{85}\text{Rb}$	18.7 d	14.8
Sr	$^{84}\text{Sr}$	64.8 d	13.2
Zr	$^{96}\text{Zr}$	16.7 h	248
Mo	$^{98}\text{Mo}$	65.9 h	53.1
Ag	$^{109}\text{Ag}$	249.7 d	17.5
Cd	$^{114}\text{Cd}$	53.5 h	39.6
Sn	$^{112}\text{Sn}$	115.1 d	48.4
Sb	$^{121}\text{Sb}$	2.70 d	33.0
Sb	$^{123}\text{Sb}$	60.2 d	28.8
Cs	$^{133}\text{Cs}$	2.06 y	12.7
Ba	$^{130}\text{Ba}$	11.8 d	24.8
Sm	$^{152}\text{Sm}$	46.7 h	14.4
Eu	$^{153}\text{Eu}$	8.56 h	5.7
Cd	$^{158}\text{Cd}$	18.6 h	31.0
Tb	$^{159}\text{Tb}$	72.1 d	17.9
Ho	$^{165}\text{Ho}$	26.8 h	10.9
Tm	$^{169}\text{Tm}$	128.6 d	14.5
Ta	$^{181}\text{Ta}$	114.4 d	32.3
W	$^{186}\text{W}$	23.9 h	13.7
Re	$^{185}\text{Re}$	90.6 h	15.4
Au	$^{197}\text{Au}$	2.7 d	15.7
Th	$^{232}\text{Th}$	27.4 d ( $^{233}\text{Pa}$ )	11.5
U	$^{238}\text{U}$	2.35 d ( $^{239}\text{Np}$ )	103.4
B:			
Na	$^{23}\text{Na}$	15.0 h	0.59
P	$^{31}\text{P}$	14.2 d	0.49
K	$^{42}\text{K}$	12.4 h	0.97
Ca	$^{46}\text{Ca}$	4.53 d	1.3
Sc	$^{45}\text{Sc}$	83.8 d	0.43
Cr	$^{50}\text{Cr}$	27.7 d	0.53
Fe	$^{58}\text{Fe}$	44.5 d	0.97
Co	$^{59}\text{Co}$	5.27 y	1.99
Cu	$^{63}\text{Cu}$	12.7 h	1.14
Zn	$^{64}\text{Zn}$	243.9 d	1.91
La	$^{139}\text{La}$	40.3 h	0.88
Ce	$^{140}\text{Ce}$	32.5 d	0.83
Nd	$^{146}\text{Nd}$	11.0 d	2.00
Eu	$^{151}\text{Eu}$	12.4 y	0.87
	$^{235}\text{U}$	(fission)	0.48

Table II. Feasibility of ENAA for some elements yielding short-lived isotopes ( $T_{1/2} < 10$  h) by  $(n,\gamma)$  reactions. A: Favourable by ENAA, B: Favourable by thermal NAA

Element	Target nuclide	$T_{1/2}$ of product nuclide	$I_0/\sigma_0$ [21]
<b>A</b>			
F	$^{19}\text{F}$	11.2 s	23.4
Br	$^{79}\text{Br}$	4.42 h	13.2
		17.7 m	11.0
Rh	$^{103}\text{Rh}$	42.3 s	7.6
		4.34 m	7.5
In	$^{115}\text{In}$	54.2 m	16.8
Sn	$^{124}\text{Sn}$	9.52 m	60.1
I	$^{127}\text{I}$	25.0 m	24.8
Lu	$^{175}\text{Lu}$	3.64 h	34.8
Th	$^{232}\text{Th}$	22.3 m	11.5
U	$^{238}\text{U}$	23.5 m	103.4
<b>B:</b>			
Mg	$^{26}\text{Mg}$	9.46 m	0.64
Al	$^{27}\text{Al}$	2.24 m	0.71
Cl	$^{37}\text{Cl}$	37.2 m	0.69
Ca	$^{48}\text{Ca}$	8.72 m	0.45
Sc	$^{45}\text{Sc}$	18.7 s	0.55
Ti	$^{50}\text{Ti}$	5.76 m	0.67
V	$^{51}\text{V}$	3.75 m	0.55
Mn	$^{55}\text{Mn}$	2.58 h	1.05
Ni	$^{64}\text{Ni}$	2.52 h	0.67
Cu	$^{65}\text{Cu}$	5.10 m	1.06
Ba	$^{138}\text{Ba}$	83.1 m	0.88
Dy	$^{164}\text{Dy}$	1.26 m	0.25
		2.33 h	0.19
U	$^{235}\text{U}$	(fission)	0.48

reactions in the determination of elements such as Na, Mg, Al, and Cr becomes more troublesome and must be carefully controlled.

