

# BOOK OF ABSTRACTS

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**INTERNATIONAL CONFERENCE ON  
MODERN TRENDS IN ACTIVATION ANALYSIS**

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# Plenary

## **Chemical quality control of materials by conventional and $k_0$ -based internal monostandard NAA and PGNAA methods utilizing research reactor facilities at BARC, India**

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**Keywords:** chemical quality control, research reactor facilities, BARC, INAA, IM-NAA, PGNAA

Chemical characterization of a material of interest is the first and most important step in chemical quality control (CQC) exercise. It involves quantification of elemental concentrations at major, minor and trace levels by suitable analytical technique(s) with good accuracy and precision. Compared to various conventional analytical techniques, Nuclear Analytical Techniques (NATs) like Instrumental Neutron Activation Analysis (INAA) and Prompt Gamma-ray NAA (PGNAA) are preferred due to their advantages like non-destructive analysis of solid or as received samples, simultaneous multielemental capability from major to trace concentration levels, negligible/less spectral interference and matrix effects and inherent accuracy and precision. It is feasible due to availability of research reactors with higher thermal neutron flux and high-resolution gamma-ray spectrometry using HPGe detector coupled to multi-channel analyzer. In India, NAA of samples using reactor neutrons started since 1956, when the first research reactor of India 1 MWt Apsara commissioned at Bhabha Atomic Research Centre (BARC), Trombay, Mumbai. Presently, BARC has two high neutron flux research reactors namely 100 MWt Dhruva ( $\sim 10^{14}$  n/cm<sup>2</sup>/s) and 2 MWt Apsara-U ( $6 \times 10^{13}$  n/cm<sup>2</sup>/s) and one low neutron flux ( $\sim 10^7$  n/cm<sup>2</sup>/s) research reactor called Critical Facility. In addition to tray-rod, self-serve and Pneumatic Carrier Facilities (PCF), thermal neutron beam facilities ( $10^5$  to  $10^7$  n/cm<sup>2</sup>/s) are available at Dhruva reactor for neutron-based studies including PGNAA. Since 1994, Radiochemistry Division, BARC is engaged in developing  $k_0$ -based conventional and Internal Monostandard NAA (IM-NAA) and PGNAA methods utilizing neutron flux/beam from research reactors at BARC and applying them for chemical characterization of materials in different fields like geology, environment, food and agriculture, archeology, forensic sciences, materials sciences and nuclear technology. Our lab has participated in four IAEA CRPs utilizing PGNAA and INAA methods. The research reactor and gamma-ray spectrometry facilities and developed NAA/PGNAA methods are very helpful for various R&D works and CQC of materials that are difficult by other conventional analytical and radionalytical methods. The materials analyzed are relevant for energy/nuclear energy having complex matrices like alloys, ceramics, oxides, glass and high purity materials. The IM-NAA method in conjunction with in situ relative detection efficiency was developed at BARC to deal with small as well as larger size and non-standard geometry samples

like reactor structural materials namely zircalloys, stainless steels, Ni-alloys an, high purity aluminium and graphite samples for CQC purposes as well as archeological potteries and forensic glass samples for grouping/provenance studies.

### **Acknowledgments**

Author thanks all co-workers and operation crews of research reactors of BARC.

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## **Nuclear analytical activities at NIST: methods, history, user facility, and plans**

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**Keywords:** reactor, activation analysis, INAA, NDP, PGAA, user facility

The nuclear analytical facilities at the 20 MW NBSR reactor include rabbit tubes for instrumental neutron activation analysis (INAA), thermal and cold neutron prompt gamma activation analysis (PGAA), and neutron depth profiling (NDP). These analytical techniques have been employed over the past several decades for the certification of standards reference material (SRM) as the internal mission of NIST. Additionally, as a part of the User Facility at the NIST Center for Neutron Research, these instruments hosted university student and post-docs as well as industrial R&D efforts in the composition analysis and characterization of a wide range of materials and systems. However, the reactor has been down for the past 4 years, interrupting the SRM certification and research effort. This presentation will give a brief review of the history of the nuclear methods group leading up to the shutdown and reports on the various activities in the interim. Samples from planned projects were prepared and sent to other facilities for irradiation and testing. Reconciliation of results from different facilities and techniques post challenges, example of which will be discussed. Research projects on gamma ray imaging methods are continuing -- Compton imaging was carried out with radioactive sources, and ghost imaging was performed with its x-ray analog at a synchrotron radiation light source. A development in PGAA is an expansion of data utility -- instead of the traditional quantitative elemental analysis using individual peaks in the prompt gamma spectra, the library of whole spectra was used to train machine learning models for the purpose of rapid material classification. An on-going effort in both experimental and simulation of portable neutron generator based PGAA for chloride in concrete will also be reported. Finally, we provide an outlook of the facilities in the next few years in conjunction with the reactor cold source upgrade, and the plan for the nuclear analytical capability incorporated into the design of the next generation reactor.

## **Sustainable contributions by NAA and metrology to producers and consumers in Brazil's agronomical landscape**

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**Keywords:** traceability, authenticity, environmental impact

Agriculture has historically been one of the main pillars under Brazil's economy, with an omnipresent influence on daily life, not only within the country but also globally, given its leadership in commodity exports. Innovations in planting, fertilizing, harvesting and production increasingly require sustainable practices minimizing environmental footprint and optimizing yields. The long-term impact of agricultural and industrial activities on Brazil's green legacy, notably the Amazon and Atlantic Forest, along with their natural resources, have gained national and international attention. Addressing these concerns require reliable and robust measurements of undisputable quality. Contamination, health and environmental risks, as well as traceability and authenticity throughout the farm-to-fork chain, are pivotal issues for which authorities, industry, farmers, and the public have turned for many decades to the competence and analytical support from CENA/USP.

Neutron activation analysis has been used at CENA for almost 50 years to provide answers by trace element data. Trustworthy data built on the metrology of the entire chain, from sampling to analysis, supplemented by robust methods for interpretation of the multi-element patterns. The interaction of the various stakeholders in the Brazilian agricultural industry with the NAA program at CENA will be illustrated and discussed by end-user driven case studies. Their current and future needs, together with opportunities forthcoming from analytical and information technology developments lay out the challenges for continuing our efforts.

## Neutron activation analysis in Denmark, the contribution of Dr. Kaj Heydorn

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**Keywords:** neutron activation analysis, quality assurance, uncertainty, medical application, forensics

Dr. Kaj Heydorn, a well know nuclear scientist and dedicated most of his life in methodology and application of neutron activation analysis, passed away on 2<sup>nd</sup> February 2024, just before his 93th birthday. Denmark is the place where neutron activation was originally discovered by George Hevesy and Hilde Levi in 1936, while the wide application of neutron activation analysis was started and bloomed from 1958 led by Dr. Kaj Heydorn after the first 5MW nuclear reactor (DR-2) was established at Risø, Denmark.

Dr. Kaj Heydorn joined the newly established nuclear research centre in Denmark (Danish Atomic Energy Commission Research Establishment Risø, later named Risø National Laboratory) in 1956, and led the section of Isotope Laboratory from 1965 until his retirement in 1998. He is the author of the first report of the series Risø Reports (Risø-R-01) entitled on Radioactivity in Risø Area. He is respected as one of the best-known experts in the field of neutron activation analysis with more than 150 publications and countless remarkable talks at international conferences. His last paper appeared in Trends in Analytical Chemistry in 2015 at the age of 84. The books in analytical quality assurance and neutron activation analysis written by him are also famous. This presentation briefly introduced the research activities of neutron activation analysis with highlights of his major contribution in the methodology and application of neutron activation analysis.

### Acknowledgments

Many documents, photos and information in this presentation were provided by colleagues at Risø as well as other institute all over the world, they are highly appreciated.

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## NAA activities at the JSI, Slovenia

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**Keywords:** NAA,  $k_0$ -standardization method, TRIGA reactor, CRM, validation

Neutron Activation Analysis (NAA) is one of the primary ratio methods [1] frequently used for the characterization of unknown samples or proposed new certified reference materials (CRMs). In principle, it is a bulk analysis technique with panoramic analysis of many elements suitable to interact with neutrons, typically via  $(n,\gamma)$  reactions. Due to its sensitivity, versatility and high reliability, neutron activation analysis is the most prominent of all the activation techniques.

NAA at the Jožef Stefan Institute (JSI), Slovenia started in 1966 with the first criticality of the Institute's 250 kW TRIGA Mark II research reactor. Since then, Institute's researchers have been used this facility for its utilization and for various applications. In addition to instrumental NAA (INAA), radiochemical NAA (RNAA) was also introduced for the determination of some elements in trace and/or ultra-trace levels. Should be mentioned also an important activity at JSI for installation and validation of the  $k_0$ -standardization method of NAA in the early 1990s [2, 3]. The method is mono-standard and uses gold (Au) as the standard and comparator for the simultaneous determination of over 70 % of the elements of the periodic table. The  $k_0$ -method of NAA is continuously improving, along with its nuclear data library, so-called,  $k_0$ -database [4], where JSI colleagues participated to improve the accuracy and reliability of nuclear data. After the method validation, we were able to participate in different studies and international projects including inter-laboratory comparisons (ILCs) at the highest level (CCQM, SIM, APMP, IAEA, etc.). It is also worth mentioning the participation in certification campaigns for various materials (organic and inorganic) with internationally recognized institutions. Some of the more important activities of NAA at JSI will be presented and discussed.

### Acknowledgments

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## **Nano-ayurvedic medicine—Discovery of a new medical modality through nuclear analytical and radiochemical techniques—Preclinical and clinical investigations**

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Cancer continues to be a major public health problem worldwide. In 2022, there were an estimated 20 million new cancer cases and 9.7 million deaths. The estimated number of people who were alive within 5 years following a cancer diagnosis was 53.5 million. About 1 in 5 people develop cancer in their lifetime, approximately 1 in 9 men and 1 in 12 women die from the disease. By 2040, the global burden is expected to grow to 27.5 million new cancer cases with estimated 16.3 million cancer deaths simply due to the growth and aging of the population. The future burden will probably be even larger due to increasing prevalence of factors that increase risk, such as smoking, unhealthy diet, physical inactivity, and fewer childbirths, especially in economically transitioning countries. A number of new therapeutic interventions to combat various forms of human cancer have been developed over the last two decades. However, cures and lifesaving arrestation of this disease have been rare because tumors bear innate characteristics to become resistant to various forms of treatment. It is becoming increasingly clear that various chemotherapeutic, immunotherapeutic and radiation-based treatment modalities activate NF- $\kappa$ B transcription factors, which are responsible for triggering various pro-tumorigenic cascade of processes within the tumor microenvironment. Tumor progression and evasion of systemic immune surveillance are all dictated by significantly high levels of various immunosuppressive factors, such as IL-10, IL-6, and TGF- $\beta$ . Tumor progression is further catalyzed by the immune cells, including regulatory T cells, dendritic cells, MDSCs and TAMs, which are known to express a low level of MHC class I molecules within the tumor microenvironment. Most cancer drugs, in current use which belong to specific targeted or cytotoxic agents, rely on “one gene, one target, one disease” approach despite the fact that cancer is a very complicated multi-target and multi-gene defective disease. However, several examples of phytochemical-based therapeutic approaches have shown the power of cocktail of phytochemicals in traditional Indian Ayurvedic Chinese and African medicines to be multi targeted by enhancing the CD4<sup>+</sup>/CD8<sup>+</sup> T cell ratio in the tumor microenvironment. Herbal-based Ayurvedic medicine are also known to reeducate the macrophage by promoting the M1 differentiation of TAM, suggesting the poly-pharmacological and poly-targeted nature of phyto agents. Sub-optimal bio availability of phytochemical-based therapeutic agents has been a major bottleneck in translating effectiveness of Ayurveda and related herbal-based drugs from cell cultures to cancer therapy agents for human applications.

We have used Neutron Activation Analysis (NAA) and application of radiochemical techniques to develop radioactive ayurveda herbs-functionalized Gold-198 nanoparticles. NAA and scintigraphy have allowed precise estimation of bio localization and bioavailability of tumor specific gold nanoparticles in vivo. Combination of NAA and radiochemical methodologies have provided credible scientific rationale to phytochemical-based herbal (Ayurvedic) medicine—paving the way for the discovery of a new Precision Medicine modality referred to as ‘Nano-Ayurvedic Medicine’. This lecture will discuss how Green Nanotechnology, through nuclear analytical tools, has resulted in the development of reproducible formulations of herbal and classical Ayurvedic medicines thus providing a pathway for clinical trials for internal/external validity, to allow the safety and efficacy of specific herbal medicines in a more accurate and scientifically verifiable way. This lecture will further discuss details on how green nanotechnology can be used to develop small-molecule phytochemical(s)-functionalized gold nanoparticles to simultaneously achieve: (i) Inhibition of NF- $\kappa$ B activation; (ii) Targeting TAM; and (iii) Inhibition of TNF- $\alpha$  induced p65 phosphorylation; and concomitant immunomodulatory therapeutic action. Details on the discovery of a new medical modality, referred to as ‘Nano-Ayurvedic Medicine’, recently approved by the US Patents and Trademarks Office will be presented (1,2). The lecture will also highlight the importance of clinical translation in medical research through recent results from human clinical trials on cancer patients of Nano-Ayurvedic Nanomedicine drugs derived through green nanotechnology (1,2).

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## Radiochemical and preconcentration neutron activation analysis: Recent achievements, present status and trends

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**Keywords:** radiochemical neutron activation analysis, preconcentration neutron activation analysis, separation procedures, speciation analysis

Radiochemical neutron activation analysis (RNAA) dramatically improves the selectivity of signal measurement and result in improvement of limits of detection (LOD) of many elements by several orders of magnitude compared with instrumental neutron activation analysis (INAA). Similar improvements can also be achieved using preconcentration (preseparation) neutron activation analysis (PNAA). Therefore, both RNAA and PNAA are extremely useful for trace- and ultratrace determination of a number of elements in a variety of matrices. Merits and demerits of both NAA modes, recent achievements and trends are reviewed based on articles published since 2000.

Most RNAA procedures have been developed for determination of trace- and ultra-trace levels of a number of elements, such as Ag, As, Au, Cd, Co, Cr, Cs, Cu, Fe, halogens (mostly Br, Cl, I), Hg, Mn, Mo, Ni, P, platinum group elements (PGE, namely Ir), rare earth elements (REE), Rb, S, Sb, Si, Sn, Sr, Th, U, V, W, Zn in a variety of matrices, mostly of biological, environmental, geo- and cosmochemical, nutritional, and medical origin, in high-purity materials and especially in low-level reference materials of various matrices. The above elements have been separated either in single element fractions or group separations were performed from one test portion, especially for halogens, PGE and REE. Sample decomposition procedures varied according to matrices assayed and involved mainly acid digestion with strongly oxidizing agents (mineral acids and their mixtures, possibly with  $\text{H}_2\text{O}_2$  addition) in open or closed, pressurized systems (the latter frequently with micro-wave assisted heating), fusion in various fluxes, and combustion in air or oxygen with subsequent trapping of the combustion products. For separation, most frequently used traditional procedures comprised ion-exchange chromatography using both inorganic and organic exchangers, liquid-liquid extraction, extraction chromatography, solid phase extraction using commercially available or custom-tailored resins, precipitation and co-precipitation, electrolytic deposition, etc. Some novel procedures were also applied, such as such separation using graphen modified with  $\text{MnO}_2$ .

PNAA is especially advantageous due to no radiation exposure of personnel and no time constraints. Therefore, PNAA procedures were mainly developed for determination of elements forming short-lived radionuclides, such as Al, V, and Cu. The formerly considered risk of losing the blank-free nature of NAA is not that critical nowadays, provided that all operations are carried out in a clean laboratory, the number of operations is minimized, and high-purity laboratory ware and chemicals are used. An especially appreciable feature of PNAA is that in addition to determination of the total element contents, this NAA mode also allows for speciation analysis [A. Chatt, this conference], not accessible in RNAA. A combination

of pre- and post-irradiation separations proved a further LOD improvement of REE determination and is mandatory for  $^{129}\text{I}$  determination by NAA. Interesting application of PNAA has also been reported for “inverse” preconcentration – a complete removal of well-activated matrices, such as Fe ores or U compounds, allowing trace element determination, the latter being important for nuclear forensics.

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## Numerical study on the characterization of FeNdB permanent magnets with fast-neutrons induced ( $n, n' \gamma$ ) reactions

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**Keywords:** inelastic scattering, fast neutrons, Monte Carlo, prompt gamma-rays, characterization, magnets

Neodymium iron boron NdFeB permanent magnets are a critical component of modern technologies and their recycling is of strategic importance in view of sustainable handling of valuable resources. Such magnets contain the rare earth elements neodymium, praseodymium and dysprosium which are classified together with 37 other elements and 9 substances as critical raw materials in the European Union's Critical Raw Materials Act. An efficient recycling requires in front end a sorting of the magnets according not only to their type but also to their rare-earth elements content. In industrial scale this is a new challenge to take up because the analysis needs to be done rapidly and on bulk dense materials. The traditional methods used for determination of rare-earth elements content, such as ICP-MS or -OES, XRF, LIPS or PGNAAs with cold or thermal neutrons reach their performance capabilities in such applications. These limits are attributed to either the necessity to dissolve part of the sample or due to attenuation of the interrogation beam in bulk and dense material flows. Prompt Gamma Analysis based on Inelastic Neutron Scattering (PGAINS) could be suitable for a rapid determination of the elemental composition of permanent magnets. This method measures the element specific gamma-rays induced by the inelastic scattering of fast neutrons using a spectrometer, allowing to unfold qualitative and quantitative information about the magnet elemental composition. As a non-destructive analytical tool it requires design of the corresponding measurement system, which includes a neutron source, measurement cell and detectors used to measure the prompt gamma-ray spectrum. However, to achieve a high qualitative and quantitative performance of the measurement system, the impact of various parameters on the fast neutron inelastic scattering physics needs to be well studied and understood. Among such parameters are optimal detector type and configuration, flux parameters and minimal required measurement time. In this work, we investigate the detection of neodymium and dysprosium in NdFeB magnet samples by means of numerical simulations in PHITS code using a beam of 2.5 MeV neutrons. The gamma-ray spectra are collected either with a high-resolution HPGe-detector with 50 % efficiency or an array of 8 cadmium zinc telluride (CZT) detectors. The physical principle of the method is described in detail, results of the simulation as well as the conditions necessary to perform such characterization are presented.

### Acknowledgments

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## **An overview of the development of neutron activation analysis at the Nuclear Technology Development Centre, Belo Horizonte, Brazil**

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**Keywords:** neutron activation analysis,  $k_0$ -standardization method

The Laboratory for Neutron Activation Analysis (LNAA) is located at Centro de Desenvolvimento da Tecnologia Nuclear (Nuclear Technology Development Centre) sponsored by Comissão Nacional de Energia Nuclear (Brazilian Commission for Nuclear Energy), CDTN/CNEN, in Belo Horizonte, Minas Gerais. In this Laboratory, the activities that have been developed since the starting up of the IPR-R1 TRIGA Mark I research reactor in 1960 are described from a historical point of view. Delayed fission neutron analysis was the first method to use the reactor as an analytical tool. Over the years, the establishment of new procedures of neutron activation analysis was stimulated to provide the analysis demand, meeting the clients' analytical needs and researches developed by LNAA, by other departments of CDTN and by other institutions. Throughout the time, the work has been linked to the goals of the country and the institutions.

