



APPLICATION OF THE INAA METHOD FOR THE DETECTION OF SEIZED ILLEGALLY TRANSPORTED DRUGS: RELEVANT RADIATION PROTECTION ASPECTS

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Abstract. Various physical methods are successfully used for the non-destructive detection of drugs or other narcotics. In some applications, instrumental neutron activation analysis (INAA) proves particularly suitable for its high sensitivity and reliability. This method of analysis uses the interaction of neutrons with the sample material, which then emits photons, charged particles, and secondary neutrons, the properties of which uniquely reflect the elemental composition of the examined sample. Relevant information on the presence of individual elements in the sample is obtained from the spectrometry of radiation from the activated sample. The method is non-destructive and requires virtually no specific sample preparation. As with any use of radiation, also here, due attention should be paid to ensure adequate radiation protection of workers and minimization of the impact on the surrounding environment.

Keywords: neutron, activation analysis, drugs, detection, identification, spectrometry, radiation protection

1. INTRODUCTION

Instrumental neutron activation analysis (INAA) represents a sensitive analytical method for determining and identifying a tiny amount of different elements in an analyzed sample. The technique is based on the nuclear properties of constituent elements. This method involves placing a small amount of sample material in a flux of neutrons which will activate some specific nuclides in the sample.

One of the following neutron sources can serve as a neutron source for INAA [1,2]: a radionuclide neutron source, a nuclear reactor, or a neutron generator. Each of these sources has specific properties, especially in terms of neutron emission (number of neutrons per second) and the shape of the energy spectrum, which is crucial in terms of interaction processes and parameters of the radiation produced. Nuclear reactors produce the highest neutron output from these sources but are very limited by their availability, size, and high logistical overhead. Radionuclide sources include, for example, AmBe (Americium-Beryllium) and PuBe (Plutonium-Beryllium), where neutrons are produced by the interaction of alpha particles with beryllium nuclei. Another radionuclide source, Californium 252 (Cf-252), produces neutrons as a result of spontaneous fission. For all radionuclide sources, their continuous neutron production and their half-life must also be taken into account, leading to a constant decrease in emissions. Fusion neutron sources produce neutrons by merging deuterium-deuterium or deuterium-tritium nuclei. For some purposes, this type of source is very advantageous due to its small size and still reasonable emission.

Due to the fact that the work takes place near strong neutron sources, which also emit gamma radiation, it is necessary to ensure adequate protection of personnel and other persons who could experience unwanted

radiation, especially in the event of an emergency radiation situation. The paper discusses the optimal possibilities of radiation protection in these specific conditions, where it is necessary to use particular approaches to monitor mixed fields of n-gamma radiation.

2. PRINCIPLES OF THE INAA METHOD

2.1. Physical aspects

In general, the INAA is a radioanalytical technique using nuclear reactions in an analyzed sample caused by the intensive flux of neutrons from reactors or other suitable strong neutron sources. After the irradiation, the sample becomes radioactive and emits radiation which is then analyzed spectrometrically. Radionuclides are formed and subsequently decay by emitting gamma rays unique for a particular radionuclide in terms of half-life and energy. Gamma-ray intensity is proportional to the element content in the sample. Instrumental neutron activation analysis is the most sensitive analytical technique used for the quantitative multielement analysis of major, minor, and trace elements in samples from almost every conceivable field of scientific or technical interest [3]. It is also widely used for the detection and identification of drugs and other narcotics. Based on the unique presence of trace elements, it would be possible to establish the origin of the seized drugs since each region is characterized by some specific composition of these elements [3]. The principle of the INAA is illustrated in Fig. 1.