For a more comprehensive treatment of ENAA the reader is advised to review papers [8-10].

### 3. ENAA IN MULTI-ELEMENT STUDIES OF ENVIRONMENTAL SAMPLES

Since Brunfelt and Steinnes [11] reported the first multi-element study by means of ENAA, dealing with silicate rocks, this technique soon became a routine tool in many earth science laboratories. It may therefore seem surprising that ENAA hardly seems to have been used in corresponding studies of environmental samples such as soils and sediments, which normally consist mainly of mineral matter and therefore should be similar to silicate rocks with respect to mixture of radionuclides after neutron activation.

Another example where ENAA was early shown to exhibit an excellent multi-element capability, was in the case of coal and coal fly ash [12]. It is not known to the authors to what extent ENAA may have been used later for routine analyses of coal and coal ashes, but there is a deficiency of published material on this matter.

Epithermal NAA of biological material was first studied in a systematic manner by Kucera [13]. Although a considerable number of papers on this topic has appeared in the literature, ENAA of biological material has not proved equally feasible as for geological material. Reasons for this appear to be threefold:

1. Many elements with favourable epithermal activation properties are present at extremely low levels in biological tissue.
2. One of the major interfering nuclides in INAA of biological material,  $^{82}\text{Br}$ , is produced from a nuclide with a high  $I_p/\sigma_0$  ratio.
3. Biological samples generally require more space during irradiation, and are more sensitive to high temperatures.

In general the improvement with epithermal activation relative to conventional INAA seems to be greatest for the elements Ni, As, Se, Sr, Mo, Ag, and I.

Two areas of environmental analysis seems to deserve particular attention when the feasibility of ENAA relative to non-nuclear multi-element techniques is to be discussed: the analysis of airborne particulate matter, and the related subject on analysis of biomonitors of atmospheric deposition.

#### 3.1. Analysis of airborne particulate matter

Ever since the first applications of INAA to study trace elements in air particulate, a majority of investigations related to trace elements in air at rural and remote locations have relied on this technique. This is mainly due to the fact that the total mass of the aerosol collected on a filter sample is often rather small, and thus favours direct instrumental techniques rather than those depending on dissolution of the sample prior to analysis. Air filter analysis therefore seems to be an area where INAA hardly can be replaced by any non-nuclear analytical technique at the present state of the art. Comprehensive texts on INAA of airborne particulate matter have appeared in the literature [14,15].

The elements that form the major activity upon neutron activation of aerosols are very much the same ones as in the case of *e.g.* silicate rocks and fly ash. This means that the advantages of

H															H	He	
Li ○	Be											B ○	C	N	O	F	Ne
Na ○ □	Mg ○ □											Al ○ □	Si	P	S	Cl □	Ar
K ○ □	Ca ○ □	Sc ○ ■	Ti ○ □	V ○ □	Cr ○ □	Mn ○ □	Fe ○ □	Co ○ □	Ni ○ □	Cu ○	Zn ○ □	Ga ○	Ge ○	As ○ ■	Se □	Br □	Kr
Rb ○ □	Sr ● □	Y ○	Zr ○ □	Nb ○ □	Mo ○ □	Tc	Ru	Rh	Pd	Ag ○ □	Cd ○	In	Sn ● □	Sb ○ ■	Te ○	I □	Xe
Cs ○ □	Ba ● □	La ○ □	Hf ○ ■	Ta ○ ■	W ○ □	Re	Os	Ir	Pt	Au ○ ■	Hg ○ □	Tl ○	Pb ○	Bi ○	Po	At	Rn
Fr	Ra	Ac															
		Ce ● □	Pr ○	Nd ● □	Pm	Sm ○ □	Eu ○ □	Gd ○ □	Tb ○ □	Dy ○	Ho ○	Er ○	Tm ○ □	Yb ○ □	Lu ○ □		
		Th ○ ■	Pa	U ○ □	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr		