The activities of the LNAA have already comprised the delayed fission neutron activation analysis, instrumental (comparative and  $k_0$ -method) and radiochemical methods. However, the procedures have been changed according to modifications of infrastructure such as acquisition of better instrumentation, and profile of the team of technicians. Nowadays, delayed fission neutron activation analysis and relative method to determine few elements are still applying. Concerning the  $k_0$ -standardization method it is the most applied method determining several elements in various matrixes and range of concentrations.

The applications of neutron activation analysis, mainly  $k_0$ -method will be briefly described as the new approaches, for instance, to analyse large samples and samples with unusual shapes will be mentioned. The actions toward quality control will be discussed. In this presentation, the importance of neutron activation analysis to several fields of science, contributions to nuclear knowledge and education will be shown.

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## Current activities at HANARO research for metrological aspect in Korea

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**Keywords:** reference materials, Neutron Activation Analysis, HANARO

Access to research reactors worldwide has worsened, and despite the crucial role of neutron activation analysis (NAA) in quantitative analysis, its usability appears sluggish or is declining. This decline can be attributed to the emergence of instrumental analysis devices boasting higher analytical sensitivity and improved accuracy compared to the NAA. Factors such as the limited accessibility of NAA, where users lack direct control over the analysis process, contribute to this trend. Despite these challenges, NAA remains a fundamental method acknowledged by the International Bureau of Weights and Measures/CCQM, retaining a significant position in national weights and measures standards. To this end, the NAA group at the HANARO research reactor participates in the IAEA Proficiency Interlaboratory Test and strives to maintain analysis capabilities and analysis personnel. And, we are focusing on supporting the development of inorganic standard materials in various fields, including advanced industrial materials like semiconductors, geology, food, and environments and so on through cooperation with KRISS, a national metrology organization. The neutron characteristics of the reactor underwent rigorous characterization, leading to the establishment and validation of diverse analysis techniques. These include the k<sub>0</sub>-standardization method, internal mono-standard method, and the relative method. In addition to the aforementioned efforts in NAA, my presentation will highlight various ongoing activities at HANARO research reactor for metrological aspect in Korea.

### Acknowledgements

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## The High-Brilliance Neutron Source project

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**Keywords:** neutron source, CANS, HiCANS, HBS

Accelerator driven neutron sources provide a cost-efficient and attractive alternative to classical neutron sources like fission reactors and spallation sources. With the advent of high current proton accelerator systems, a novel class of such neutron facilities can be established termed High-Current Accelerator-driven Neutron Sources (HiCANS). Such sources can counteract the increasing shutdown of existing fission-based neutron sources and a decline in available neutron beam days in Europe.

The High Brilliance neutron Source project (HBS) [1, 2] at the Forschungszentrum Jülich develops such a HiCANS facility. It utilizes a 70 MeV and 100 mA pulsed proton linear accelerator providing tailored proton pulses with frequencies of 24 Hz or 96 Hz up to three optimized target stations. Due to the low energy nuclear reactions releasing neutrons from a tantalum target, the target stations are compact in comparison to spallation neutron sources, requiring only 4% of the shielding material. It allows for an efficient neutron production, moderation and extraction and thus allowing competitive neutron instrument performances.

A detailed technical design report describing all relevant components ranging from accelerator, target, moderators up to the instruments was published recently. Due to the flexibility and modularity in target station design, high performant instruments for basic applications as well as specialised applications for activation analysis can be built. It shows a potential national neutron source facility with up to 24 instruments for all kinds of applications.

We will present the current status of the High-Brilliance Neutron Source (HBS) HiCANS project. The next steps and milestones for this next generation neutron source will be reported as well as the performance of instruments for activation analysis will be estimated.

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# Oral Presentations

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## AA facilities used by the industry, industrial applications

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### Investigation of the $^{99}\text{Mo}$ production via neutron capture $^{98}\text{Mo}(n,\gamma)^{99}\text{Mo}$ with a high-current accelerator-based neutron source

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**Keywords:** compact accelerator-based neutron sources, medical isotope production, activation

The demand for  $^{99\text{m}}\text{Tc}$ , the most widely used radioisotope in medical imaging, is steadily increasing, with approximately 80% of all nuclear medicine procedures relying on it [1]. The precursor,  $^{99}\text{Mo}$ , is primarily produced through the fission of  $^{235}\text{U}$  in high neutron flux reactors [2]. This poses challenges in supply chain disruptions and radioactive waste management and as aging reactors face potential shutdowns, alternative production methods become crucial.

This study explores an alternative approach to  $^{99}\text{Mo}$  production utilizing a high-current accelerator-based neutron source, as proposed in the ambitious HBS (Jülich High Brilliance Source) project [3]. The method involves the  $(n, \gamma)$  reaction with fast neutrons generated by 70 MeV protons interacting with a tantalum target, moderated in water and reflected by lead. Numerical simulations, employing the PHITS code, investigate various target parameters and proton beam currents while limiting the target power density to 3 kW/cm<sup>2</sup>.

Compared to traditional methods, this accelerator-based neutron source offers potential advantages, such as simplified processing schemes and reduced radioactive waste. The study assesses the thermal and epithermal neutron flux, crucial for  $^{99}\text{Mo}$  activity, considering different target and molybdenum plates' surfaces and thicknesses. Analytical methods, incorporating corrections for thermal and epithermal neutron self-shielding, complement the simulation results.

As current reactor-based  $^{99}\text{Mo}$  production faces challenges, especially in regions like Germany with substantial demand [4], the pursuit of alternative, sustainable methods gains significance. The ongoing developments in accelerator-based technologies, exemplified by the HBS project, signify a promising avenue for securing the supply of critical medical isotopes, addressing both efficiency and waste management concerns. This work contributes valuable insights into the feasibility and optimization of  $^{99}\text{Mo}$  production through accelerator-based neutron sources, paving the way for future advancements in medical radioisotope production technology.

**Acknowledgments**

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## Applications of Activation Analyses

### Nuclear and complementary analytical methods for elemental abundances investigation of rock samples from Egypt

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**Keywords:** INAA, XRF, elemental composition, rocks, geochemical provenance, multivariate statistical analysis

The present work was carried out to describe and characterize the elemental composition of the rocks with regard to geochemistry and origin. The study emphasizes the problem of integrating results from different analytical methods. For this purpose, a total of 29 rock samples were taken from 8 profiles of Gabal El-Sela in the southeastern part of the Eastern Desert in Egypt. It is covered by basement rocks of different compositions (Abouelnaga et al., 2014). The rock samples were subjected to instrumental neutron activation analysis (INAA) and a complementary analytical technique, X-ray fluorescence (XRF). The rock samples were analysed in the Institute of Nuclear Physics, Almaty, Kazakhstan. The combined data were statistically analyzed using univariate and multivariate statistical analysis. The results showed that the maximum mass fraction in mg/kg for Si was found to be  $(31 \pm 0.6)$  % at site #P1. The lowest value was determined for Sc at site #P5 with  $0.99 \pm 0.04$  mg/kg. The results are normalized to the corresponding values of the upper continental crust UCC by Rudnick and Gao (2014) that showed that all elements are higher than indicated for UCC, with the exception of Al, Si, Ca, Sc, Ti, Fe, Sr, Cs, Ba and La. Multiple ratio indicators and discriminative diagrams were utilized to decipher the provenance of the rocks and demonstrate that the rocks are different as the rocks have different trends like igneous, shale, and sedimentary trends. In addition, the rocks have significant values of Th and Zr indicating that the samples are a mixture of granite and felsic acid rocks. The study proved that there is no significant variation in the results and that the integration of INAA and XRF results is appropriate when prior treatment of outliers and normality is ensured. The results of the current research can serve as baseline data for the investigated area in terms of the elemental composition of rocks and it has a great economic and industrial benefit for Egypt.

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## Multi-elemental contamination study of soils adjacent to iron and steel industry by INAA and XRF

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**Keywords:** soils, iron and steel, contamination, INAA, XRF, health risk

Many industrial practices, such as iron and steel production, may increase the risk of soil contamination with potential toxic elements in the vicinity of industrial installations, due to specific emissions, processing of minerals, iron ores and coal, and transportation and disposal of industrial wastes and metal-bearing materials. The management of soil quality in the vicinity of integrated steel enterprises is very important for ecosystems and human health and sensitive techniques should be employed for accurate assessment of chemical elements (heavy metals, toxic elements, rare earths, radioelements) in soil and further evaluation of potential ecological and health risk. In this paper multielemental and non-destructive analytical techniques were used in combination for the elemental quantification in soils located around a large integrated iron and steel works in Galati, SE Romania: instrumental neutron activation analysis (INAA) and X-ray fluorescence analysis (XRF). INAA was applied at IBR-2 nuclear reactor of Frank Laboratory of Neutron Physics (FLNP), Joint Institute of Nuclear Research (JINR) at Dubna and energy-dispersive XRF at “Dunarea de Jos” University of Galati, Romania, using an INNOV-X Alpha series XRF spectrometer, Innov-X Systems, Inc. The total concentrations of 42 major, minor and trace elements (Na, Mg, Al, Si, Cl, K, Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Zn, As, Br, Rb, Sr, Zr, Mo, Sb, Cs, Ba, La, Ce, Nd, Sm, Eu, Tb, Dy, Yb, Hf, Lu, Ta, W, Au, Th, U, Cu, Pb) were determined in industrial topsoil (0-5 cm depth). The distribution patterns indicate inputs of toxic metals and radioelements in the sites close to the slag dump, blast furnaces, steel plants and agglomeration and sintering factories. For selected elements, a comparison with world and legislated concentration values in soil was performed and contamination and health risk indices were assessed in the SE region of Romania affected by metallurgical industry, which might have important impact on underground water, aquatic resources and contamination of vegetation and crops.

### Acknowledgments

The support of JINR-Romania programme, FLNP JINR theme No: 03-4-1128-2017/2022 “Investigations in the Field of Nuclear Physics with Neutrons”, is highly acknowledged.

## Study of spectroscopic properties of $^{108}\text{Ag}$ using the $^{107}\text{Ag}(n_{\text{th}}, 2\gamma)$ reaction

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**Keywords:** neutron capture, gamma spectroscopy

Determination of the accurate values for gamma transitions, level scheme, nuclear level density and radiative strength function is crucial in low-energy nuclear physics. Accurate experimental values of those parameters are very important for the analysis of astrophysical reactions, production of medical isotopes and rare isotope beams, reactor technology, etc. The two-step gamma cascade method, involving the detection of gamma-gamma coincidences following thermal (cold) neutron capture, ie. the  $(n_{\text{th}}, 2\gamma)$  reaction, has proven to be a suitable technique for obtaining spectroscopic data and insight into level density and radiative strength functions[1-3]. The investigation of the spectroscopic properties of  $^{108}\text{Ag}$  nucleus using an enriched (99.07%)  $^{107}\text{Ag}$  target was conducted at the PGAA station of the Budapest Neutron Centre, Budapest, Hungary, with a cold neutron beam, 3 HPGe detectors with appropriate shielding and acquisition system for coincidence measurements. In this talk, a brief overview of the method will be presented, as well as the spectroscopic results for  $^{108}\text{Ag}$  nucleus obtained through  $^{107}\text{Ag}(n_{\text{th}}, 2\gamma)$  reaction, focusing on the information on gamma transitions and level scheme.

### Acknowledgments

This project has received funding from the Euratom research and training programme 2014–2018 under grant agreement No 847594 (ARIEL).

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## Accelerator produced $^{233}\text{Pa}$ for nuclear forensics analyses?

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**Keywords:** Pa-233, isotope production, nuclear forensics

Nuclear forensics' mission is to identify the origin and history of nuclear materials found outside of regulatory control through the examination of the confiscated materials using various analytical techniques. [1] Radiochronometry, or age dating of nuclear material, is an important forensic examination technique that provides information about the production or last purification date of the material. [2] During production, the nuclear material is purified to a certain level by removing impurities including the progeny isotopes. Thus, performing the analyses of the parent and progeny isotopes, it is possible to estimate the age of the nuclear material. These are referred to by the forensics community as “model ages” or “model separation dates”, and are ideal values calculated assuming complete removal of progeny isotopes during production, and no loss or gain of parent or progeny isotopes., Radiochronometry systems often used to determine model ages include -  $^{234}\text{U}/^{230}\text{Th}$ ,  $^{235}\text{U}/^{231}\text{Pa}$ , and  $^{241}\text{Pu}/^{241}\text{Am}$ .

In the past decade,  $^{235}\text{U}/^{231}\text{Pa}$  model age measurements have significantly improved and became a vital part of nuclear forensics investigations resulting in increased demand for  $^{233}\text{Pa}$  as an isotope dilution tracer used in the determination of  $^{231}\text{Pa}$ . [3] This isotope has short half-life (27 days), and it is typically produced and calibrated at Los Alamos National Laboratory by separating it from its  $^{237}\text{Np}$  ( $2.144 \times 10^6$  y) parent. Due to the short half-life of  $^{233}\text{Pa}$  and the long half-life of  $^{237}\text{Np}$ , the availability of sufficient amount of  $^{233}\text{Pa}$  tracer is limited and no longer satisfies the increasing demand.

In our study, we investigated an alternate  $^{233}\text{Pa}$  source, obtained as a by-product from proton irradiation of thorium targets for production of  $^{225}\text{Ac}$ . [4] We characterized the  $^{233}\text{Pa}$  fraction from two different production experiments using gamma, alpha and mass spectrometric techniques and conducted a feasibility study to investigate utility of this material as tracer for nuclear forensics analysis.

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## Validation of MCNP and FISPACT calculations to predict the activation of medical radioisotope target materials in the Budapest Research Reactor

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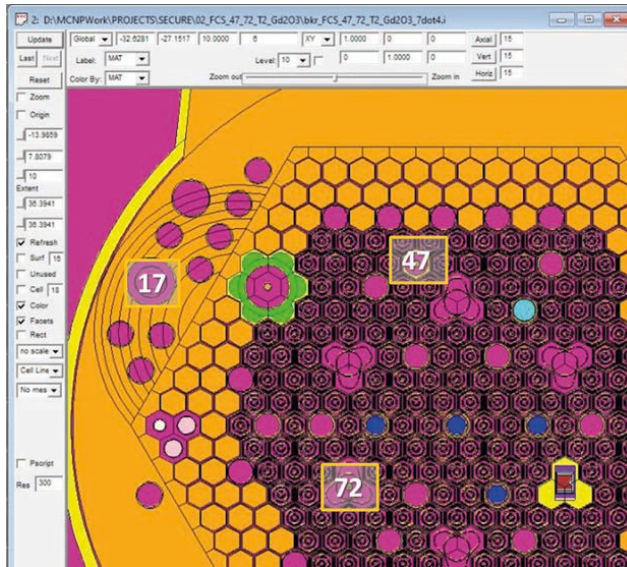
**Keywords:** medical isotope, neutron activation, MCNP simulation, FISPACT calculation, validation

The aim of the SECURE project is to make a major contribution to the sustainability of medical isotope production and its safe application in Europe. That includes target development, and reliable solutions for production methods of therapeutic and diagnostic radionuclides for  $\alpha$  and  $\beta$  emitters. Our group is involved in the task “Feasibility calculation of producing beta-emitting radionuclides via MCNP6 and FISPACT software”. Using the full-scale MCNP6 model of the 10 MW Budapest Research Reactor (BRR), we characterized several vertical channels.

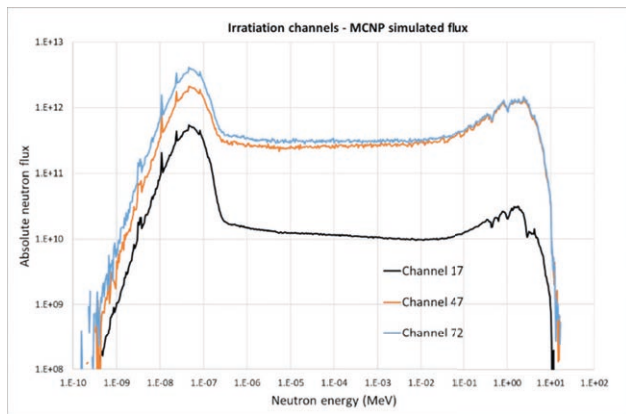
Three vertical irradiation channels with highly different neutron-energy distributions were selected for the validation experiments (Fig.1.). The MCNP F4 tally output data were tabulated according to the CCFE-709 group energy structure, one of the native energy-bin sets of FISPACT, ranging from  $10^{-5}$  eV to  $10^9$  eV. Using the neutron field parameters and the composition of the target, a radioisotope inventory for any time instant during and after the irradiation can be obtained by using the FISPACT-II code.

Initially, we used the FISPACT’s default TENDL-2017 nuclear data library for our calculations, later the calculations were repeated with the ENSDF/B.VIII data. We identified a few discrepant cases, while most of the results were in good agreement.

Gd, Pd, Pt, and Lu-containing samples were prepared for neutron activation in the Nr. 17, 47, and 72 vertical channels, to produce Tb-161, Ag-111, Au-199, and Lu-177 radionuclides, respectively. The experimental activities of the irradiated samples were measured using well-calibrated HPGe detectors placed inside low-level iron counting chambers. The results were compared with the FISPACT calculations and an agreement within 30% was found. The method can be generalized to assess the yield for any yet untested irradiation site.



a)



b)

**Figure 1:** a) MCNP visualization of the reactor core and the selected vertical irradiation channels for the validation experiments. b) Visualization of the neutron energy distribution based on the MCNP F4 tally output data.

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## Elemental composition and radionuclide content of diagenetic manganese nodules from the Pacific deep sea

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**Keywords:** neutron activation, neutron self-absorption, manganese nodules, high cross-section materials

Due to their high content of manganese and other metals, manganese nodules represent a largely untapped reservoir of economically relevant raw materials. These nodules have the potential to become particularly relevant for energy production in the near future.

The aim of this work was therefore to use neutron activation analysis and gamma spectrometry to determine the chemical and isotopic composition of diagenetic manganese nodules from the Clarion-Clipperton Zone and the Peru Basin. Because of their low growth rate, they can also be used as time capsules and radiochemical equilibria can be used to obtain information about marine chemistry. Neutron activation analysis of high-manganese samples faces certain challenges due to neutron self-absorption within the sample. In order to overcome these challenges, neutron flux depression needed to be quantified and carefully minimized using dilution of the finely ground sample material.

In this presentation, we will discuss analytical challenges and present the results of our radioanalytical and chemical characterization of one manganese nodule.

## Validation of Tb-161 activation calculations using $k_0$ -NAA

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**Keywords:** neutron, activation, self-shielding, calculation

Terbium-161 is an emerging radioisotope with significant potential as a future cancer treatment[1]. It can be produced by neutron irradiation of gadolinium via the  $^{160}\text{Gd}(n,\gamma)^{161}\text{Gd} \rightarrow ^{161}\text{Tb}$  reaction and high specific activities can be achieved after post-irradiation separations. Targets isotopically enriched in  $^{160}\text{Gd}$  are a necessity due to the very high neutron capture cross-sections of other gadolinium isotopes, particularly  $^{155}\text{Gd}$  (60740 b) and  $^{157}\text{Gd}$  (252930 b). Even when highly-enriched target materials are used, where these undesirable isotopes have been significantly depleted, remnant trace-level quantities of these isotopes still result in not insignificant levels of neutron self-shielding, reducing achievable yields. Predicting post-irradiation activities are further complicated by the fact that the intermediate nuclide  $^{161}\text{Gd}$  (3.7 min half-life) also has a high neutron capture cross-section of 11752 barns.