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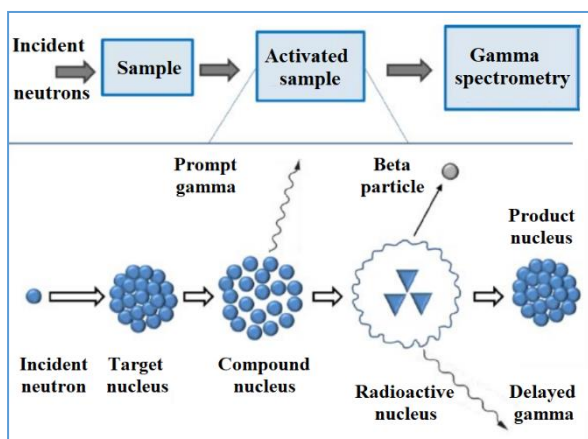


Figure 1. Schematic representation of neutron activation analysis steps and illustration of the neutron capture process (based on [3]).

2.2. Instrumentation

The procedure and instrumentation for INAA are illustrated in Fig. 2. The results are then brought to the computer for the final evaluation of data and their presentations in terms of activities of individual elements present in the sample. The results are then brought to the computer for the final evaluation of data and their presentations in terms of activities of individual elements are present in the sample.

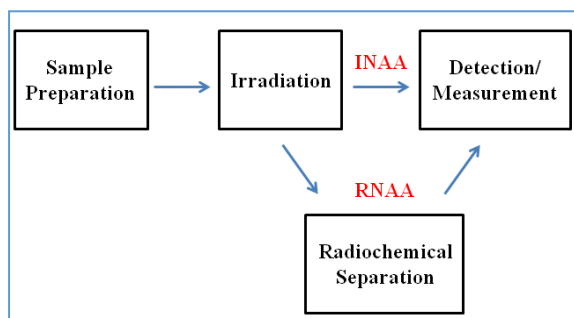


Figure 2. Main procedures and blocks for INAA and RNAA.

This was done in addition to a standard method based on the analysis of gamma rays produced by an activated sample after its irradiation. It can be, in principle, also used as an alternative modality to obtain information about the elemental composition of the sample base on the spectrometry of the radiation from the sample after its radiochemical separation. This method, known as Radiochemical Neutron Activation Analysis (RNAA), is not discussed in the paper, which concentrates on applying the INAA.

Fig. 3 shows the principal conventional individual parts of the INAA instrumentation, which includes the detector cooled by liquid nitrogen accommodated in the Dewar container and electronic blocks starting from the power supply, the preamplifier, linear amplifier, the analogue to digital converter and the computerized multichannel analyzer. In most nowadays the instrumentation for INAA, most of these blocks, including a dedicated computer, are integrated into one compact piece of equipment.

The germanium detector is positioned in a sufficient lead shield to reduce its background radiation response.

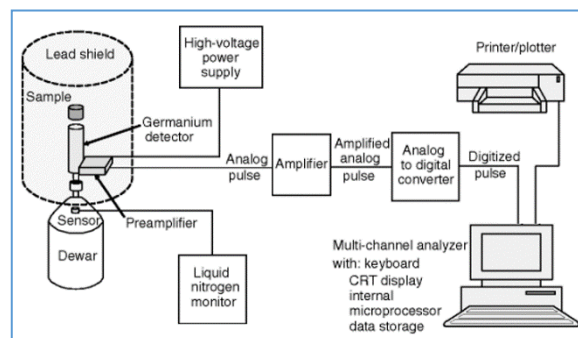


Figure 3. Schematic representation of neutron activation analysis steps (based on [4]).

3. SOME RESULTS OF THE ANALYSIS OF SELECTED DRUGS

Results of the determination of elements in heroin and cocaine samples from the narcotic and psychotropic substances of the Criminal Investigation Institute of the Czech Police in Prague are listed sequentially in Table 1 and Table 2 in the form of the determined value combined. In the last right columns, these tables show the ratios of the highest and lowest determined element contents, which clearly show the differences in the elemental composition of the analyzed drug samples.

Table 1. Contents of elements I in heroin samples determined by the INAA method (based on [6]).