Fig. 1. Elements suitable for determination in moss by the present version of INAA (□) and by ICP-MS (○). Closed symbols indicate the preferred method for the element in question.

using epithermal activation should also be similar, provided that the induced activity is sufficient to yield satisfactory counting statistics. As shown by Landsberger [16] this is indeed the case for several trace elements of importance in studies of long range atmospheric transport. It is therefore surprising that ENAA has been so little used in aerosol studies. In the opinion of the authors this is an area where much remains to be done, and at JINR, Dubna, ENAA is now being used in several projects involving the analysis of aerosol filters.

### **3.2. Analysis of moss used as biomonitor of atmospheric deposition**

Another challenging area to the environmental analyst is the determination of trace elements in mosses and lichens used as biomonitors of atmospheric deposition. Such primitive plants have no root system, and therefore absorb nutrients and other chemical substances on their surface predominantly from the atmosphere. Mosses have been used in Norway since 1977 on a regular basis to monitor atmospheric deposition of heavy metals in a nationwide grid. The analytical approach used from the beginning was INAA, supplemented by atomic absorption spectrometry for the elements Pb, Cd, Cu, and Ni. Prior to the survey in 1990 it became evident that ICP-MS, which had then become available, was able to produce results of acceptable quality for all elements of first priority [17], and has therefore been used since then in the regular monitoring work.

During the same period, work has continued to test the feasibility of other analytical techniques for the trace element determination in moss. In particular the use of ENAA was investigated employing the IBR-2 pulsed fast reactor in Dubna, which is characterised by a particularly high fraction of resonance and fast neutrons in the total spectrum. By this means the determination of 15 elements (Zr, Sn, Hf, Ta, W, Au, Th, and eight REE) previously not detected in such samples was demonstrated [18], and the relative merits of ENAA and ICP-MS in moss analysis were discussed on the basis of an intercomparison exercise [19].

Prior to a recent deposition study in 1995 covering large parts of Europe three moss reference samples were produced and distributed among more than 60 laboratories in 15 countries to test the relative performance of different laboratories and analytical techniques [20]. The results of this intercomparison demonstrated that good ICP-MS laboratories are now capable of producing apparently satisfactory results for about 55 elements in mosses. The corresponding figure for ENAA is around 45. In the periodic table shown in Fig. 1 those elements which can be determined in moss by either ENAA or ICP-MS are marked. In 40 cases both techniques can be used, and for a majority of these elements it is difficult on the basis of present evidence to give preference to one technique or the other. In 7 cases (Sc, As, Sb, Hf, Ta, Au, Th) ENAA is judged to be the preferred technique, whereas ICP-MS seems preferable in 5 cases (Sr, Sn, Ba, Ce, Nd). Four elements (Cl, Br, I, Se) could only be determined well by ENAA, whereas the reverse situation applied for another 14 elements (Li, Cu, Ga, Ge, Y, Cd, Te, Pr, Dy, Ho, Er, Tl, Pb, Bi). The choice of either multi-element technique thus depends on the purpose of the investigation and the priority of elements.

It should be added that the simultaneous determination of the halogens Cl, Br and I, which is rather straightforward by ENAA in mosses as well as in several other environmental sample types, is hardly possible at all by any existing non-nuclear technique at the levels concerned.

### **3.3. Environmental analysis: total concentrations or acid-extractable fractions of elements?**

In classical chemical analysis the determination of an element in a given sample always meant the **total** concentration of the element, regardless of chemical form. Environmental



samples are often a mixture of organic and mineral material, and it is therefore difficult to find a simple method of total decomposition prior to wet-chemical analysis. Techniques such as INAA/ENAA not requiring sample dissolution therefore have a great advantage if the total concentration is the aim of the analysis.