Several gadolinium oxide samples were analysed by neutron activation analysis (NAA) using the  $k_0$ -method of standardisation[2] to test the accuracy of the self-shielding corrections[3] commonly in use. One natural and two isotopically enriched samples of identical mass and geometry underwent short-irradiation NAA, utilising gamma-rays emitted by both  $^{161}\text{Gd}$  and  $^{161}\text{Tb}$ . An additional natural gadolinium oxide sample of identical mass but different geometry was also analysed. Formulae were modified to enable isotopic rather than elemental measurements. Utilising short irradiations reduced the number of neutronic effects that would need to be accounted for in longer irradiations, namely the “burn-up” of intermediate ( $^{161}\text{Gd}$ ) and interfering nuclides.

Activation calculations were then performed for longer-term, higher flux irradiations designed to produce gigabecquerel quantities of  $^{161}\text{Tb}$ . Activation and burn-up of all present nuclides was accounted for, revealing a significant reduction in the quantities of  $^{155}\text{Gd}$  and  $^{157}\text{Gd}$  as irradiation progressed. These nuclides are responsible for the majority of the neutron self-shielding. Typically neutron self-shielding factors are calculated once, based on the initial quantities of isotopes present in target material and the assumption that these do not reduce appreciably over the duration of the irradiation. In this work self-shielding was recalculated on a continuous basis to account for the evolution of the target during long irradiations. Calculations showed that neutron self-shielding effects can change significantly during relatively long irradiations and calculating the self-shielding correction factors only once based on the initial target compositions can result in underestimated activities. Finally, predictive calculations were compared to the measured activities of actually irradiated enriched gadolinium targets.

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## Comparison with other analytical techniques

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### Benchmarking handheld XRF spectroscopy against PGAA, NAA for the elemental composition analysis of electronic waste

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**Keywords:** instrumental neutron activation analysis, prompt gamma neutron activation analysis, handheld X-ray fluorescence spectroscopy, electronic waste

Nowadays, the waste of electrical and electronic equipment (WEEE) has become one of the major environmental problems. The recycling of such a waste type can significantly reduce its negative impact on the environment, while it can also become an important secondary resource of many elements for the industry. The elemental composition of WEEE is typically unknown and varies from manufacturer to manufacturer. However, this knowledge is necessary for designing an efficient, economical, and environmentally friendly recycling technology. In this case, it is required to determine the elemental composition of these wastes before recycling, with a particular focus on hazardous elements, valuable elements or elements with a high supply risk, which all to be recovered. The neutron-based analytical techniques, such as INAA and PGAA, have advantages which make these techniques suitable to contribute to this task.

They are multielement techniques for the simultaneous determination of the concentrations and detection limits of several elements. These techniques are capable of determining the composition in solid samples without preliminary dissolution. They provide information on the volume average composition due to the high penetration capability of neutrons and gamma rays. They are typically free of matrix effects, or these effects can be corrected. However, it is difficult to implement these methods in an industrial environment. On the other hand, handheld X-ray fluorescence spectroscopy (hh-XRF) is an affordable and fast technique, suitable for the simultaneous determination of the concentrations of many elements in solid samples and the applicability of this technique in industry is much easier than that of neutron-based techniques. However, this technique has many limitations, such as limited sampling depth of X-rays and matrix effects that are much more significant with this method than with any neutron-based techniques.

In this work, the comprehensive study is reported on the elemental composition of different WEEE types, such as LED chips and printed circuit boards. The concentrations were determined with a combination of INAA and PGAA. The applicability of the hh-XRF technique to determine the elemental composition of these wastes was assessed using the results of neutron-based methods as a reference. These devices are typically used with matrix-matched calibration modes provided by the manufacturer for qualitative and quantitative analysis. However, for the comprehensive analysis of the elemental composition of different WEEE, no suitable factory calibration is available to this date. Based on the measured XRF spectra

and the INAA and PGAA results, we identified elements that are measurable with hh-XRF in the analyzed waste types. The possibility of developing our own matrix-matched calibration for quantitative analysis is also being investigated, by using the peak areas determined from hh-XRF spectra based on the concentrations measured with neutron-based methods.

**Acknowledgments**

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## Do we need INAA in nuclear forensics? Comparison of the analytical performance of INAA and ICP-MS in the nuclear forensic investigation

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**Keywords:** nuclear forensics, elemental analysis, INAA, ICP-MS

Nuclear forensic analysis, involves examining nuclear materials, other radioactive materials that contain radioactive substances, or evidence that is contaminated with radionuclides, in the context of legal proceedings under international or national law related to nuclear security. The purpose of analysing nuclear or radioactive material is to identify its composition, origin, and intended use. This includes both radiometric and non-radiometric measurement techniques. Elemental analysis is one of the characterisation methods used in forensic analysis, ICP-MS and ICP-OES are most commonly used for this purpose.

In this work, INAA was applied to characterizing uranium ores, determining impurities in uranium compounds and impurities in additional materials, eg. packaging material. Neutron activation was performed in a MARIA nuclear reactor (Poland) with a thermal neutron flux of  $10^{14} \text{ n}\cdot\text{cm}^{-2}\cdot\text{s}^{-1}$ . Depending on the sample, the activation time ranged from 5 min (stainless steel) to 1 h (geological samples). The cooling time was 1–10 days, measurement time: 10 min. – 5 h.

The comparison of the applicability of INAA and ICP-MS for the study of the chemical composition of these matrices was carried out.

### Acknowledgments

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## Comparative analysis of Florida chert using LA-ICP-MS and INAA

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**Keywords:** INAA, LA-ICP-MS, chert

The present work will discuss the analytical aspects of a study that is attempting to assess the provenance for a large collection of archaeological specimens composed of silicious tool-stone (chert), including raw materials and museum artifacts, using elemental characterization. These artifacts cannot be subjected to destructive analyses. Laser ablation ICP-MS (LA-ICP-MS) has been demonstrated to be a worthwhile technique for effectively non-destructive, discriminate analysis of geological materials. LA-ICP-MS has relatively low throughput, however, and, as a microprobe technique, is subject to bias due to sample inhomogeneity; therefore, we are using INAA and LA-ICP-MS as complementary methods. The INAA work will accommodate a large number of field samples, and will likely improve the discriminatory power of the analysis versus relying solely on LA-ICP-MS. Additionally, matrix-matched LA-ICP-MS standards will be characterized by INAA with a goal of improving the accuracy of the former method.

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## Fast Neutron Activation Analysis

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### High-energy neutron activation analysis of various target materials

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**Keywords:** quasi-monoenergetic neutrons, induced reactions, gamma-ray spectra, background spectrum

The iThemba Laboratory for Accelerator-Based Sciences (iThemba LABS) in South Africa offers a unique neutron beam experimental facility capable of providing quasi-monoenergetic neutron beams with an energy range of 25 MeV to 200 MeV [1]. Quasi-monoenergetic neutron beams at iThemba LABS are produced in the neutron beam vault via the (p,n) reaction on thin Li and Be targets [2]. The team at iThemba LABS is currently involved in a joint research project focused on measuring high-energy neutron induced cross-sections for the (n, xn) reactions using various target materials that are important for reactor dosimetry, fusion and fission studies. Currently, there are missing experimental data for high-energy neutron cross-section libraries of (n,xn) reactions in these materials. Additionally, the existing limited experimental data disagree with theoretical models [3]. At neutron energies below 30 MeV, the cross section for these nuclear reactions are considered to be well-known. Test experiments commenced at the iThemba LABS facility with irradiations of various target materials using quasi-monoenergetic neutron beams at high energies.

For this contribution, we report on the neutron activation analysis of the following target materials; Co, Au, Tm and Bi using a quasi-monoenergetic neutron beam of about 90 MeV and 140 MeV. The discussions will focus on whether the resulting gamma-ray spectra can be clearly distinguished from the background levels of the counting system. This will include calibration of the system using certified reference sources and analyses of the gamma-ray spectra to identify the (n,2-6n) reactions for each activated material. Further analysis of the gamma-ray spectra will include the estimation of the activity uncertainties. For cross-section determinations, the following parameters; neutron peak fluence, peak to continuum ratio in the neutron spectrum, neutron fluence monitor and the activities from the gamma-ray spectrum will contribute to the uncertainty budget.

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## Fast Neutron-induced Gamma-ray Spectrometry (FaNGaS)

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**Keywords:** inelastic scattering, fast neutron, cross section, gamma ray, detection limit

Prompt Gamma Neutron Activation Analysis (PGNAA) based on cold or thermal neutron capture is a powerful technique for non-destructive elemental analysis of small and thin samples. However, due to limited penetration and attenuation effects, PGNAA is not suited for a precise investigation of large objects. The feasibility of Prompt Gamma Analysis based on Inelastic Neutron Scattering (PGAINS) to determine the elemental composition of large samples was already demonstrated several decades ago [1]. The FaNGaS (Fast Neutron-induced Gamma-ray Spectrometry) instrument, installed at Heinz Maier-Leibnitz Zentrum (MLZ) in 2014, advances this non-destructive analytical technique and makes it available for a broad community of industry and research [2-8]. Using the intense fission neutron beam delivered by the research reactor FRM II (Forschungs-Neutronenquelle Heinz Maier-Leibnitz) to investigate fast-neutron induced prompt gamma-ray emission, it offers new possibilities for the chemical analysis of large or small samples as a complementary method to conventional thermal- or cold-neutron based PGNAA. The predominant reaction channel of fast neutrons at FaNGaS is the  $(n,n'\gamma)$  inelastic scattering reaction, currently with only one existing database: the “Atlas of Gamma-rays from the Inelastic Scattering of Reactor Fast Neutrons”, published in 1978 by Demidov et al. [9]. This data compilation is valuable and a relational database has been recently developed based on this Atlas [10]. However, it was yet never validated and previous measurements with FaNGaS show the need for a critical and meticulous validation [3-6,8]. Apart from building up a comprehensive catalogue of  $(n,n'\gamma)$  reactions another main objective is a continuous optimization of the instrument to improve the analytical sensitivity. In this talk the experimental set-up and technical specifications of FaNGaS will be given. Relative intensities and partial gamma-ray production cross sections of fast-neutron-induced prompt gamma rays derived from the measurement of various elements will be presented along with literature comparisons.

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## Instrumental Neutron Activation Analysis

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### The system for certification of reference materials in Korea using standard comparator neutron activation analysis

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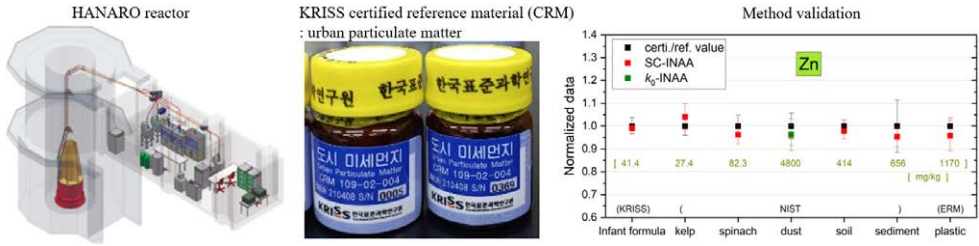
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**Keywords:** metrology, standard comparator NAA, certified reference material, certification

Instrumental neutron activation analysis (INAA) is a robust analytical technique for determining multi-elements in various matrix samples. INAA exhibits high metrological characteristics, particularly when based on the standard comparator (SC), along with isotope dilution-mass spectrometry (ID-MS). These methods, well-known as primary ratio methods<sup>1</sup>, are regularly employed for certifying inorganic elements in certified reference materials (CRMs). The standardization of analytical methods for characterizing reference materials (RMs) is a challenging issue to maintain high qualities such as accuracy, traceability, and uncertainty, ensuring competitiveness in laboratory results. In Korea, the Korea Research Institute of Standards and Science (KRISS) is a national metrological institute that has established an ID-MS method to certify the mass fractions of inorganic elements in matrix CRMs. However, for mono-isotopes (Na, Al, Sc, As, Co, etc.), the application of the ID-MS method is fundamentally impossible, and for certain matrices, dissolution is difficult. These challenges can be overcome through the INAA method.

Recently, the KRISS-KAERI Joint Research Center was established for the development of RMs using a research reactor. Currently, our focus is on developing CRMs in advanced materials, food matrices, geology, the environment, and matrices requested by customers. In this study, a system has been established for the certification of elements in candidate RMs using the SC-INAA method, including sample preparation, measurement, validation, and uncertainty calculations. We will describe the difficulties encountered during the establishment of the primary INAA method, such as reactor neutron flux, detection system parameters (efficiency, P/T ratio, FWHM, pile-up, effective depth distance), and contamination issues in comparator preparation. Additionally, we will provide examples of applying the SC-INAA method to KRISS CRM certification.

Figures



Characterization of CRMs using SC-INAA at KRISS-KAERI NAA Group in Korea

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## Instrumental Neutron Activation Analysis of Inka pottery from fifteen Inka archaeological sites in the Lurín Valley, Central Coast of Peru: Insights into production and exchange

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**Keywords:** Neutron Activation Analysis, archaeology, pottery, South America, Andes, Inka, Pachacamac, Lurín Valley

Instrumental Neutron Activation Analysis (INAA) can be used to study the pottery of prehistoric societies: major and trace elemental concentrations can be used to investigate choices made by potters during the production process and to track the exchange of finished vessels across the landscape. This is especially useful in the study of the Inka empire, where pottery used by the state was made in a restricted number of forms and decorated with a standard suite of designs, but was produced through multiple organizations. In the provinces, Inka pottery was made locally by skilled potter subjects who were serving their *mit'a* labour obligation to the Inka empire; it was made by groups called *mitmaq*, who were laborers (including potters) relocated from their home territories to serve state-needs in other provinces of the empire; additionally, Inka pottery moved between centres and provinces, sometimes at great distances. INAA is used to evaluate three forms of Inka pottery most commonly seen in the provinces from fifteen archaeological sites in the Lurín Valley of Peru's central coast. At the mouth of this valley, the site of Pachacamac was the political centre of the Ychsma polity and home to an oracular *wak'a* deity prior to Inka conquest of the region. The Inka transformed the site into a major state political and ritual centre, constructing many buildings and spaces where Inka pottery was used in state-sponsored ceremonies. In addition to this major regional Inka center, Inka structures (and pottery within them) appeared at sites up-valley from Pachacamac, towards the next major Inka center at Hatun Xauxa in the highlands. Pottery from Pachacamac, and from additional sites in the Lurín valley, are analysed to investigate networks of production, distribution, and exchange for Inka pottery, and results from INAA are compared to other methods of investigation into archaeological pottery, including LA-ICP-MS and thin section petrography.

### Figures



Sites included in this study.

## Developments in the application of medical cyclotrons for neutron activation analysis

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**Keywords:** cyclotron-based NAA, radionuclide production

One ‘modern trend in activation analysis’ (MTAA) during the past ~30 years, which has been detrimental to the discipline, is the global decline in operating research reactors either through their decommissioning or permanent shutdown. This decline has resulted in the loss of neutron activation analysis (NAA) and reactor-based radionuclide production capabilities, together with the loss of research, teaching, training and outreach opportunities.

Conversely, in the last 10-15 years, there has been a substantial increase in the commissioning of cyclotrons worldwide, particularly medical cyclotrons in the 10-25 MeV range<sup>1</sup>. Exemplifying these trends, the University of Alberta (UofA) commissioned a TR-24 cyclotron in 2013 and, following 40 years of operation, decommissioned its SLOWPOKE research reactor in 2017.

The UofA TR-24, like other medical cyclotrons, is primarily designed for the production of medical radionuclides through ( $p, xn$ ) nuclear reactions. These radionuclides are crucial for diagnosing and treating diseases, especially cancers, as well as for use in the development of new and effective radio-pharmaceuticals.

However, during operation cyclotrons generate neutrons as a by-product which can serve as a neutron source for NAA and radionuclide production. The use of cyclotrons as neutron sources for NAA is not new<sup>2,3</sup>. However, keeping in mind that the primary function of a medical cyclotron is to produce radionuclides for diagnostic and therapeutic purposes alternate uses of the cyclotron should, ideally, not interfere with this function. Considering this, and with the decommissioning of the UofA SLOWPOKE reactor, this author has for the past 4 years been utilizing neutrons, generated during medical radionuclide production with the TR-24, for the NAA of a broad spectrum of sample types.

This presentation will summarize the utility and limitations of using a medical cyclotron, such as the TR-24, for NAA. Examples from various fields, including geology, archaeology, soil science, environmental science, and analytical chemistry, will be discussed. Industrial applications, such as ore assay and enhancing hydrocarbon production, will also be covered. Additionally, the use of the cyclotron as a neutron source for teaching and training purposes will be presented.

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## A Customized Rabbit for diagnosing the HANARO Pneumatic Transfer System for INAA

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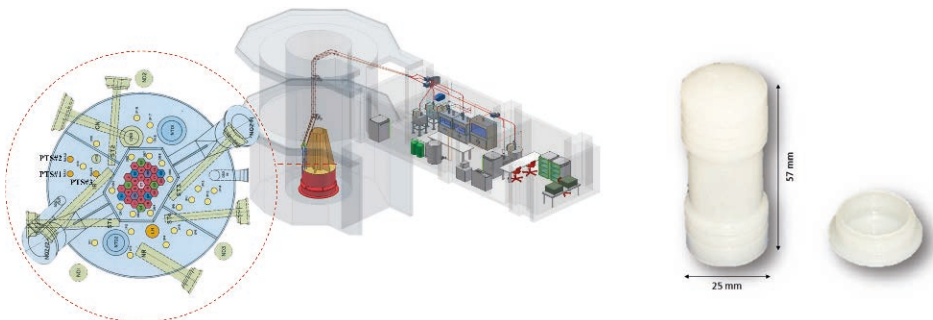
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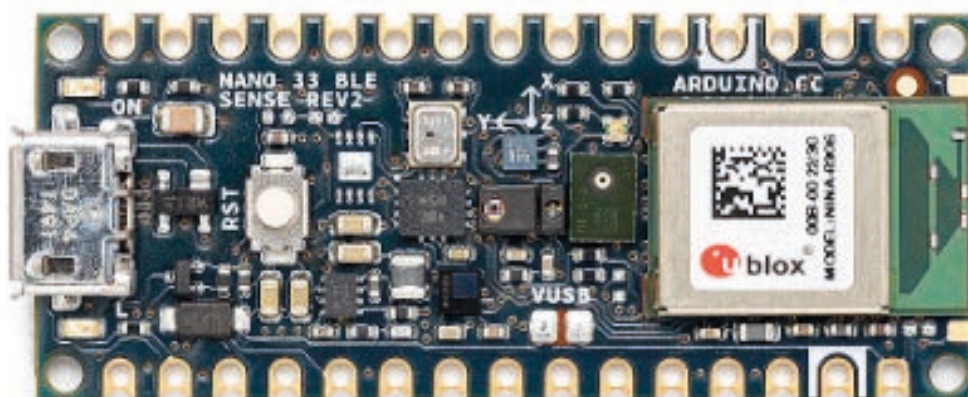
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**Keywords:** HANARO reactor, Instrumental Neutron Activation Analysis (INAA), Pneumatic Transfer System (PTS), PTS Diagnostics

In the Instrumental Neutron Activation Analysis (INAA), knowing exactly how long a sample has been irradiated to neutron flux is crucial for accurate analysis. This is because the more accurately the beginning and ending times of a sample's exposure to neutron flux can be determined, the more reliable the INAA will be. Especially for short term (within a minute or less) irradiation, this is essential. The research reactor "HANARO" operates a facility for INAA, which consists of a pneumatic transfer system (PTS) for neutron irradiation of samples (fig.1). The samples prepared for analysis are placed in "Rabbit" (fig.2) and sent to the irradiation hall via PTS and back to the analytical facility. The PTS is a closed pipe line, making it difficult to determine what's going on inside the pipe and how the rabbit is moving through it. The four photosensors (PS) installed at each point to check the movement of the rabbit in the PTS route and the acoustic sensor (AS) installed to check the arrival of the rabbit at irradiation point through the sound of the falling are used to determine the movement of the rabbit, but it is difficult to accurately determine the behaviour of rabbits in the PTS pipe line. Here, we aimed to develop a customized rabbit to improve the precision of the start time of neutron irradiation of a sample by using an infrared camera to diagnosing the inside of the PTS pipe line through video recording and Arduino Nano 33 BLE Sense ReV (gyro sensor) to record the actual behavior of the rabbit. The customized rabbits, which is designed to observe the internal environment of the PTS and judge the behaviour of the rabbit, will be used as a reference to improve the maintenance of the PTS facility and the accuracy or the neutron irradiation time.