Nuclide, unit	Sample code			Ratio C _{max} /C _{min}
	H963	H1056	H210	
Na, mg.kg ⁻¹	2080±30	294±4	701±11	9.5
Al, mg.kg ⁻¹	11±3	52±2	21±2	5.5
Cl, mg.kg ⁻¹	686±21	44±14	25±14	3 100
Ca, mg.kg ⁻¹	640±50	2260±100	3220±120	5.0
Mn, mg.kg ⁻¹	0.29±0.03	4.15±0.19	3.41±0.18	32.8
Fe, mg.kg ⁻¹	5.5±1.4	80±3	112±3	20.4
Zn, mg.kg ⁻¹	2.91±0.10	2.85±0.09	27.9±0.04	8.7
Br, mg.kg ⁻¹	0.98±0.15	0.11±003	0.19±0.04	170000
Sr, mg.kg ⁻¹	< 4	10.0±0.8	13.3±0.9	> 4.2
I, mg.kg ⁻¹	15.8±1.1	< 1.5	< 1.5	> 16
Sm, µg.kg ⁻¹	< 7	5.6±1.3	< 5	> 1.4
Th, µg.kg ⁻¹	< 7	9±3	16±3	> 3.7
Co, µg.kg ⁻¹	< 9	12±3	19±4	> 4.7
Sc, µg.kg ⁻¹	1.2±0.2	11.4±0.3	5.2±0.3	9.5

The somewhat different composition of the matrix of this material from the design of the matrices of heroin and cocaine samples is not a problem, given the significant matrix independence of the INAA method. In this control material, the contents of 22 elements are

certified, and the information contents are given for 18 other ingredients.

The results show the different elemental compositions of the analyzed samples of heroin and cocaine. It was possible to determine 16 elements in at least one of the heroin samples analyzed samples (see Tab. 1), the contents of another 28 elements were below the detection limit in all analyzed samples. Detection limits for elements Ag, Sb, Cs, Tb, Dy, Ho, Tm, Yb, Lu, Hf and Ta were in the range of units to tens of pg kg^{-1} , for elements V, Ni, Cu, Ga, As, Se, Rb, Mo, Cd, B4 Pr, Nd, W and U was the range of detection limits of tenths to units of mg kg^{-1} , for the determination of elements with the lowest sensitivity Mg, Ti and S were detection limits in the range of tens to thousands of ng kg^{-1} .

Table 2. Contents of elements l in cocaine samples determined by the INAA method (based on [6,7]).

Nuclide, unit	Sample code			Ratio C_{\max}/C_{\min}
	K1300	K1301	K1328	
Na, mg.kg^{-1}	189±3	8.64±0.14	2.27±0.06	83
Cl, mg.kg^{-1}	10.39±	8.95±0.15	10.3±0.17	1.2
Mn, mg.kg^{-1}	5.6±0.4	1.5±0.3	< 1.1±	5.6
Zn, mg.kg^{-1}	9.6±0.2	19.3±0.4	0.81±0.05	37
Co, $\mu\text{g.kg}^{-1}$	< 9	11±3	< 8	> 1.4
Sc, $\mu\text{g.kg}^{-1}$	2.1±0.2	1.3±0.2	1.1±0.2	1.9
Br, mg.kg^{-1}	8.53±0.15	5.20±0.09	11.31±0.18	2.2

4. RADIATION PROTECTION ASPECTS

4.1. General considerations

Any use of ionizing radiation and nuclear technologies, including their applications in the INAA, requires ensuring appropriate safety and security of persons as well as adequate protection of the environment. This is why the applications and handling of radiation sources should be in line with the relevant national and international standards containing appropriate safety and security requirements and recommendations. In order to understand and follow these standards, it is necessary to assess the related radiation risks, which should be quantified by using specific dosimetry and radiation protection quantities and units. It is important to understand these quantities and units and evaluate the biological harms attributed to both stochastic and deterministic effects. The correct use and interpretation of radiation quantities are important to follow relevant regulations and to communicate radiation risks to workers and the public. The following part of this paper takes into account the latest situation in the field, relying on the recent position of relevant international expert bodies such as ICRP [8], ICRU [9], IAEA [10] and some others. For the countries in the Europe region, an important role is also played by the EU Directive (Based on the IAEA BSS), which are for the Member States obligatory [11]. The EU Directive establishes uniform basic safety standards and

requirements for the adequate protection of the health of workers, patients and members of the public against the dangers of ionizing radiation.