In environmental studies there is now a strong tendency to use strong acids, most often concentrated nitric acid, for sample decomposition, *e.g.* in high-pressure bombs. This treatment leaves a large part of mineral matter undissolved, but it is very likely to extract all forms of an element that could become bioavailable on a short time scale. Being rather simple it is now the preferred procedure in many environmental studies, including monitoring programs such as the aforementioned European moss survey. In the intercomparison on trace elements in reference moss samples preceding the 1995 deposition survey the laboratories were therefore requested to use concentrated nitric acid if decomposition was required for their method. This resulted in systematically high results by INAA for elements primarily associated with soil particles supplied to the moss by wind erosion [20]. For elements in the moss typically associated with air pollution there was no such bias.

The advantage of «non-destructive» nuclear methods stated above may therefore turn to a disadvantage if acid-extractable rather than total concentrations are preferred. The choice of ENAA or an alternative non-nuclear multielement-technique may therefore depend on whether the total concentration is requested or not.

#### 4. CONCLUSIONS

Activation with epithermal neutrons offers a distinct advantage to conventional INAA for a number of trace elements in many categories of samples involved in environmental studies, and extended use of ENAA is therefore recommended. Analysis of air particulate is a case where extensive application of ENAA is likely to be particularly fruitful. A similar case where ENAA has shown strong performance is in the analysis of moss samples used to monitor atmospheric deposition, where as much as 45 elements have been determined.

The strongest competitor among non-nuclear multi-element techniques is definitely ICP-MS. In cases where samples can be dissolved in a small volume of nitric acid, and where the chloride content of the sample is low such as in moss, the number of trace elements that can be determined with acceptable quality may be very high. In the case of moss there were only 4 elements (Cl, Br, I, Se) among those determined by ENAA where ICP-MS failed to give any useful result. The three halogens however represents a case where no non-nuclear techniques can match the performance of ENAA for environmental studies.

The choice of analytical technique also depends on whether the total content or an acid-extractable fraction of the elements in question is desired. In this case the force of nuclear techniques such as ENAA is evident when total concentrations are required.

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## Эпитепловой нейтронный активационный анализ для изучения окружающей среды

Эпитепловой нейтронный активационный анализ (ЭНАА) имеет ряд преимуществ по сравнению с обычным инструментальным нейтронным активационным анализом (ИНАА) при определении многих микроэлементов с точки зрения точности анализа и снижения предела обнаружения, уменьшения активности матрицы и интерференции с продуктами деления, если таковые имеются. Дается обзор современного состояния ЭНАА и его приложений. Обобщается опыт ЭНАА на реакторе ИБР-2 в Дубне при мониторинговании атмосферных выпадений с помощью мхов-биомониторов. Целесообразность применения ИНАА для многих объектов окружающей среды хорошо известна, и этот метод анализа должен найти еще более широкое применение в экологии. Анализ аэрозолей — это тот случай, где применение ЭНАА имеет особые преимущества. Другой областью эффективного применения ЭНАА является анализ мхов-биомониторов при исследовании атмосферных выпадений, при этом возможно определение до 45 элементов. Однако для обоих приведенных примеров сильным конкурентом является метод масс-спектрометрии со связанной плазмой (ICP-MS), который позволяет определять еще большее число элементов. Приводится сравнение ИНАА и ICP-MS и обсуждаются те случаи, когда применение ИНАА является единственным возможным.

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## Epithermal Neutron Activation Analysis for Studying the Environment

Epithermal neutron activation analysis (ENAA) has certain advantages over the conventional instrumental analysis (INAA) in terms of improvement in precision and lowering of detection limits, reduction of high matrix activity and fission interferences if any. The current status and the applications of ENAA to environmental samples are reviewed. Experience in the use of ENAA in the monitoring of atmospheric depositions by means of moss-biomonitors at pulsed fast reactor IBR-2 in Dubna is summarised. INAA has shown to be useful for a number of sample types of interest in environmental studies, and should find more extensive use in this area. Analysis of airborne particulate matter is a case where ENAA should be particularly useful. A similar case where ENAA has shown strong performance is in the analysis of mosses used as biomonitors of atmospheric deposition, where 45 elements were determined. In this and other cases, however, induction-coupled plasma mass spectrometry is a very strong competitor, offering data for even more elements. A comparison of ENAA and ICP-MS for moss analysis is presented, and cases where ENAA is unique are discussed.

The investigation has been performed at the Frank Laboratory of Neutron Physics, JINR.

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