**Figure 1.** Pneumatic transfer system and irradiation holes (left) and the Rabbit (right)



**Figure 2** Arduino Nano 33 BLE Sense Rev2

### **Acknowledgments**

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## Multi-element profiling by NAA of Amazon wood species for forensic timber identification

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**Keywords:** timber trade, illegal logging, timber tracking

The Amazon Rainforest occupies a territorial extension of 8.4 million km<sup>2</sup> and is recognized as the largest tropical forest in the world. About 64% of its area is located in Brazil and, despite its immense ecological and biological importance, the rapid advance of human activities, such as agriculture, mining and logging, has caused the unprecedented degradation of forested areas. In 2017, the Federal Police launched the Archimedes Operation to seize illegal timber, listing the main destinations as the United States, the Netherlands, Belgium, Portugal and France. The most illegally exploited and traded tree is endangered species *Handroanthus* spp. (Ipê), with 96% of exports coming from Brazil. Even with efforts to control deforestation, in 2022, 58% of the total deforested area in Brazil was located in the Amazon region. Through fraudulent actions, part of this wood is marketed as if it originated from legal areas. To combat this problem, chemical and isotopic patterns of wood are proving useful in identifying intrinsic characteristics that provide support for forensic analysis. Multi-element analysis has the potential for tracing geographical origin of timber. Here, neutron activation analysis (NAA) was used to obtain chemical profile of samples of *Handroanthus* spp (Ipê) collected in the Floresta Nacional do Tapajós (3° 31' 1" S, 55° 4' 23" W), in Santarém-PA. Three wooden discs, approximately 5 cm thick and 40 cm in diameter, were removed from the trunk at chest height (1.30 m). The discs were polished using sandpaper to reduce irregularities and remove surface contamination, and 60 sampling points measuring 5x0.8 cm were collected with a diamond core drill from the wood tissues. Analytical portions of 250 mg were irradiated in the IEA-R1 nuclear research reactor of the Nuclear Energy Research Institute, of the Brazilian Nuclear Energy Commission (IPEN/CNEN), São Paulo, SP, Brazil, and the induced radioactivity was measured by high-resolution gamma-ray spectrometry. The mass fractions of fifteen chemical elements - Ba, Br, Ca, Ce, Co, Eu, Fe, K, La, Na, Rb, Sc, Sm, Sr and Zn were quantified. The Mann-Whitney U test showed that there were no significant differences ( $p > 0.05$ ) in the mass fractions of the elements either in heartwood or sapwood tissues. This homogeneous distribution makes it easier to take representative samples for analysis. Nevertheless, statistically significant differences ( $p < 0.05$ ) were observed for the chemical elements between heartwood and sapwood. Br, Co, Fe, K, La, Rb and Sc showed mass fractions up to twelve orders of magnitude higher in the sapwood. These results featured important aspects of the inorganic chemical composition of wood. The heartwood, as the central and oldest part, being physiologically inactive gradually accumulates chemical elements over time, while the sapwood represents the actively growing xylem, characterized

by intense metabolic and nutrient-carrying activities. These findings highlight the complexity of chemical composition of wood and the need for insightful sampling planning, aligned with the specific approaches for geographic origin identification.

**Acknowledgments**

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## Reuse of irradiated reference materials contributes to lower running costs in quality control and calibration in NAA

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**Keywords:** reference materials, re-use, cost reduction, NAA, quality control

(Certified) reference materials are used in NAA for routine quality control and/or for calibration. The consumption rate may be high if recommended minimum test portion masses of hundreds of milligrams are required. Prices of reference materials are still increasing to several hundreds of euros/dollars for amounts in the order of 50 grams, especially for those with metrological traceable property values. This price tag can therefore be a burden for NAA laboratories with limited resources. Use of much smaller amounts than the recommended test portion mass is known being practised for these economic reasons. Such a practice affects, especially if used for calibration, unnotedly the degree of trueness of the results.

Reuse of irradiated reference materials after sufficient decay provides an opportunity to reduce the consumption rate of reference materials and thereby the running costs, without sacrificing the required minimum recommended test portion mass. A background measurement of the remaining activity of long half-life radionuclides prior to a new irradiation is subtracted from the new measurement results. Obviously, this is not even necessary for if elements measured on basis of radionuclides with half-lives up to 1-2 days which decay fully within, e.g., one month. Uncertainties of measurement of the long half-life radionuclides increase inevitably but are accounted for in the evaluation of the results. Reuse of reference materials also facilitates identification of unanticipated sources of error if successive results are not in agreement within the uncertainty ranges since effects from sample preparation can be excluded.

The radiation resistance of the encapsulation material and the associated maximum acceptable integrated radiation dose are limiting factors for the number of repetitions of re-irradiating the same test portion.

Examples on such a reuse of reference materials in NAA and the impact on the analytical quality will be presented.

## Innovative neutron activation approach for analysis of large water samples based on short-lived radionuclides

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**Keywords:** flowing sample activation analysis, liquid samples, short-lived radionuclides

Flowing sample neutron activation analysis (FSNAA) has been developed for analysis of liquid samples [1]. It involves continuous pumping of a large volume of the sample, in a tube, between an irradiation site and an HPGe [1]. Analysis of large volume improves the detection limits, while continues pumping allows better measurement of short-lived radionuclides. The set-up was previously tested with <sup>252</sup>Cf as a neutron source [1, 2]. The purpose of the present work is to install FSNAA at a research reactor.

Irradiation durability test was performing to select a suitable tubing material based on a simple bending test [3]. The general procedures involve pumping the liquid sample to the irradiation site, and then record a  $\gamma$ -ray spectrum with HPGe. A run with de-ionized water was carried out for leakage test and to check the performance of the components as well as for background measurements. FSNAA was tested for analysis of tap and river waters samples. However, irradiation durability test showed that all tested tubes have acceptable radiation resistance, TYGON was used for constructing the system due to its excellent radiation durability.

Nine elements were detected and quantified (Al, Mn, Mg, V, Na, K, Cu and Ca), while Cl, Br, I, and <sup>18</sup>O were detected but not quantified due to the lack of reference standard. The detection limits obtained from this preliminary study are satisfied in comparison to conventional NAA and other techniques. Levels of the quantified elements in the tap water are below the WHO guidelines [4]. Under the current experimental conditions, the decay time (the travelling time between the irradiation site and HPGe) was ~ 3.5 min. This relatively long decay time hinders the analysis of several elements (those with shorter half-lives isotopes ex: <sup>20</sup>F, <sup>46m</sup>Sc, <sup>77m</sup>Se, <sup>107m</sup>Pb,...). Also, it adversely affects the detection limits of <sup>28</sup>Al, <sup>52</sup>V, and <sup>66</sup>Cu due to the decay of major fraction of their radioactivities before reaching the HPGe. As a future plan, use of powerful pump will be considered to reduce the decay times; and hence it is expected to increase the number of measured elements and improves the detection limits for some others. The capability of FSNAA to measuring <sup>18</sup>O reveals its potential in paleoclimatology studies.

**Table** Analysis results of tap and river water samples (ND: not detected, D: Detected only)

Element	Tap water		River water	
	$\mu\text{g/l} \pm \%$	DL	$\mu\text{g/l}$	DL
O ( $^{19}\text{O}$ )	D		D	
Na	$9700 \pm 2$	110	$8800 \pm 2$	105
Mg	$1300 \pm 5$	135	$3720 \pm 3$	200
Al	$25.5 \pm 6$	2.2	$240 \pm 1.3$	2.0
S	$8000 \pm 25$	7400	$10500 \pm 20$	6600
Cl	D		D	
K	$1350 \pm 23$	980	$1350 \pm 25$	1180
Ca	$10100 \pm 4$	280	$15900 \pm 2.5$	240
V	$0.40 \pm 14$	0.14	$0.37 \pm 15$	0.15
Mn	$1.7 \pm 30$	1.5	$132 \pm 2$	1.9
Cu	$28.0 \pm 35$	25	ND	
Br	D		D	
I	D		D	

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## Detection limits in $k_0$ -NAA

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**Keywords:** Kayzero, element analysis, gamma spectrometry

The  $k_0$ -method for neutron activation analysis is well established in open literature. All aspects of the conversion of a peak area, measured in a gamma spectrum, to an element concentration are well defined and experimentally verified. In recent literature several software programs were evaluated and compared based on the calculation of concentrations. Detection limits for were not determined and not considered. This paper is addressing the aspects of detection limit determination based on experimental data.

The concepts used in Kayzero for Windows (KfW) are explained. For KfW the spectrum evaluation (peak area determination) is based on HyperLab. Detection limits in NAA are based on the minimal detectable peak area in the measured gamma spectrum, for this information on the peak background, or the spectrum baseline is needed. Hyperlab however does not give peak backgrounds, so this is done by KfW. Especially if a spectrum does not have any peaks, the baseline needs to be determined.

All aspects concerning determination of detection limits are described for spectra with low- and high count rates as well as the use of a loss-free counting system like the LFC-module. Detection limits are important for validation of the presence of nuclides and elements. Special cases where nuclide interferences play a role are also discussed.



## Research Progress of Neutron Activation Analysis at CARR

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**Keywords:** INAA, Chang'E-5 samples, lunar meteorites, CARR

Nuclear activation analysis (NAA) is a widely applied analytical technique which allows qualitative and quantitative elemental analysis of various types of samples by means of nuclear radiation measurements. There are four types of NAA technologies are developing at the 60 MW China Advanced Research Reactor (CARR) of China Institute of Atomic Energy (CIAE) with the thermal neutron fluence rate of  $8 \times 10^{14} \text{ cm}^{-2} \text{ s}^{-1}$ . The main goal is to establish a scientific CARR-based NAA science centre including instrumental NAA (INAA), prompt gamma-rays activation analysis (PGAA), neutron depth profiling (NDP), and delay neutron counting (DNC). The centre will be equipped with advanced devices, analysis techniques, optimized parameters and expert system. As a consequence, the NAA are potential multi-functional tools for science research in China. Therefore, the subject of this study represents an important step toward the development of a multi-radiation NAA platform for several applications in materials, geochemistry, environment, biology, archaeology and nuclear science, etc. We look forwards to the adoption of NAA in various fields of research in China based on the advanced platform. Besides, some recent research works (i.e., extraterrestrial materials like China's Chang'E-5 lunar samples, lunar meteorites and air particles analysed by INAA, B and H by PGAA, and NDP experiments) will be introduced. Furthermore, we are looking forward to use these NAA techniques developed at CARR for more deep-space exploration samples such as lunar far-side samples, asteroid and Mars samples, etc.

### Acknowledgments

Thanks to the China National Space Administration (CNSA) for providing the Chang'E-5 lunar samples. We would like to thank all the staff of China's Chang'E-5 project for their hard work returning lunar samples. We also thank CIAE for its strong supporting on the NAA work.

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## Instrumentation for Activation Analysis

### Development of $4\pi\beta\text{-}\gamma$ coincidence counting system for absolute radioactivity measurement

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**Keywords:** radioactivity measurement,  $4\pi\beta\text{-}\gamma$  coincidence counting, SiPM

Radionuclides are used in various applications, such as research, medical, or industrial fields. Some of such radionuclides are produced in High-flux Advanced Neutron Application Reactor (HANARO) at the Korea Atomic Energy Research Institute (KAERI), and provided to various institutes with their information such as production date and radioactivity. For measuring radioactivity of produced radionuclides, a  $4\pi\beta\text{-}\gamma$  coincidence counting system (Fig. 1) was developed in HANARO utilization division at KAERI.

The key components of the system are liquid scintillator, NaI crystal scintillator and SiPMs. These two types of scintillators detect  $\beta$  and  $\gamma$ , respectively. The scintillation lights emitted from the scintillators are converted to electric signals, by SiPMs.

The developed system was tested using certified materials  $^{134}\text{Cs}$  and  $^{177}\text{Lu}$ , and the characteristics and performances of the system were investigated. After the tests were completed, the absolute radioactivity of the radionuclide  $^{177}\text{Lu}$  produced by HANARO was measured using the system. And the uncertainties for absolute radioactivity measurement were listed and the total error was calculated.

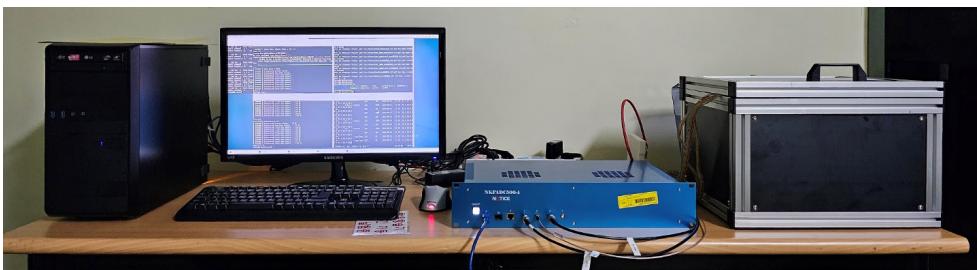


Figure 1.  $4\pi\beta\text{-}\gamma$  coincidence counting system

#### Acknowledgments

This work was supported by the National Research Foundation of Korea (NRF) Grant funded by the Korea government (MSIT) (NRF-2021M2E7A2079439).

## $k_0$ -based activation analyses

### Significance of Westcott g-factor for the assay of non- $1/\nu$ nuclides using $k_0$ -NAA

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**Keywords:** neutron temperature ( $T_n$ ), Westcott g ( $T_n$ ) factor, Høgdahl and Westcott convention

Present study examines the impact of Westcott g-factor on the accuracy of the nuclides, whose thermal neutron capture cross-sections deviate from the conventional  $1/\nu$  behaviour and quantified using  $k_0$ -NAA. The elemental standards of various non- $1/\nu$  nuclides, namely Iridium ( $^{193}\text{Ir}$ ), Indium ( $^{115}\text{In}$ ), Rhenium ( $^{185,187}\text{Re}$ ), Ytterbium ( $^{168}\text{Yb}$ ), Lutetium ( $^{176}\text{Lu}$ ), and Europium ( $^{151}\text{Eu}$ ), were prepared from standard solutions on a weight basis. The prepared standards, along with Gold (Au), underwent irradiation for varying time intervals at the PFTS location of the KAMINI reactor. The quantitative analysis in  $k_0$ -NAA utilized recently characterized neutron spectrum parameters such as neutron temperature, thermal to epi-thermal neutron flux ratio, modified spectral index, and epithermal flux shape factor. The crucial Westcott g-factor for these non- $1/\nu$  nuclides was computed based on the neutron temperature ( $T_n$ ) at PFTS. This computation involved establishing a correlation between  $g(T_n)$  and the neutron temperature, spanning a range from 0 to 100°C, derived from the latest ENDF/B-VIII.0 nuclear data library. The BROADR module from NJOY21 was employed to process neutron capture cross-section data for the aforementioned nuclides at multiple temperatures. The impact of  $g(T_n)$  on accuracy was observed to be minimal, i.e., less than 5%, for In, Ir, and Re. However, a significant enhancement of 80% was noted for Lu, 11% for Eu, and 5% for Yb. These findings contribute valuable insights into the nuanced effects of the Westcott g-factor on the accuracy of  $k_0$ -NAA measurements for specific non- $1/\nu$  nuclides under varying neutron temperature conditions.

Figures

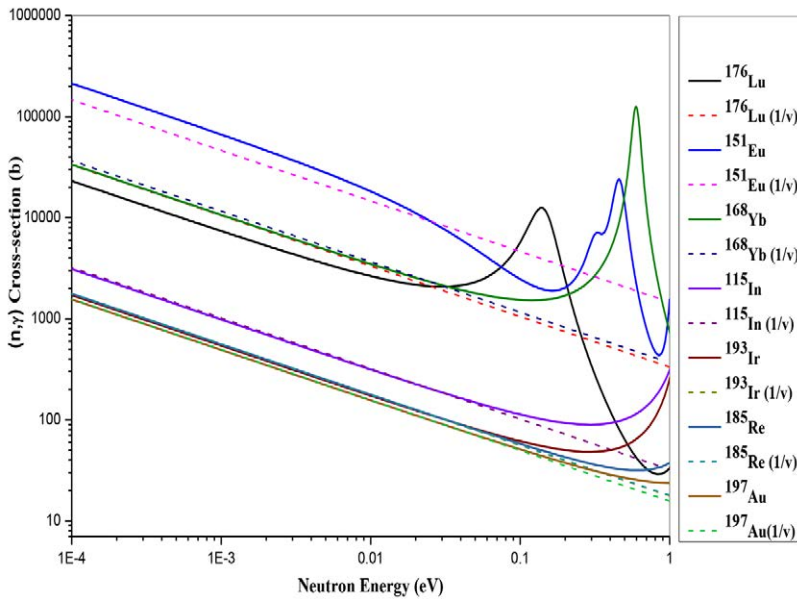


Fig. 1 Variation of  $\sigma_{(n,\gamma)}$  for non-1/v with neutron energy

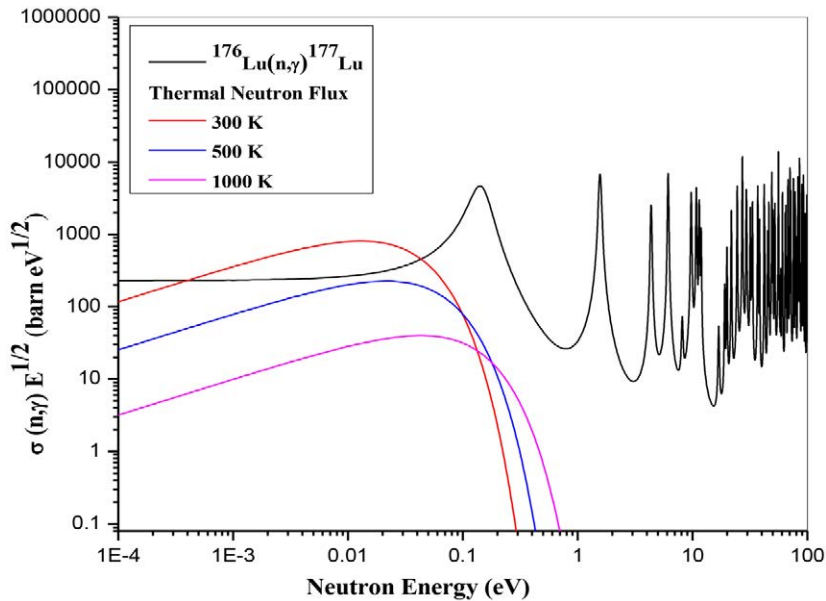


Fig. 2 Maxwell-Boltzmann flux distribution for different temperature

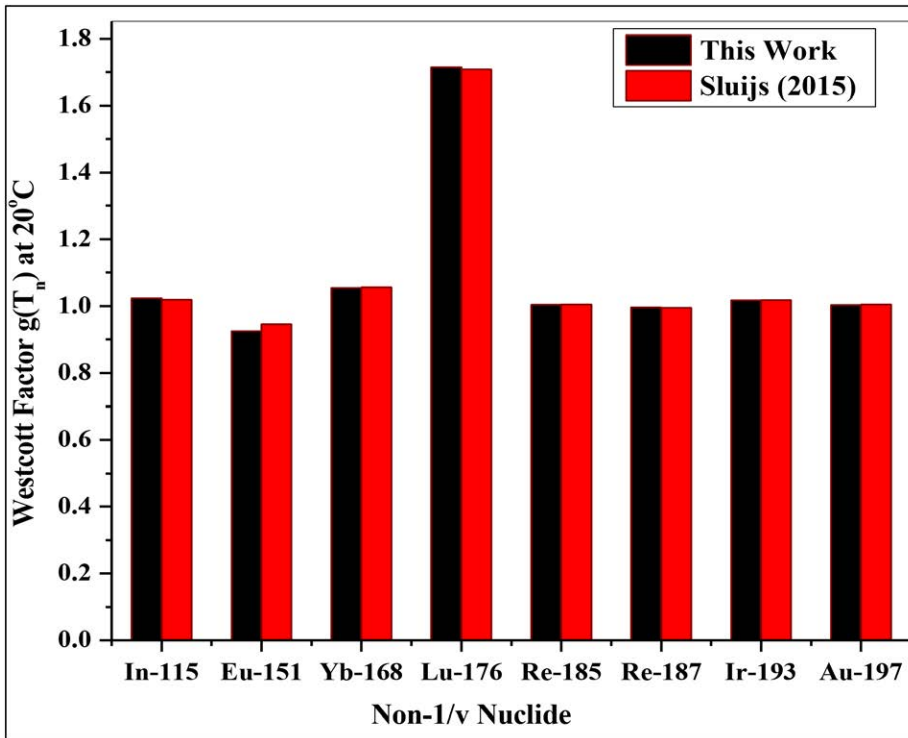


Fig. 3  $g(T_n)$  from the present work & Sluijs at 20 °C

Table 1. Neutron Spectrum parameter of PFTS, KAMINI Reactor

Parameters	Thermal Flux	$\alpha$	$f$	$r_0\sqrt{T_n/T_0}$	$T_n$ (°C)
Value	$1 \times 10^{11}$	$-0.0494 \pm 0.0071$	$25.47 \pm 0.71$	$0.037 \pm 0.001$	47.2

## Acknowledgments

We sincerely thank the reactor operational engineers of KAMINI reactor for their valuable support towards the irradiation experiments.