In the case of INAA, one has to consider protection from several types of radiation, including neutrons which, together with gamma photons, may form mixed radiation n-gamma. The dosimetry, in this case, is more complicated since we have to consider the different responses of detectors to each of the radiation components.

Following radiation exposure by external radiation or internal radioactive contamination (inhalation or ingestion), one may expect two kinds of biological effects: stochastic effects and tissue reactions (formerly known rather as deterministic effects). The process and its consequences in these two cases differ (Fig. 4) [12]. While at low doses, only stochastic effects occur, the probability of which is proportional to the exposure level. At the dose above a specific threshold level, tissue reactions appear. Here the probability of their occurrence is above this level of 100%, but the severity of health consequences increases with the dose.

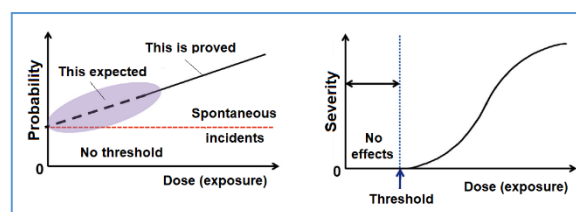


Figure 4. Comparison between stochastic and deterministic effects.

The above figure shows that there should be two different systems for quantifying radiation risks attributed to both stochastic and deterministic effects. Specific quantities and units should be introduced to express these risks.

4.2. Radiation protection measures

Introducing any radiation protection measures aimed at keeping exposure within relevant limits and reference levels requires quantifying the risks based on quantities specifically selected and defined for this purpose. The aim consists of preventing any emergency situation and keeping the exposure per the principle ALARA (As Low As Reasonably Achievable), taking into account societal and economic factors. It means that it is not enough to keep the dose just below the prescribed limits set by the national regulatory authority but to try to find a way to reduce the exposure to a minimum under the circumstances. Obviously, the level of protection may differ in various countries depending on their economic potential.

4.2.1. Stochastic effects

In the case of *stochastic effects*, the principal quantity for this purpose is the effective dose which is defined as

$$E = \sum_T w_T \sum_R w_R D_{R,T} \quad (1)$$

where $D_{T,R}$ is the dose averaged over a tissue or organ T due to radiation of type R incident on the body or emitted by radionuclides in the body and weighted by radiation weighting factors w_R , and w_T is the tissue weighting factor. The factor w_R is related to the Linear Energy Transfer (LET), which reflects the average amount of energy transferred per unit of distance travelled) and is usually expressed in units of keV/ μ m. The values of w_R for some radiations are as follows: low-LET radiation (photons, electrons, muons), 1; protons and charged pions, 2; and alpha particles, fission fragments and heavy ions, 20. For neutrons, this factor depends on the energy [13].

The unit of the effective dose is Sv (sievert), which is too big; therefore, much smaller units are used, such as mSv instead. Since this quantity reflects stochastic (late effects) that occur with a certain probability proportional to the level of exposure in Sv. It means that the quantity can be used only up to the exposure resulting in deterministic effects, i.e. up to about 500 mSv – 1 Sv. This has not always been respected, and even in scientific literature, some mistakes in using the unit of Sv often appear [14].

The application of the effective dose as the main quantity for assessing possible health consequences of stochastic effects must be seen in the context of other quantities (Fig. 5)[12]. The problems with the effective dose and other protection quantities consist of difficulties related to their direct measurement and monitoring. Here different quantities, so-called operational quantities, have been introduced to assess the effective dose.