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## Standardization of $k_0$ -INAA method for high $f$ value at HANARO reactor, South Korea

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**Keywords:** HANARO reactor, flux monitors, Cd-ratio method, bare method, validation, zeta score

Presently, the HANARO reactor has been actively used for the certification of inorganic elements in candidate reference materials (RMs) developed by KRISS, South Korea, through the single (standard) comparator instrumental neutron activation analysis (SC-INAA) method. In addition to SC-INAA, we aim to standardize the  $k_0$ -based INAA ( $k_0$ -INAA) methodology at highly thermalized neutron flux positions of the HANARO reactor for the chemical characterization of certified RMs (CRMs). The neutron flux characteristics of the neutron shape parameter ( $\alpha$ ) and sub-cadmium to epithermal neutron flux ratio ( $f$ ) were determined at all three irradiation positions of the HANARO reactor (NAA#1, NAA#2, and NAA#3) using the bare triple (<sup>197</sup>Au, <sup>94</sup>Zr, and <sup>96</sup>Zr) monitor method and confirmed using the Cadmium ratio method. The experimental values of  $f$  were about and above 1000 at NAA#1 and NAA#2, which are close to the reflector and far from the core position, and about 100 at NAA#3 (close to the core position), respectively. Positions NAA#1 and NAA#3 were characterized for  $f$  and  $\alpha$  20 years ago, right after the initiation of the  $k_0$ -INAA work at the HANARO reactor using the Cd ratio method. The thermal flux at maximum reactor operation power at 30 MW at is  $5 \times 10^{14}$  n.cm<sup>-2</sup>.s<sup>-1</sup>. In the present work, we newly investigated of  $f$  and  $\alpha$  values at NAA#2 along with other positions for the establishment of the  $k_0$ -INAA methodology. The efficiency of gamma-ray spectrometry was well calibrated using coincidence-free standard radionuclides sources (<sup>241</sup>Am, <sup>109</sup>Cd, <sup>57</sup>Co, <sup>57</sup>Co, <sup>51</sup>Cr, <sup>133</sup>Sn, <sup>137</sup>Cs, <sup>54</sup>Mn, and <sup>65</sup>Zn). HyperGam and HyperLab software were employed for complex spectra analysis, and the Kayzero software for  $k_0$ -INAA methodology was established for elemental concentration determination. The  $k_0$ -methodology was standardized and validated by analyzing KRISS CRMs and NIST SRMs (environment and geological matrix samples). The results are in good agreement with certified or recommended values. The  $k_0$ -based NAA methodology was applied for the chemical characterization of candidate RMs developed by KRISS, including new matrix materials.

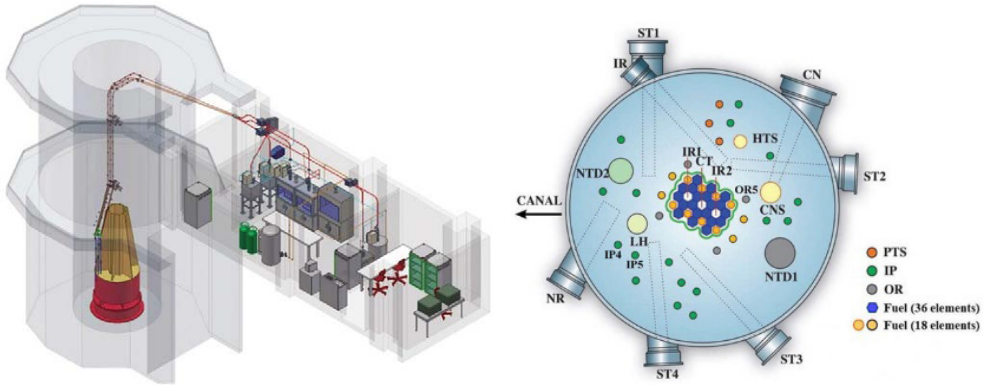


Fig. 1. (Left) PTS configuration at HANARO reactor, (Right) HANARO core and PTS irradiation holes.

### Acknowledgements

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## Study on a dual method using SMELS for neutron flux self-monitoring and quality control in the k<sub>0</sub>-NAA

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**Keywords:** k<sub>0</sub>-NAA, reactor neutron flux, certified reference material, SMELS

A dual method for neutron flux self-monitoring and quality control in the k<sub>0</sub>-based neutron activation analysis (k<sub>0</sub>-NAA), is described in this study. The synthetic multi-element standards (SMELS) consists of three different types respectively short lived (SMELS Type I), medium (Type II) and long-lived (Type III) radionuclides, used for the validation of k<sub>0</sub>-NAA. All three SMELS types containing certified amounts of Au can be used as neutron flux monitors, to which the Au content can be automatically entered into the k<sub>0</sub>-Dalat software to calculate the gold specific activity [Asp(Au)] needed to derive the mass fraction of the elements of interest by k<sub>0</sub>-NAA in the sample. Moreover, the certified concentration of elements in SMELS are also used for the k<sub>0</sub>-NAA quality control purposes. Replicate analyses of a NIST-SRM-2711a (Montana II Soil) shows that the determination of 21 elements by k<sub>0</sub>-NAA of duplicate samples gives uncertainties with coefficients of variation from 0.8% to 6.3%, of which 15 elements have a precision of better than 4.5%. This demonstrates that the use of SMELS as a dual method for both neutron flux self-monitoring as well as k<sub>0</sub>-NAA quality control is considered convenient and consistent with the results obtained by the conventional Au-independent monitoring method.



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## Neutron Depth Profiling

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### Investigation of $C_{60}$ / $LiCoO_2$ cathode for All Solid-State Lithium Ion Batteries using Thermal Neutron Depth Profiling

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**Keywords:** Neutron Depth Profiling, Lithium Ion Batteries, Ferroics based cathodes

Thermal Neutron Depth Profiling (NDP) technique was employed to investigate the lithiation and delithiation processes in thin film materials for All-Solid-State Lithium-Ion Batteries. The studied films were combinations of buckyball  $C_{60}$  and  $LiCoO_2$  as a cathode. The operation voltage range was higher than usual (up to 4.2 V), to study the possible application of the film for prototypes. The films were produced by different PVD methods, from co-deposition of  $C_{60}$ , Co, and Li by Electron Beam Evaporator, to Ion Beam Sputtering. In-situ and in-operando NDP measurements, in combination with other off-line morphological investigations, such as Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM), will be presented together with the results obtained for the over-voltage range investigation.

#### Acknowledgments

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## KAERI-NDP System: Development and performance analysis

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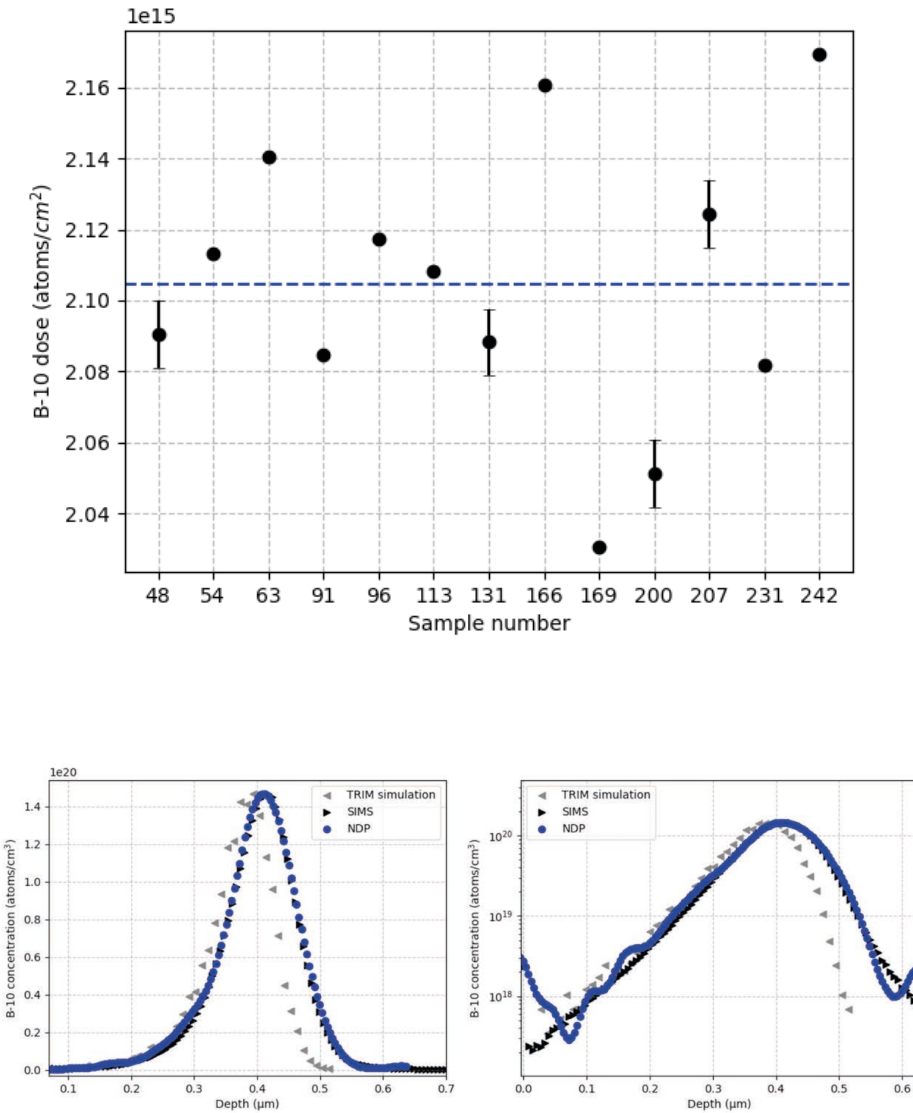
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**Keywords:** NDP, HANARO, cold neutron, depth profiling, SiC wafer

Neutron depth profiling (NDP) is a non-destructive analytical technique that enables the absolute quantification of the depth distribution of specific light elements, such as Li-6 and B-10, within materials. In NDP analysis, elements within the sample that have high neutron capture cross-sections emit charged particles upon reacting with irradiated thermal neutrons. As they traverse the material, these charged particles lose energy. The depth distribution is estimated by analysing the relationship between the residual energy of the detected charged particles and their depth. Recently, the Korea Atomic Energy Research Institute (KAERI) has developed an advanced NDP system, the KAERI-NDP, which takes advantage of cold neutrons (~4 meV) with a flux of  $1.70 \times 10^8 \text{ cm}^{-2}\text{s}^{-1}$  generated from the HANARO research reactor. The previous KAERI-NDP system exhibited a high background level due to neutrons escaping through the gap between the guide tube and the shielding body. However, shielding improvements have significantly reduced the background level. To evaluate our system, a 4-inch HPSI-4H SiC wafer was ion-implanted with a concentration of  $2.00 \times 10^{15} \text{ atoms/cm}^2$  and diced into  $5 \times 5 \text{ mm}^2$  pieces to create a total of 278 reference materials. Out of these, 13 samples were selected for quantification analysis using the relative method with the NIST SRM-2137 standard sample, determining the dose at  $2.10 \times 10^{15} \pm 5.25 \times 10^{13}$ . The evaluated dose was confirmed to be within 5% of the ion-implanted data, demonstrating a high degree of accuracy. The uncertainty in the NDP analysis of the SiC reference material was found to be 2.50%, which includes the sample homogeneity, standard sample uncertainty, repeatability, and Poisson uncertainty. Additionally, the depth distribution estimated by NDP was also in good agreement with SIMS data, validating the performance of the KAERI-NDP system.

Figures



Acknowledgments

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## 3D Neutron Imaging for Depth Profiling Applications

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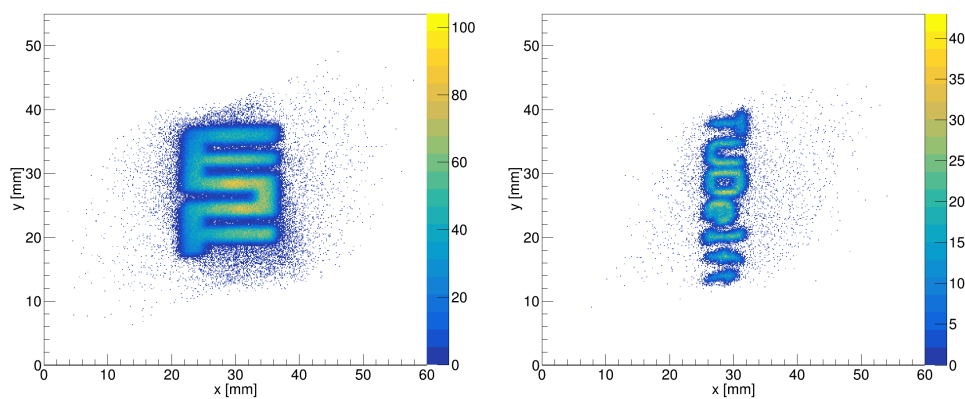
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**Keywords:** Neutron Depth Profiling, Double-Sided Silicon Strip Detector, N4DP

Neutron Depth Profiling (NDP) is a non-destructive, element-specific nuclear analytical technique, which is commonly used to analyze the concentration profiles of elements like lithium, boron, nitrogen, helium, and other light elements in various host materials. The N4DP instrument is located at the Prompt Gamma Activation Analysis (PGAA) beamline of the Heinz Maier-Leibnitz Zentrum (MLZ), which provides a cold neutron flux up to  $5 \times 10^{10} \text{ s}^{-1} \text{ cm}^{-2}$ . The NDP technique makes use of the capture reaction of a certain nuclide with a subsequent decay into ions with well-defined energies. The depth from which these ions originate within the material can be determined from their energy loss, with a precision of tens of nanometers.

There is a significant demand for monitoring light elements in operating systems. For instance, when operating thin film batteries, there is a lack of detectors with high detection efficiency and time stamping to track the movement of the Li cloud during charging and discharging. Additionally, inhomogeneities in such electrochemical processes require good spatial resolution to obtain concentration information at various locations within the battery. We developed a detector system for the N4DP instrument, employing double-sided silicon strip detectors (DSSSD) with ultra-thin, uniform entrance windows, providing a new state-of-the-art type of NDP measurements. A highly segmented DSSSD with 32 x 266 stripes, including integrated, self-triggering electronics, was successfully tested and evaluated at the research reactor in Delft (RID), Netherlands. Using a dual-detector setup based on the camera-obscura principle, we achieved the image and reconstruction of Li-containing targets with a spatial resolution down to  $\sim 100 \mu\text{m} \times 200 \mu\text{m}$ .



TUM and TUDelft Logos reconstructed from two detectors setup

### Acknowledgments

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## Progress on Neutron Depth Profiling at China Advanced Research Reactor

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**Keywords:** NDP, lithium battery electrodes, nanoscale films, semiconductors

The China Advanced Research Reactor (CARR) cold neutron beam features a neutron depth profiling system with a vacuum level reaching  $3.4 \times 10^{-5}$  Pa. Operating at its maximum power of 60MW, the CARR reactor produces a cold neutron flux of approximately  $10^9 \text{ cm}^{-2} \cdot \text{s}^{-1}$ . Under a 15 MW power output, neutron fluence at the end of the neutron guide tube was measured to be around  $4.8 \times 10^8 \text{ cm}^{-2} \cdot \text{s}^{-1}$  using gold activation method<sup>[1]</sup>.

In 2023 the CARR has been operational for over 100 days, conducting various experiments utilizing the NDP apparatus. These experiments encompassed several objectives: firstly, determining the lithium distribution within different lithium-ion battery by employing nuclear reactions of neutrons with  $^6\text{Li}$ . Secondly, measuring the thickness of nanoscale films based on the energy loss of particles generated from neutron reactions with  $^6\text{Li}$  and  $^{10}\text{B}$ ; utilizing neutron bombardment on LiF material, the experiments measured various thicknesses of nanoscale Cu films, achieving a highest resolution of up to 1.3 channels/nm with a measurement deviation of less than 1.5%<sup>[2]</sup>. Lastly, employing neutron reactions with  $^{10}\text{B}$  to generate  $\alpha$  particles, the experiments measured the concentration and depth distribution of B in semiconductor materials(Fig.1).