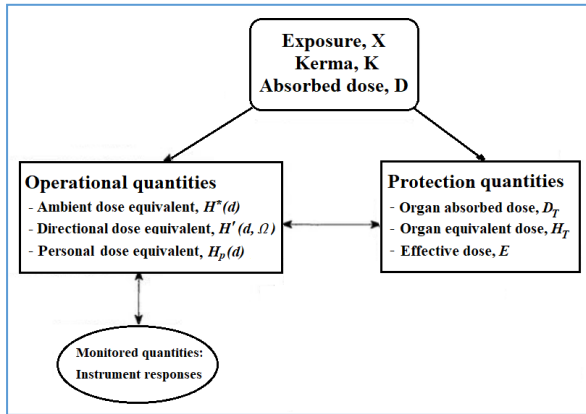


Figure 5. Relationship between quantities used in radiation protection.

The quantities mentioned in Figure 5 are mainly related to external radiation, which strikes the body from the outside. However, the effective dose also includes the exposure from internal doses, causing some difficulties in specifying the doses to individual organs from radionuclides which entered the body through inhalation or ingestion. Both components of the effective dose (external and internal) should be consistent, and in practical cases, it is not easy to comply with such requirements.

Under normal circumstances, exposure of radiation workers and the public are strictly controlled based on compliance with applicable dose limits (Table 3). Only in the case of accident, sabotage or radiological

terrorism may the exposure become much higher. The risk assessment from such events cannot be quantified using the unit of Sv.

Table 3. Dose limits on occupational and public exposure (based on [15]).

Quantity	Organ	Dose limit for exposure	
		Occupational	Public
Effective dose, E	Whole body	20 mSv/y averaged over five consecutive years, and 50 mSv in any single year	1 mSv/y
Equivalent dose, H_T	Lens of the eye	20 mSv/y, averaged over defined periods of five years, with no single year exceeding 50 mSv	15 mSv/y
Equivalent dose, H_T	Skin	500 mSv/y (average dose over 1 cm ² of the most highly irradiated area of the skin)	50 mSv/y
Equivalent dose, H_T	Extremities (hands and feet)	500 mSv/y	-

4.2.2. Deterministic effects

These effects occur with 100% probability almost immediately or within a few days or weeks when the doses exceed certain threshold levels. Here, neither radiation weighting factors nor tissue weighting factors developed under the epidemiological studies at low doses can be applied. Instead, the RBE (Radiobiological Efficiency) should be used. The system for the quantification of high-dose exposures is not as developed as it is in the case of stochastic effects.

The RBE factor has a historical origin and, at present, does not fully be applied to the exposure of various organs to different types of radiation. In fact, the RBE was introduced as the relative amount of absorbed dose of a reference radiation (usually 250 kVp X-rays or cobalt-60 gamma rays) required to produce the same magnitude of the same effect as the absorbed dose of the radiation in question in a particular experimental organism or tissue (an RBE >1 indicates the radiation is more effective than the reference radiation). This factor is influenced by both the biological effect (cell killing, cell survival with mutations) and the LET of the radiation.

It looks like, under present circumstances, the best way to call the main quantity for the assessment of the risk associated with the deterministic effects in terms of the RBE dose is defined as

$$RBE \text{ dose} = RBE \times D \tag{2}$$

with the unit Gy-Eq (gray equivalent). Therefore, a dose in Gy-Eq is the absorbed dose in Gy multiplied by a recommended RBE, which takes into account the effects on living organisms caused by radiation of different types and energies.

4.3. Too many quantities and possible solution

The efficient control of radiation exposure is affected by too many quantities, most of which cannot be directly measured or monitored. Moreover, they are inconsistent and complicated as to their definitions which seem to be too theoretical and use some factors which cannot be established.

The problem with quantities is in both assessing the risk and consequences of exposure in humans. This can be attributed especially to the quantification of deterministic effects, but there are also some problems in the evaluation of the probability of stochastic biological effects.