Fig.1. Lithium battery electrode samples in NDP chambers

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## Neutron sources and their characterization

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### The research reactor TRIGA Mainz: a highly flexible neutron source for activation experiments

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**Keywords:** research reactor, neutron source, pulse mode operation, neutron activation

Research reactors of the TRIGA-type [1] are light water-cooled reactors using fuel-moderator elements composed of an alloy of uranium-zirconium-hydride (UZrH) with 20% enrichment in  $^{235}\text{U}$ . In the steady state mode, the TRIGA Mainz [2] can be operated at power levels ranging from about  $100 \text{ mW}_{\text{th}}$  up to  $100 \text{ kW}_{\text{th}}$ , depending on the requirements of the different experiments. Pulse-mode operation is also possible, corresponding to a maximum pulse peak power of up to  $250 \text{ MW}_{\text{th}}$ , a neutron flux in the order of  $10^{15} \text{ cm}^{-2}$  per pulse and a pulse width (FWHM) of about 30 ms. Here, the large prompt negative temperature coefficient of the TRIGA-reactor, an inherent characteristics of the fuel-moderator elements, reduces the power of the reactor within a few thousandths of a second, faster than any engineered device can operate. Pulse mode operation is especially advantageous to produce very short-lived nuclides with half-lives below one minute. In these cases, the activity obtained in pulse-mode is higher than the corresponding saturation activity in steady-state operation. For long-term irradiations the TRIGA Mainz is equipped with a central experimental tube (central thimble) and a rotary specimen rack with 40 positions which allows the irradiation of up to 80 samples at the same time. Here, the samples are transferred manually. Furthermore, for the production of nuclides with half-lives up to a few minutes, two pneumatic transfer systems are available. From terminals located in the reactor hall or in a radiochemical laboratory the samples are transferred pneumatically to the irradiation position and back. With these systems, transport times of 1–5 s can be achieved. A thermal column is a further irradiation unit that provides well-thermalized neutrons suitable for physical research or biological/medical irradiations. It consists of a boron-lined aluminum container filled with blocks of graphite. For irradiations in the thermal column up to five graphite blocks (102 mm x 102 mm x 1270 mm) can be removed to introduce the sample. In addition, the TRIGA Mainz includes four horizontal beam ports penetrating the concrete shielding and extending inside the pool towards the reactor core. Figure 1 shows a cross section view and corresponding neutron flux values for  $100 \text{ kW}_{\text{th}}$ . About 80% of the beam time is used for reactor operation at  $100 \text{ kW}_{\text{th}}$  and the rest for pulses and steady state operation below  $100 \text{ kW}_{\text{th}}$ .

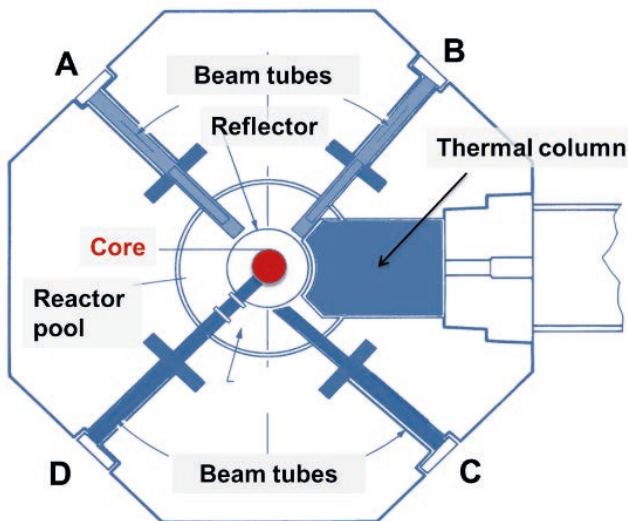
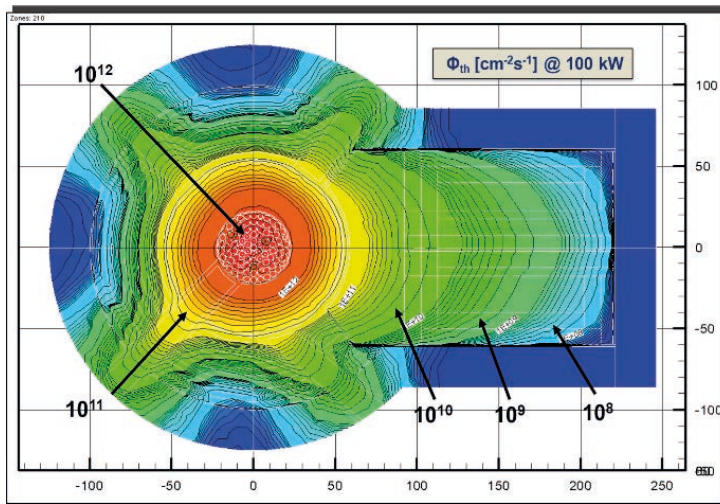


Figure 1: Cross section view of the TRIGA Mainz and corresponding neutron fluxes for a thermal power of 100 kW

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## Experimental and numerical evaluation of the performance of an integrated activation device for routine neutron spectrum confirmation at THOR-BNCT

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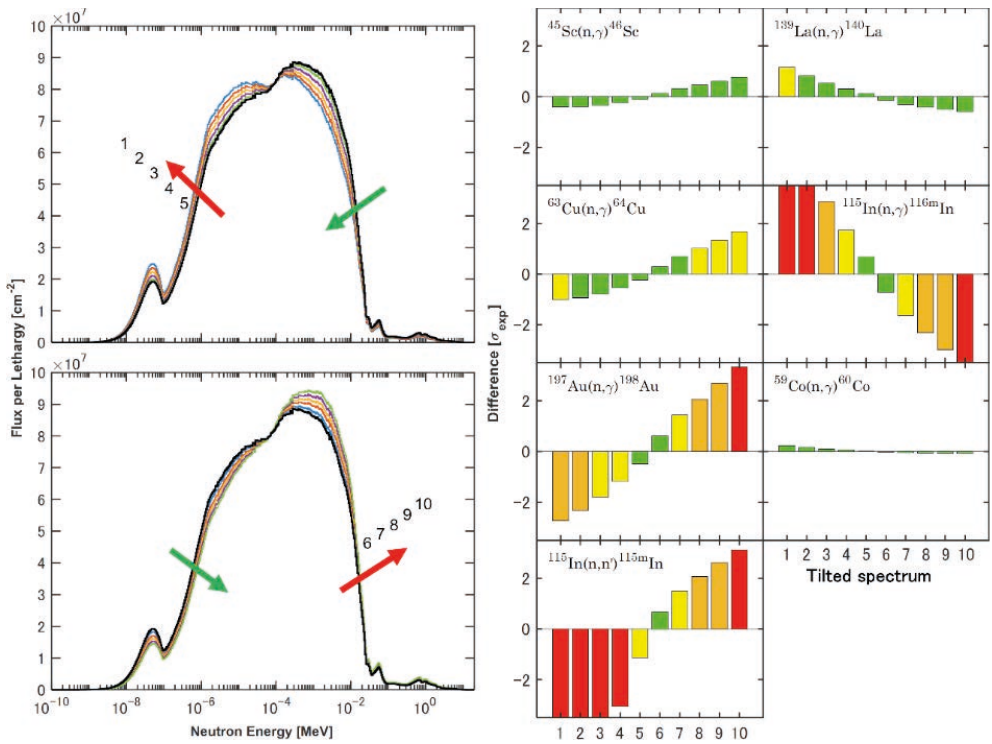
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**Keywords:** boron neutron capture therapy, neutron spectrum, beam quality assurance, Monte Carlo, activation foils

Experimentally ensuring the quality of neutron beam before patient treatment is a topic of central importance for a boron neutron capture therapy (BNCT) facility. The procedure of beam quality assurance currently adopted at THOR-BNCT involves two kinds of measurements: (1) paired ion chambers to discriminate neutrons and gamma rays, and (2) Cu and Au activation foils in a phantom to detect epithermal neutrons. The procedure works well but lacks a direct indication related to the energy distribution of neutrons. A new device based on multiple activation foils was proposed at THOR-BNCT, aiming to provide a quick confirmation of the unchanged neutron spectrum before patient irradiation. The device's response to the THOR-BNCT neutron beam is presented. A series of experimental tests were repeated to establish the baseline performance of the device, and further numerical simulations were conducted to determine its sensitivities to various perturbations in the neutron spectrum. To increase the rigorousness of beam quality assurance, the device has been suggested to replace the role of Cu/Au activation foils in the current procedure at THOR-BNCT by supplementing additional information in the neutron spectrum.

Figures



Ten artificial tilted spectra used in numerical evaluation and the caused reaction rate differences in the unit of experiment uncertainty.

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## Prompt Gamma Activation Analysis

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### Development of on-line analysis device for selection and smelting process based on PGNAA technology

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**Keywords:** PGNAA, Monte Carlo simulation, pulsed neutron, uranium measurement, spectra analytical method

Prompt Gamma-ray Neutron Activation Analysis (PGNAA) is a real-time, online and non-destructive elemental analysis method which is widely applied in environment, health sciences, and industry, etc. In this research, the element composition analysis of the mining and enrichment process of the mineral resource "uranium" is taken as the application object. Aiming at the online analysis requirement of the element composition in solution and resin, a device with new analysis method for multi-element online analysis is developed based on PGNAA technology, and it realizes the requirements of real-time online measurement of elements in solution and resin tower during different processes, including leaching, extraction and adsorption, etc.

(1) The response relation of the time spectrum ratios between the epithermal neutrons and thermal neutron were investigated under different conditions (uranium content, solution composition and environment). Finally, the quantitation method for uranium measurement was built based on the ratio method of epithermal neutron and thermal neutron.

(2) The non-linearity on the spectra was corrected, and the standard single-element response spectra analysis method (SSERS) was established. Finally, the standard single-element response spectra library was established based on the experimental tests.

(3) Based on the novel optimization method, the model of built-in PGNAA analytical instrument was designed and built for element measurement.

Figures

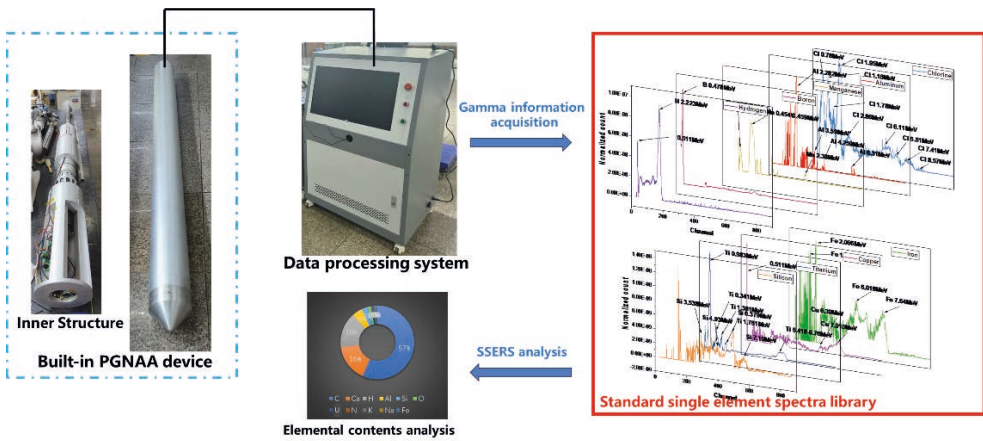


Fig.1 Element analysis process of Built-in device

Acknowledgments

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## Revision of the analytic database for PGAA

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**Keywords:** prompt gamma activation analysis, element analysis, database

In prompt gamma activation analysis (PGAA), the reliability of the element analysis highly depends on the quality of the spectroscopy database. No such database existed in the last century that met the requirements of chemical analysis, which restrained the development of the technique. The establishment of a spectroscopy database of prompt gamma lines was started in Budapest in 1997 after the PGAA facility was put into operation. The first version of the analytic database was published in the Handbook of Prompt Gamma Activation Analysis [1] and later a compiled version appeared in a Technical Document of the International Atomic Energy Agency [2]. The catalog contains the spectroscopic data (energies, partial gamma-ray production cross sections and their uncertainties) for the prompt gamma rays induced by cold neutron capture on every chemical element occurring in nature, except helium.

Three types of spectra have been measured for all naturally occurring elements:

- 1) spectra of elements (sometimes as oxides, or simple compounds),
- 2) energy calibration, i.e. irradiation together with chlorine (PVC) whose precisely-known prompt gamma lines were used to determine the energies from the element of interest,
- 3) standardization: measurement of stoichiometric compounds (e.g. chlorides) or homogeneous mixtures (e.g. water solutions) to determine the partial cross-sections of the lines relative to a comparator (Cl or H).

Most of the previous measurements have been repeated using high-purity compounds, metal foils etc. exploiting the unique conditions of the high-flux PGAA instrument in Garching. These data are supported by the remeasured ones in Budapest. The evaluation is being performed in parallel with the measurements.

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## Experimental database of gamma coincidence spectra recorded in Prompt Gamma Neutron Activation Analysis

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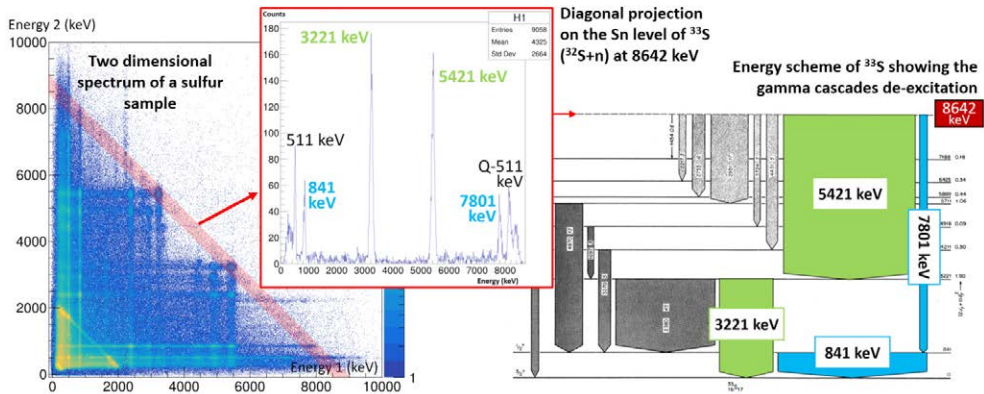
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**Keywords:** Prompt Gamma Neutron Activation Analysis, pulsed neutron generator, radiative capture gamma rays, gamma-gamma coincidences, 2D gamma-gamma spectroscopy

In the frame of CEA waste recycling R&D program, an innovative neutron interrogation elemental analyser has been developed for non-intrusive inspection of batteries, permanent magnets or e-waste from electronic and electric equipment (WEEE). Six large volume NaI(Tl) serve to determine the elements composing the inspected materials, based on their specific gamma spectra signature induced by thermal neutron capture. More precisely, gamma-gamma coincidences between the NaI(Tl) scintillators allow a multidimensional spectrometric analysis and the identification of gamma cascades (see Fig. 1) that are specific to the isotopes of the elements of interest [1]. This work reports the creation of an experimental database of gamma-gamma coincidence spectra for Prompt Gamma Neutron Activation Analysis (PGNAA), in the form of two-dimensional spectra representing the number of counts as a function of the two energies of the gamma rays recorded in coincidence, see Fig. 1 (left panel). Diagonal projections of such spectra can also be used to improve the signal-to-noise ratio (Fig. 1, zoom), as well as three-dimensional coincidence spectra, when applicable. Measurements are performed on a wide panel of pure elements (S, Cu, Si, Fe, Cl, Co, Ni, Cr, Mg, Ti, Mn, Dy) to retrieve their specific signature in multidimensional spectrometry. The two-dimensional coincidence spectrum associated with an object of unknown composition can be unfolded into the linear combination of the measured pure elements' spectra, allowing for the determination of the object's composition.



**Figure 1** : Gamma-gamma coincidence spectroscopy analysis – 2D spectrum of a sulfur sample – diagonal projection highlighting signal of interest – comparison and reconstruction of the energy scheme associated

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## Machine learning for Prompt Gamma Activation Analysis

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**Keywords:** PGAA, machine learning, neural networks

Prompt gamma activation analysis is a well-established method of chemical analysis with neutrons. Compared to decay spectra, prompt gamma-ray spectra contain a much higher density of gamma lines. This often results in laborious and time-consuming manual post-processing. Machine learning algorithms can help to speed up the evaluation process.

The aim of the new EvalSpek-ML project is to provide a new machine learning toolbox for the evaluation of spectra - especially for spectra that can be approximated as a convex combination of their constituent parts. The project is a collaboration between Helmholtz-Zentrum Hereon, Technical University of Munich, Helmut Schmidt University Hamburg and AiNT GmbH. We will present the concept and preliminary results of the project in the context of prompt gamma activation analysis.

### Acknowledgments

The authors thank the German Federal Ministry of Education and Research for funding and support. The funding is provided in the framework of the German action plan “ErUM-Data” (collaborative project 05D2022).



## Time-of-Flight Prompt Gamma Neutron Activation Analysis

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**Keywords:** PGNA, CANS, neutron

Prompt Gamma Neutron Activation Analysis (PGNAA) is a powerful technique to determine the elemental compositions of samples of various origins. It measures the emitted prompt gamma rays due to neutron capture which have an isotope specific energy. The comparison of the emission rate of different gamma ray lines allows thus to determine the concentration in a homogenous sample very precisely. For inhomogeneous samples the gamma line emission rate depends on the exact elemental distribution within the sample probed as the neutron flux and therefore the gamma yield changes due to neutron scattering and neutron absorption. A precise determination of the elemental composition is thus not possible for inhomogeneous samples with classical PGNA methods. A way to improve PGNA is to use the capabilities of pulsed neutron sources which are becoming more common. As neutrons with different energies probe different volumes within a sample, the correlation of the neutron energy to the time resolved gamma spectra allows an indirect access to depth resolved information of the elemental composition. By extending PGNA with a Time-of-Flight (TOF) option, information about the homogeneity as well as depth resolved elemental composition can be obtained.

We will present OpenMC simulations as well as an experimental investigation done at the RANS source in RIKEN [1]. We will show the results for an idealized sample based on a layered structure where the position of an inhomogeneity can be changed. The simulation as well as the experiment show that it is possible to access depth resolved information and thus improve the determination of the elemental composition in an inhomogeneous sample.

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## Radiochemical and Preconcentration Neutron Activation Analysis

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### Szilard-Chalmers investigations of neutron-irradiated gold foils

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**Keywords:** Szilard Chalmers, <sup>198</sup>Au

In 1934 L. Szilard and T. A. Chalmers discovered changes in the chemical environment in the course of nuclear reactions. In an (n,γ) reaction, the resulting recoil energy from prompt gamma emission is big enough to break up the chemical bond between the activated atom and its residual molecule. Over the course of conducting numerous experiments the Szilard-Chalmers effect has been examined concerning the creation of the radioactive isotope Au-198 via the <sup>197</sup>Au(n,γ)<sup>198</sup>Au nuclear reaction. Various experiments involving gold foils have been tested to study in depth the effect and possible side-reactions taking place when the gold foil is placed in a liquid during activation. The experiments were carried out using the *TRIGA Mark II reactor* at the *TRIGA Center Atominstitut*.

## Simultaneous multielement speciation analysis using neutron activation

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**Keywords:** speciation, preconcentration, neutron activation

Neutron activation analysis (NAA), in particular instrumental NAA (INAA), is a well-established analytical technique for the simultaneous determination of multielement concentrations in a variety of matrices. Although various forms of INAA and radiochemical NAA (RNAA) have traditionally been applied to measuring the total concentrations of elements, the scope of NAA can be further extended in conjunction with pre-irradiation chemical separations, commonly called preconcentration NAA (PNAA), to determine the species of an element. We called it speciation NAA (SNAA). We have further extended SNAA to simultaneously determine several species and termed it simultaneous SNAA (SSNAA). Since much of the toxicity of an element depends on its physico-chemical forms, commonly referred to as its species, there is an increasing interest in studying elemental speciation. A number of characteristic features of NAA, which other techniques normally do not possess, can also be advantageously exploited in SNAA and SSNA. For example, we have used SSNAA for the: (i) simultaneous multielement speciation with high specificity, (ii) speciation of elements which are not chemically similar such as Cd, Mn and Se, (iii) speciation of elements such as Cl, Br and I which are rather difficult to determine by most other analytical techniques, etc. We have been developing SSNAA methods in our laboratory for more than 20 years. This technique can be reliably used for the simultaneous determination of not only various species of a single element, for example As(III) and As(V), but also species of other elements, such as Sb(III), Sb(V), Se(IV) and Se(VI) present in the same sample. We have also developed SSNAA methods for separating various inorganic arsenic species from its organic counterparts, such as monomethylarsonic acid (MMA) and dimethylarsinic acid (DMA), in water and seafoods which are rather difficult to measure accurately by many other techniques. Furthermore, we have developed SSNAA methods to characterize protein-bound trace element species in biological macromolecules. An overview of the SSNAA methods developed in our laboratory will be presented.