As to stochastic effects, the contribution to the main quantity the effective dose should include a contribution from both external radiation and from internal radioactive contamination, which affects the organs from inside. While for external radiation, the quantity is taken into account all significant organs and the quality of radiation involved, there is no such counterpart if it comes to the assessment of internal effective dose.

The effective dose is supposed to serve as a measure of exposure which has to keep below dose limits introduced by the regulatory authorities, and at the same time, every effort is expected to be made to have the doses as low as under the circumstances possible in compliance with the well-known ALARA principle [16]. In some cases, it is not easy to ensure full consistency with this principle since, for example, in the case of the control of skin exposure the limit is related to the location with the maximum exposure. Of course, this is not known; usually, the dosimeter, the result of which confirms that the limit has not been exceeded, is normally in another place.

There is even a bigger problem with estimating radiation hazards due to the severe exposure where deterministic effects are expected. There is no unified approach in the quantification of the health hazards when it comes to the exposure of several organs above their threshold doses. This applies to both external and internal exposures.

Since it seems that we have now too many quantities currently used, it would be appropriate to reduce their numbers and simplify their definitions, especially for routine applications. The present rather complicated system can continue to be used for research and scientific investigations. Such suggestions were already proposed by the author some time ago [14,17]. A similar approach is implemented in many other branches of science and technology, where a system of quantities used is usually simplified.

5. CONCLUSION

The analysis of illicit drug composition is required for effective actions of Law Enforcement Agencies (LEAs). Determination of major as well as trace elements provides additional parameters that could help in the identification of drug origin. Heroin, cocaine and methamphetamine samples were assayed by instrumental neutron activation analysis for the determination of mass fractions of several elements. For completeness, a set of adulterants used for drug cutting

was also included in the study. The results suggested that INAA with short-time irradiation was especially attractive due to its simplicity and short turnaround time. The instrumental neutron activation analysis is very sensitive and is, therefore, an extremely useful method for analyzing the presence of minor elements in very low concentrations. Such analysis is of great advantage, especially for detecting and identifying trace elements, even in high-purity substances. It can also be applied in forensics sciences to give useful information about the original location of drugs seized by the LEAs identified according to the so-called “fingerprint” of the individual element composition in the suspicious or examined materials. Neutron activation analysis can detect up to about 70 elements.

However, as in any nuclear technique, appropriate methods must be followed here to ensure adequate protection of persons and the environment. This is why the current standards in line with all relevant requirements set by the national regulatory authority have to be strictly observed. Since the INAA technique is based on the use of neutrons, compliance with basic radiation protection requirements is more complicated than in the case of other types of radiation. The response of dosimeters and monitors to neutrons is somewhat tricky because of the complex interaction processes and their dependence on the neutron energy and irradiation conditions where gamma radiation is always present. The individual component of mixed n-gamma radiation complicates the interpretation of the results of measurements since the radiation sensor reacts differently to neutrons and gammas.

This is why radiation protection in any application involving neutron sources is more complicated than in the case of other radiation. In any case, we have to apply all rules and techniques to assess radiation risk under such complex circumstances in order to have reliable information about the doses received by workers involved in the use of INAA. It is, therefore, necessary to apply the radiation protection principles very carefully to any applications where mixed n-gamma radiation has to be adequately measured and monitored to comply with the standards which require the assessment of radiation in line with the latest ICRP recommendations.

Based on an analysis of the present situation, it seems that the radiation protection system has to be simplified to apply it easily in the routine use of radiation where mixed n-gamma fields are too difficult to monitor because of the different responses of the dosimeter to individual radiation components.

In general, the applications of both INAA and RNAA methods is considered to be safe since under normal conditions radiation exposures in terms of effective dose do not exceed the dose limit recommended by such international bodies and organizations as International Commission on Radiological Protection (ICRP), International Commission for Radiation Units and Measurement (ICRU) or International Atomic Energy Agency (IAEA).

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