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## Reference materials in AA

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### Optimization of Neutron Activation Analysis for a cellulose reference material using Compton suppression and gamma – gamma coincidence methods

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**Keywords:** trace elements, Compton suppression, gamma-gamma coincidence

Typical trace elemental concentrations in biological materials are much lower than geological or many environmental specimens. Conversely, biological specimens often have high amounts of sodium, chlorine and bromine where its activation products through the following reactions  $^{23}\text{Na}(n,\gamma)^{24}\text{Na}$ ,  $^{37}\text{Cl}(n,\gamma)^{38}\text{Cl}$ , and  $^{81}\text{Br}(n,\gamma)^{82}\text{Br}$  give rise to high backgrounds due to Compton scattering. In addition, biological samples have elevated concentrations of phosphorous which characteristically has residual Bremsstrahlung radiation in the lower of the spectrum due to the pure  $\beta$ -decay of  $^{31}\text{P}(n,\gamma)^{32}\text{P}$  reaction. We have judiciously used thermal and epithermal neutron activation analysis in conjunction with Compton suppression and gamma-gamma coincidence to determine a series trace elements in a new IAEA reference material.

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**Related techniques (Charged-Particle AA, Photon-induced AA, ...)**

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**Determination of isotopic composition of lead using prompt and decay gamma-rays by muon induced nuclear reaction**

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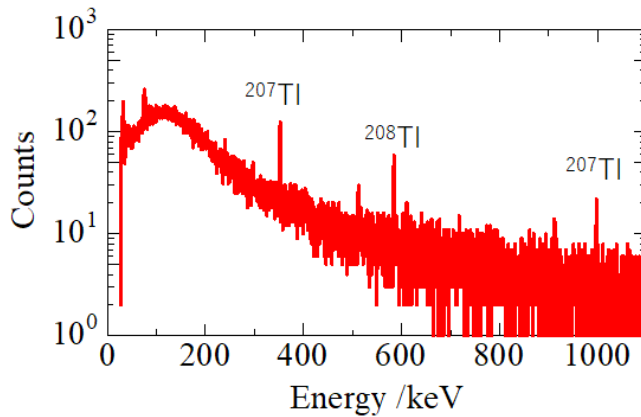
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**Keywords:** muon, non-destructive analysis, muon induced analysis, muonic atom

In recent years, non-destructive elemental analysis method using negative muon beam has been developed, and has already been applied to heritages and valuable extraterrestrial materials [1,2]. This method is based on characteristic muonic X-ray measurement emitted after muon stop in a material and formation of a muonic atom. We have also developed a non-destructive isotopic analysis method using isotope shift in muonic X-ray energies [3]. Here, we focus on gamma-rays emitted by muon induced nuclear reaction as a new probe for isotopic identification.

After formation of muon atoms, some muons are absorbed by the nucleus and cause muon induced nuclear reactions before muon decay with 2.2  $\mu$ s lifetime. In this reaction, one of the protons in the nucleus is converted into a neutron, similar to EC decay, and also the mass energy of a muon (106 MeV/c<sup>2</sup>), excluding kinetic energy of neutrino, is provided into to the nucleus. In this way,  ${}_AZ-1$  having several tens of MeV excitation energy, is formed when a muon is captured by a nucleus of  ${}_AZ$ . The excited  ${}_AZ-1$  nucleus immediately emits some neutrons and/or gamma-rays (prompt gamma-rays). Also, if the  ${}_AZ-1$  ( $n$  is the number of emitted neutrons) is a unstable nuclide, gamma-rays by radioactive decay (decay gamma-rays) are also emitted. The yield of produced nucleus is unique for the muon absorbed nucleus. Therefore, by measuring the prompt and decay gamma-rays, the muon capture nucleus and isotopic abundance of the sample can be investigated.



To demonstrate the new isotope analysis method, we selected lead isotope as the first target. Because the isotope ratio of lead differs by several percent depending on the origin, isotope analysis of Pb has been used to identify origin of lead product. We conducted muon experiments at the muon experimental facility in J-PARC (Japan Proton Accelerator Research Complex). We irradiated muon on single lead isotopes with mass of 204, 206, 207 and 208, and investigated the gamma-ray intensity pattern from each isotope. The durations of muon irradiations were about 8 hours. Furthermore, muon irradiation for a lead sample having natural isotopic abundance was also performed. By focusing on decay gamma-rays originating from  $^{208}\text{Tl}$ , the isotopic abundance of mass number 208 in the natural lead sample was determined to be  $55.2 \pm 1.9\%$ . The result was consistent with the  $^{208}\text{Pb}$  isotopic abundance of 52.9% determined from mass spectrometry.

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## User facilities

### International user access possibilities of Neutron Activation Techniques at the Budapest Neutron Centre (BNC)

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**Keywords:** instrumental neutron activation analysis, prompt-gamma activation analysis, user laboratory

Instrumental Neutron activation analysis (NAA) and prompt-gamma activation analysis (PGAA) are highly appreciated techniques for the non-destructive and bulk-representative elemental composition measurement of samples. The Budapest Neutron Centre (BNC) is one of the few neutron centers in the world, where these two techniques can be used together, and can also be coupled with cold neutron tomography (NIPS/NORMA) and thermal neutron & X-ray imaging (RAD) if needed. The recently experienced decline in the number of operational reactor-based neutron sources in Europe limited the access possibilities to NAA and PGAA measurements.

The NAA and PGAA laboratories of the Budapest Neutron Centre (BNC) serve the domestic and international user community, in addition to their significant contributions to method developments (e.g.  $k_0$ -NAA method), in-house research programs, and internal services of reactor operation.

Since 2004, numerous user access programs, as NMI3, BNC's own user program, CHARISMA, IPERION CH, IPERION HS, E-RIHS, BNC-LENS, CERIC-ERIC, REMADE@ARI, NEPHEWS are working on the integration of neutron research infrastructures within the European Research Area. Some of them are topic-oriented transnational access programs that target for example: heritage science, environmental sciences, material science, and/or offering nuclear training activities with the coordination of the IAEA. BNC offered beamtime in 2022 to the users of other European neutron facilities, where their accepted proposals were delayed due to prolonged downtimes (BNC LENS beamtime initiative). About half of these proposals requested elemental composition measurements.

The broad user community covering different scientific fields comes up with diverse measurement needs regarding the matrix of samples and the elements of interest. Thus, a high level of service for the user community requires great technical and organizational considerations. The labs need operation flexibility and a deep understanding of the method to realize these experiments. NAA and PGAA are based on the same nuclear physical processes, however, the irradiation and measurement location, and also the timing of gamma-ray detection are different, so scheduling is crucial when planning the work and also the participation of users in the experiment. In all cases, the discussion, the interpretation, and the publication of the results are done together with the users.

The increase of remote access opportunities and automatization of many processes in measurements as well as the data evaluation form new operation routines in our laboratories. Implemented innovations in the laboratories and the research highlight of these user access collaborations will be present to illustrate the prosperity of this operational model.



## Fast neutron beam facilities at iThemba LABS and the University of Cape Town, South Africa

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**Keywords:** fast neutron beam facility, AmBe source, neutron metrology, neutron activation, HPGe

iThemba Laboratory for Accelerator Based Sciences (iThemba LABS) is a national research facility in South Africa with activities at the facility based on a number of sub-atomic particle accelerators. The largest of these, a K=200 separated sector cyclotron, which accelerates protons of energies up to 200MeV, and heavier particles to much higher energies [1]. The iThemba LABS neutron beam facility (D-line) offers ns-pulsed neutron fields in the range of 25-200 MeV using <sup>7</sup>Li and <sup>9</sup>Be targets of varying thickness to produce quasi-monoenergetic neutrons beams via (p,nx) reactions [2]. The spectral distribution of these beams shows a high-energy peak resulting from transition to the ground state and the first excited states the product nucleus and an adjacent continuum resulting from break-up reactions. At present, the standard procedure for characterising the neutron beams and measuring spectral neutron fluence involves a 2'' x 4'' BC-501A liquid scintillator and a parallel-plate <sup>238</sup>U fission chamber (FC) [3]. The meteorological characterisation of the beams is based on the time-of-flight (TOF) and unfolding methods. The D-line facility is one of the few facilities available to provide quasi-monoenergetic neutron beams in these energy range, 25-200 MeV [4]. The D-line is currently undergoing a major upgrade and redevelopment towards ISO/IEC 17025 accreditation. The upgrade includes physical modifications of the vault, new instrumentation for neutron metrology and improved beam monitoring and control systems. For high-energy neutron activation of samples, the facility utilizes also the available low-background HPGe detector system at iThemba LABS.

In addition, the University of Cape Town fast neutron beam facility (n-lab) features a Thermo MP-320 sealed tube neutron generator (STNG), and a 220 GBq AmBe source. The STNG is a small accelerator which produces neutrons of approximately 14 MeV via the deuterium-tritium fusion reaction and the AmBe source is a <sup>241</sup>Am-<sup>9</sup>Be radioisotopic neutron source which produces a broad energy spectrum of neutrons from the thermal region to around 11 MeV. The n-lab also operates a well-calibrated EJ-301 organic liquid scintillator as a reference neutron detector and a well-shielded HPGe detector system [5].

Combined, neutrons from these facilities can be collimated into a beam for fundamental research and applications. Typical applications include neutron induced cross section measurements, development of instrumentation and methods for neutron metrology, non-destructive testing as well as elemental analysis of materials. For this contribution, we aim to discuss activities using fast neutron beam facilities at iThemba LABS and at the University of Cape Town.

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# Poster Presentations

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## AA facilities used by the industry, industrial applications

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### Innovative approach for sustainable and low-waste production of $^{99}\text{Mo}$ -based radiodiagnostics using accelerator-based neutron source

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**Keywords:** compact accelerator-based neutron sources, medical isotope production, activation, low-waste production,  $^{99}\text{Mo}$  from natural Mo, neutron capture method

Nuclear medicine diagnostics that are integral to modern healthcare, heavily rely on the radionuclide  $^{99}\text{Mo}$ , traditionally produced in nuclear reactors through the fission of  $^{235}\text{U}$  [1, 2, 3]. However, the complex radiochemical processing involved generates substantial radioactive waste, necessitating a shift towards more sustainable practices. This poster presents the  *$^{99}\text{Mo}$  Best* joint project, an initiative focused on developing an innovative, cost-efficient concept for the production and utilization of  $^{99}\text{Mo}$ -based radiodiagnostics, utilizing the  $^{98}\text{Mo}(n, \gamma)^{99}\text{Mo}$  reaction eliminating fissile materials and minimizing radioactive waste.

The project comprises three key sub-projects:

1. **Process Optimization:** This involves refining the processes for generating  $^{99}\text{Mo}$ -based radiodiagnostics, as well as improving their processing and utilization in clinical settings.
2. **Neutron Target Technology:** Developing high neutron flux density neutron target technology is crucial for irradiation with reduced radiation doses, ensuring safe handling and processing of Mo samples post-irradiation.
3. **Radiation Protection and Disposal:** Addressing safety concerns, this sub-project aims to determine radiation protection and disposal issues pertinent to the novel  $^{99}\text{Mo}$  production process, ensuring a secure and sustainable approach.

This comprehensive approach aims to create a paradigm shift in the field of nuclear medicine by offering a sustainable and efficient alternative to traditional  $^{99}\text{Mo}$  production methods, mitigating environmental impact and advancing the application of accelerator-based neutron radiation sources in medical radioisotope production.

**Acknowledgments**

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## **Applications of Activation Analyses**

### **Heavy metals from the oil spills incident in the Peruvian Sea, determined by $k_0$ -INAA and AAS**

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**Keywords:** heavy metals, oil spills,  $k_0$ -neutron activation analysis, atomic absorption spectrometry

The environmental impact of oil spills at sea has severe implications, can affect marine life by coating organisms, altering ecosystems, affecting the food chain and causing long-term damage. Oil spots can suffocate and kill fish, birds and other animals, while the toxic components of oil can affect reproductive and developmental processes. In addition, spilled oil can contaminate the coastlines, affecting beaches, mangroves and other habitats that are sensitive to this type of pollution. Contamination can be by heavy metals, as well as hydrocarbons. Elements such as lead, arsenic, cadmium which are toxic to marine flora and fauna, can accumulate in organisms and enter humans through the food chain. Other elements such as chromium, zinc and copper can also be harmful if are found in high concentrations and, also have adverse effects. Analytical techniques such as  $k_0$ -INAA and AAS complement each other favourably to obtain information on the mentioned elements and others. For this purpose, two types of sediment samples have been analysed: one at the site of the incident and the other one, at a distance of 135 kilometres opposite to the tidal current to be used as a control sample. Results were compare with the sediment quality guidelines for the protection of aquatic life from the Canadian Council of Ministers of the Environment and the control sample. While only lead shows a slightly higher value than that given in the guideline, the concentration of the heavy metals studied are above that of the control sample, which would indicate that there has been heavy metal contamination. In addition, the concentration factor (CF) obtained showed contamination of  $Cd > Pb > Cr > Zn > Cu$  and the enrichment factor (EF) confirms that the source of contamination is of anthropogenic origin.

## Study of spatial and temporal trace element atmospheric deposition in Romania using moss biomonitoring and combined analytical techniques (INAA, ICP-MS and AAS)

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**Keywords:** trace elements, heavy metals, mosses, atmospheric deposition, Romania

In this paper combined analytical techniques were used for the quantification of 30 trace elements in naturally growing mosses collected from various sites in Romania, SE Europe: instrumental neutron activation analysis (INAA), inductively coupled plasma–mass spectrometry (ICP-MS) and atomic absorption spectrometry (AAS). The surveys were organized within the framework of the International Cooperative Programme on Effects of Air Pollution on Natural Vegetation and Crops (ICP Vegetation), comprising two campaigns for moss collection (2010/2011 and 2015/2016) from 303 and 214 sampling sites, respectively, over the Romanian territory. The changes of elemental levels in mosses were mapped using the cloud platform developed at JINR Dubna. Multivariate statistical analysis was applied to the obtained data yielding geogenic and anthropogenic factors and pollution sources. The distribution patterns indicate a decreasing trend of toxic metals concentrations. A comparison with results obtained in other countries was performed, shedding light on temporal trends of atmospheric trace elements deposition at European level.

### Acknowledgments

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## Manganese supply to chloroplasts for optimal photosynthesis

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**Keywords:** photosynthesis, manganese

Photosynthesis has great potential to resolve the modern challenges of the society related to alternative energy, food production, and sustainable agriculture. Having originated on our planet over 3.5 billion years ago, photosynthesis continues to contribute to the atmosphere, climate, and the evolution of life, including humans.

In plants and algae, manganese (Mn) is involved in multiple physiological functions including photosynthesis and various redox processes [1]. Photosystem II (PSII) is the most important Mn-dependent enzyme in a plant. The  $Mn_4CaO_5$  cluster is bound in a pocket formed by amino acid residues of the PSII matrix at the luminal side of the thylakoid membrane [2]. When PSII undergoes degradation, Mn is released in the thylakoid lumen and recycled to build a new cluster. However, little is known about the mechanisms that supply Mn to PSII as well as on how chloroplasts reorganize their membranes and photosystems to cope with Mn deficiency.

The study addresses Mn import and incorporation into chloroplasts, particularly in response to Mn resupply in deficient plants. Isolated chloroplasts of sugar beet (*Beta vulgaris* L. cv. Orbis) were utilized for Mn content analysis as the primary model. To investigate the light dependency of Mn uptake into chloroplasts, changes in Mn content in washed chloroplast suspensions were analyzed after incubation in both light and dark conditions. As chloroplast fractions of the plant cells are highly sensitive, only a very small amount of the sample is possible to obtain, therefore, routine methods of measuring Mn concentration like mass spectroscopy are not applicable and the application of neutron activation analysis is required [3]. The concentration study was accompanied by analysis of Mn speciation using electron paramagnetic resonance.

### Acknowledgments

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## Fast Neutron Activation Analysis

### Determination of the silicon content of geological materials by reactor fast neutron activation analysis (FNAA)

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**Keywords:** FNAA, silicon analysis, geological materials

Determining the silicon concentration of geological materials, particularly igneous rocks, is highly desirable to the geologist because the silica ( $\text{SiO}_2$ ) content of such samples is an important criterion used in their classification and is of considerable value when elucidating the evolutionary history of rock suites.

Hancock<sup>1</sup> described the analysis of silicon in archaeological pottery samples using a SLOWPOKE reactor and Cd shields and reported that the analytical precision of individual silicon measurements was better than  $\pm 5\%$ . While this precision may be acceptable for provenance and technology studies in archaeology significantly better precision is required for the determination of silicon in rocks for petrogenetic studies and classification purposes. Additionally, the use of Cd shields for fast (and epithermal) NAA employing short-lived radionuclides presents several drawbacks. Cadmium is a toxic heavy element which, upon neutron irradiation, readily activates producing  $^{111\text{m}}\text{Cd}$  ( $T_{1/2} = 48.5$  min) and to a lesser extent  $^{115}\text{Cd}$  ( $T_{1/2} = 53.46$  h) which pose potential radiation hazards. This latter risk is more pronounced when Cd shields undergo repeated irradiations with minimal decay intervals.

Similar to Hancock, our laboratory developed and extensively employed a rapid method for determining the silicon content of rocks and minerals using a SLOWPOKE reactor, FNAA and the transmutional  $^{29}\text{Si}(n,p)^{29}\text{Al}$  ( $T_{1/2} = 6.56$  min) reaction. However, unlike Hancock's method our approach employs the use of a boron-carbide shield which has several advantages compared to the use of Cd shields. Furthermore, in addition to quantifying the Si content of geological materials by FNAA the *simultaneous determination* of U and Co (largely by epithermal NAA), and Mn and V (by activation predominantly with the residual thermal neutron flux) is also generally possible.

Since the initial development of our method employing  $\text{B}_4\text{C}$  shields there have been significant improvements in gamma-ray signal processing (*e.g.*, digital spectrometry) and in the efficiency of Ge detectors, for example. The impact of these advances, together with the benefits of using shields fabricated using  $\text{B}_4\text{C}$  and  $^{10}\text{B}$ -enriched boric acid, for the FNAA of silicon in geological materials will be presented.

Results will be presented demonstrating that for geological studies FNAA provides a rapid and competitive means of silicon analysis. In comparison to XRF, the method traditionally used to quantify silica and other major and trace elements in geological samples, the FNAA approach is non-destructive, essentially matrix-independent and requires a fraction of the



sample mass typically needed for whole rock XRF analysis. Silica, Mn, U, V and Co concentrations are reported for a number of USGS rock standards demonstrating the accuracy and reproducibility of the method together with examples of applications of the method.

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## Nano-sized $^{90}\text{Y}$ and $^{188}\text{Re}$ production via medium reactor and cyclotron

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**Keywords:** direct technique, medium reactor, indirect technique, compact cyclotron, nano solution, neutron activator, neutron flux, specific activity, beta emitter, transmutation, production yield

The main objective of the coordinated project development of Y-90 and Re-188 therapeutic radiopharmaceuticals is to exploit advances in radionuclide production technology. Here, direct and indirect production techniques via medium reactor and compact cyclotron are compared to evaluate derived neutron flux and production yield. First, nano-sized  $^{186}\text{W}$  and  $^{89}\text{Y}$  samples are suspended in water in a quartz vial and simulated by MCNP code. The solution is then irradiated in the reactor for 4 days under a neutron flux of  $9\text{E}+14$  n/cm<sup>2</sup>/s. In addition, a neutron activator with three layers – lead moderator, graphite reflector, and polyethylene absorbent – is simulated and a tungsten target is irradiated with 60 MeV protons from a cyclotron to generate induced neutrons for  $^{188}\text{W}$  and  $^{90}\text{Sr}$  production indirectly via neutron capture. As the neutron energy decreased, the flux gradually increased toward the epithermal range to satisfy  $(n/2n,\gamma)$  reactions. The acquired specific activities at saturation were higher than the reported experimental amounts because the accumulated epithermal flux and nano-sized samples influence the results. The beta emitters, which are widely used in brachytherapy, represent an alternative way to achieve a rational yield locally. Therefore, the proposed technique using neutron activator can meet these broad requirements. Although  $^{90}\text{Y}$  does not have gamma radiation, complicating dosimetric evaluations, its high-energy beta emission allows local delivery of high doses with lower risk of induced necrosis due to limited tissue penetration. Also, the availability of a kit formulation process without mutated radioactive type in the generator results in the treatment being fractionated or repeated. Because  $^{188}\text{Re}$  has shorter half-life compared to  $^{90}\text{Y}$ , approximately five times more activity is required to achieve an equivalent patient dose. On the other hand, the unavailability of the kit formulation process and the limited accessibility of the  $^{188}\text{W}/^{188}\text{Re}$  generator are disadvantages of the  $^{188}\text{Re}$  radionuclide. Alternatively, an indirect technique using a compact medium-range cyclotron was proposed here, which can be used locally in hospitals with a rational production yield.

### Highlights

- $^{89}\text{Y}(n,\gamma)\text{Sr}^{90}$  and  $^{186}\text{W}(2n,\gamma)\text{W}^{188}$  reactions were simulated to assess  $^{90}\text{Y}$  and  $^{188}\text{Re}$  generators.
- The neutron source from medium-range reactor and compact-cyclotron was simulated by MCNP code.
- Nano sample-water solution in quartz vial was located in core and a proposed neutron activator.
- By cyclotron technique using neutron activator, accumulated flux increased toward epithermal range.
- Production yields of beta emitters were greater than reported ones.

## Instrumental Neutron Activation Analysis

### Monte Carlo calculation of self-shielding factor for instrumental neutron activation analysis: Determination of lanthanides concentration in phosphates

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**Keywords:** INAA, Monte Carlo simulation, phosphate matrix, neutron flux

The instrumental neutron activation analysis (INAA) technique was used for the precise determination of lanthanide concentrations present in phosphate samples collected from the phosphate deposits of Jebel El Onk, eastern Algeria. The samples were irradiated under a thermal flux of  $2.36 \cdot 10^{13} \text{ n cm}^{-2} \text{ s}^{-1}$  and an epithermal flux of  $8.7 \cdot 10^{11} \text{ n cm}^{-2} \text{ s}^{-1}$  in a research nuclear reactor (CRND). The irradiation of the samples in the neutron field of a nuclear reactor is affected by the local perturbation of the neutron fluxes. Mainly produced by the sample, particularly in the case where the size of the sample during the analysis by instrumental neutron activation may not be small enough, self-shielding effects must be taken into account, when several absorbers are present in the sample. This requires knowledge of two corrective parameters: the thermal neutron self-shielding factor and the epithermal self-shielding factor.

In this study, a comprehensive procedure was developed using the Monte Carlo simulation for the calculation of thermal and epithermal neutron self-shielding factors in the phosphate sample. The results obtained by Monte Carlo simulation of the self-protection factors of thermal and epithermal neutrons are respectively  $G_{\text{th}} = 0.976$  and  $G_{\text{epi}} = 0.999$ , which were subsequently applied for the determination of lanthanide concentrations with good precision and to improve the INAA detection limit.

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## Elemental characterisation of illicit sildenafil products on the Jamaican market using instrumental neutron activation analysis (INAA)

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**Keywords:** INAA, Viagra®, forensic analysis, counterfeit pharmaceuticals, chemometrics

Sildenafil, generally branded as Viagra®, is one of the most counterfeited pharmaceuticals internationally. This high level of counterfeiting has been noted in Jamaica by Pfizer, the parent company for this erectile dysfunction drug [1]. Neutron activation analysis, specifically instrumental neutron activation analysis (INAA) has been employed in the characterization of illicit drugs [2]. Samples of illegal sildenafil products were procured in an area of the capital city of Kingston known for the sale of fake pharmaceuticals. The samples were brought to the International Centre for Environmental and Nuclear Sciences (ICENS), where they were prepared for analysis. Genuine Viagra® samples were also procured and prepared in the same manner. The samples were analysed for Al, As, Br, Ca, Ce, Cl, Co, Cr, Cs, Fe, La, Mg, Mn, Na, Sc, Sm, Sr, Th, and Zn by INAA using the SLOWPOKE-2 research reactor at the ICENS. Previous research on non-genuine sildenafil products sourced internationally has indicated that the levels of specific elements and their relationship to each other can distinguish Viagra® from fake sildenafil products [3]. A similar chemometric approach was applied to these samples. The results indicate that the methodology is robust enough to differentiate authentic Viagra® from counterfeit sildenafil products found on the international as well as the Jamaican market. Furthermore, the analysis indicates that using this methodology, the classification of counterfeit versus authentic Viagra® can be determined using a short irradiation time (3 minutes) and relatively low thermal neutron flux ( $5 \times 10^{11} \text{ cm}^{-2} \text{ s}^{-1}$ ) allowing for fairly rapid analyses of the determination of counterfeit samples by INAA.

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## **A review of total, bioaccessible fraction, and speciation analysis of iodine in nutritional materials at nanomolar levels with low overall expanded uncertainties by neutron activation at the Dalhousie University SLOWPOKE-2 Reactor Facility**

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**Keywords:** iodine, foods, neutron activation, low overall uncertainty

Iodine is considered an essential trace element for humans. Iodine plays very important roles in the synthesis of thyroxine (T4) and triiodothyroxine (T3) hormones which control oxidation/reduction ratio in cells and are responsible for many functions such as metabolism, blood circulation, muscle activity, and thermoregulation. Deficiency of iodine can lead to goitre disease commonly referred to as iodine deficiency disorder (IDD). The World Health Organization estimates that about 1.6 billion people around the world are at a risk of IDD. Excessive iodine intake can induce hypothyroidism and may contribute to autoimmune thyroid disease in susceptible individuals over the age of 40. Marine fish, seaweeds, eggs and milk are generally the richest sources of iodine in diets. Milk provides 16-30% of the daily dietary iodine intake. Iodine levels in other foods tend to vary quite a bit. Iodide is the most common form of iodine in foods and readily bioavailable. A considerable portion of iodine in muscle meats is bound to proteins although that in milk is not. Much of the iodine in fish is in inorganic form and highly bioavailable. Obviously, total iodine, its chemical species and bioavailable fraction are of much interest in both nutritional and toxicological studies. The determination of iodine at trace levels with high precision and accuracy in nutritional materials is a rather difficult task. To study total, bioaccessible fraction, and speciation of iodine in nutritional materials we have developed several neutron activation analysis (NAA) methods over the decades in our laboratory using the Dalhousie University SLOWPOKE-2 Reactor (DUSR) facility and are summarized here. These NAA methods include instrumental NAA (INAA), epithermal INAA (EINAA), INAA-anticoincidence (INAA-AC), EINAA-anticoincidence (EINAA-AC), Pseudo-cyclic INAA (PC-INAA), PC-INAA-anticoincidence (PC-INAA-AC), PC-EINAA-anticoincidence (PC-EINAA-AC), k0-INAA, Preconcentration NAA (PNAA), and radiochemical NAA (RNAA). We have evaluated these methods for interference, rapidity, accuracy, precision, overall expanded uncertainty, and detection limits. Results will be presented and discussed.

## Investigation of uranium analysis using $^{140}\text{Ba}/^{140}\text{La}$ induced from $^{235}\text{U}$ fission reaction

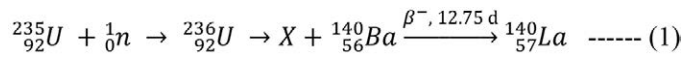
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**Keywords:** U determination, U fission products,  $^{140}\text{La}$  nuclide, INAA, HANARO reactor

In the recent studies, monitoring of uranium in the environment and geological samples has been increased due to contamination, and diverse applications. Instrumental neutron activation analysis (INAA) which has an advantage of simple and non-destructive method, is one of the suitable techniques for trace level uranium analysis in the geological and environmental samples. In general, the INAA method can determine the uranium concentration by measuring a  $^{239}\text{Np}$  nuclide ( $\gamma_E$ : 106 keV,  $\gamma_a$ : 22.7%;  $\gamma_E$ : 228 keV,  $\gamma_a$ : 10.7%;  $\gamma_E$ : 278 keV,  $\gamma_a$ : 14.1%) produced from neutron capture reaction of  $^{238}\text{U}$  nuclide. However, gamma-ray interference and high background contribution at low energy region make an effect on inaccurate analytical value and high uncertainty. Besides the  $^{238}\text{U}(n, \gamma)^{239}\text{U}/^{239}\text{Np}$ , a variety of fission products are produced by  $^{235}\text{U}$  fission reaction during the irradiation of analytical sample in instrumental NAA technique. Some nuclides of fission products induce fission interference for INAA and correction factors were experimentally determined.  $^{140}\text{La}$  is one of the daughter nuclide to be useful for trace back to U content. The  $^{140}\text{Ba}$  ( $t_{1/2}$ : 12.95 days) is a fission product (Eq. 1) and decayed to  $^{140}\text{La}$  ( $t_{1/2}$ : 40.28 hrs). Therefore, the correction factor of  $^{140}\text{La}$  varies with the decay time of an irradiated sample, and analytical value of La changes as well. In this work, we have described the analytical method for effective determination of uranium using fission induced product of La in geological and environmental samples. Three different concentration levels of U of NIST SRMs (2709a-Sanjoaqiuin Soil, 2710a-Montana Soil, 1632C-Coal) with certified or reference values were selected to validate the methodology. About 250 mg of samples were irradiated at the NAA#1 irradiation hole at the HANARO research reactor in Korea. The NAA#1 irradiation hole has high  $f$  value ( $\Phi_{th}/\Phi_e \sim 1000$ ), and high possibility of the formation of fission product like  $^{140}\text{La}$ . The first measurement for the detection of  $^{140}\text{La}$  was performed within a week cooling period and the second measurement was performed during 21~23 days' cooling time. The U contents in the SRM samples can be determined by using two different correction factors and La concentrations calculated at different cooling times, respectively. The relative deviation of the analytical results is agreed within 10% of certified or reference values of U in SRM samples. Finally, this method has been applied for the determination of U in NIST SRM 1648a-Urban Particulate Matter, which has no certified value. The measured value of U in SRM 1648a is  $4.90 \pm 0.20$  mg/kg with a 68% confidence level. This method can be used as an alternative method for the analysis of U in actual samples through INAA.

**Acknowledgements**

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## Compositional analysis of AuPd nanoparticles using INAA

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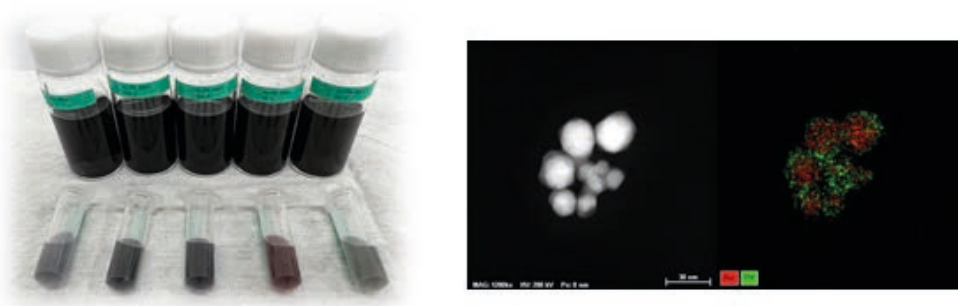
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**Keywords:** HANARO reactor, Au-Pd alloy, nanoparticles, catalyst, Instrumental Neutron Activation Analysis (INAA)

The swift advancement in nanotechnology necessitates highly proficient analytical techniques that are urgently required for characterizing newly developed materials, assessing their potential toxicity on ecological and biological systems, and comprehending nanoparticle formation. AuPd alloy nanoparticles are being studied in various fields for catalytic or medicinal applications, and quantitative evaluation of their composition is essential for their application. Unfortunately, it is difficult to quantitatively analyse the composition of metal nanoparticles synthesized in alloy form. This study employs Instrumental Neutron Activation Analysis (INAA) method, specifically neutron irradiation position at NAA#1 operated at HANARO reactor, to analyze synthesized AuPd alloy nanoparticles with different composition. The AuPd alloy nanoparticles were prepared using modified Turkevich's method and after synthesis, impurities were removed by dialysis. The NAA#1 is a facility capable of irradiating neutron fluxes of about  $4.8 \times 10^{13}$  n.cm<sup>-2</sup>.s<sup>-1</sup> at reactor operating powers of up to 30 MW, and is being utilized as a primary method for non-destructive composition analysis of a wide variety of samples. The INAA method, well-established in speciation and metallomics studies, offers a potent alternative too for future nanoparticles research.



**Figure 1** TEM (left) & EDX (right) image of synthesized AuPd nanoparticles.

### Acknowledgments

This work was supported by the Korea government (MSIT) (1711078081) and the National Research Foundation of Korea (NRF) grant funded by the Korea government (MSIT) (RS-2023-00237149).



## The determination of several rare-earth elements using short- and medium-lived epithermal Neutron Activation Analysis Products

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**Keywords:** trace elements, Compton suppression, gamma-gamma coincidence

Analysis of rare earth elements (REEs) using neutron activation analysis (NAA) can normally be difficult due to a variety of factors including spectral interferences, high Compton continuum backgrounds and the production of the identical isotopes due to uranium fission. To optimize the evaluation of REE concentrations within geological samples, epithermal NAA was used in conjunction with Compton suppression techniques to calculate the concentrations of Dy, Sm, Gd, and Ho. A special consideration is given to the several spectral interferences for the determination Sm. The elements of Gd and Ho are rarely reported in routine NAA and are reported here for several geological reference materials.

# INAA of bone remains of Bohemian Duke John of Görlitz: An attempt to explain his sudden death in the age of twenty-five years

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**Keywords:** instrumental neutron activation analysis, bone, aluminium, trace elements, cultural heritage

John of Görlitz (1370–1396), was a member of the House of Luxembourg and the only Duke of Görlitz (Zgorzelec) from 1377 until his death. He was the third son of Emperor Charles IV (1316–1378), Holy Roman Emperor and Bohemian King, from his fourth marriage with the princess Elizabeth of Pomerania (1347–1393). John of Görlitz was a younger half-brother of Wenceslaus IV, elected King of the Romans (1376–1400) and Charles' IV successor on the Bohemian throne (1378–1419). The Duke of Górlitz died unexpectedly on March 1, 1396, in Neuzelle Monastery in Lower Lausitz. The cause of his death is still unknown. The sudden death triggered speculations and rumours about him being poisoned, which was typical for similar cases in the Middle Ages. Recently, his ilium bone underwent histomorphometry examination accompanied, among others, by histochemical staining reaction for Al (aluminon technique). Results of the histomorphometry examination will briefly be discussed in the presentation. The histochemical staining reaction yielded linear red deposits on the surface of the bony trabeculae localized between their mineralized and unmineralized parts. This finding resembled aluminium osteopathy in dialysed patients. For this reason, the bone was assayed by INAA for a number of minor and trace elements to verify whether the red deposits are really due to an elevated Al content in the bone or whether they are due to elevated levels of other, interfering elements.

A slice of the bone sample was removed from a block of poly(methyl methacrylate) glass (PMMA) used for the histomorphometry examination. The major part of PMMA was removed mechanically, the remaining part by dissolution of PMAA in chloroform. After removing the possible external contamination by washing in dilute HNO<sub>3</sub>, demineralized water, and drying, the bone sample was assayed by INAA using both short- and long-time irradiation (30 s and 3 h, respectively) in a neutron flux of  $\sim 3 \times 10^{13} \text{ cm}^{-2} \text{ s}^{-1}$  in the LVR-15 reactor in Řež. Simultaneously, a sample of NIST SRM 1486 Bone Meal was irradiated for quality control, and a piece of mechanically removed PMMA as a process blank. The irradiated samples were counted with HPGe detectors connected to a Canberra Genie gamma-spectrometer. For quantification of element contents, relative standardization was employed using calibrators prepared from certified solutions. In total, 34 macro-, minor- and trace elements were determined. The agreement of the element contents determined in NIST SRM 1486 with NIST certified and/or information values proved accuracy of our results. The results for Duke's of Görlitz bone were compared with literature values. From this comparison, it could be concluded that the Al content was not elevated in the analysed bone (although only a value below the detection limit mostly affected by the  $^{31}\text{P}(n,\alpha)^{28}\text{Al}$  interference reaction with fast neutrons could be determined), whereas the values for Mn, As, Sb, and especially for Ag

were significantly higher. The results obtained are discussed in terms of the specificity of the aluminum staining reaction, possible effects of elevated contents of the above elements on Duke's health status, and a possible reason for his sudden death. This work is an example of the use of INAA for cultural heritage studies.

**Acknowledgments**

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## Elemental analysis of blended cement samples in Korea by neutron activation analysis

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**Keywords:** neutron activation analysis, cement, toxic element

Cement is a critical construction material widely utilized worldwide, playing a crucial role in infrastructure development and urbanization. Recently, there has been a noticeable market demand for cement products blended with sand and hardener, particularly for interior design and DIY projects. However, these products are susceptible to exposure to harmful elements such as heavy metals due to the diverse range of raw materials used in their production. In this study, we conducted an analysis of trace elements in cement products in Korea using neutron activation analysis (NAA).

A total of 13 blended cement samples were prepared, comprising seven gray and six white samples. These samples underwent neutron irradiation at HANARO in September 2023 using a pneumatic transport system (PTS). After cooling time, gamma rays from each sample were measured by the HPGe detector for both short and long irradiation periods. The NIST SRM 1889b (a portland cement blended with limestone) was used as the reference material.

Figure 1 highlights toxic elements such as As, Br, Cr, and W. Some of samples contained high level of toxic elements. The chromium concentration of six samples (#2, #3, #5, #11, #12, and #13) exceeds 20 mg/kg. The maximum concentration of Cr exceeds 130 mg/kg. Even if only a portion of the chromium concentration is Cr (VI), this level is remarkably high. According to the domestic voluntary agreement regulation, the concentration of Cr (VI) should not exceed 20 mg/kg. However, compliance with this voluntary agreement regulation is not mandatory. The European Union imposes strict regulations, limiting the concentration of Cr (VI) in cement to 2 mg/kg, which is ten times lower than the agreement in Korea. This high chromium concentration is primarily attributed to the inclusion of waste materials during cement production, as there are no specific regulations addressing waste content or heavy metal concentrations in Korea. Enhancing domestic regulations to restrict the use of waste from industrial zones as raw materials for cement production would be essential in ensuring the availability of safe construction materials.

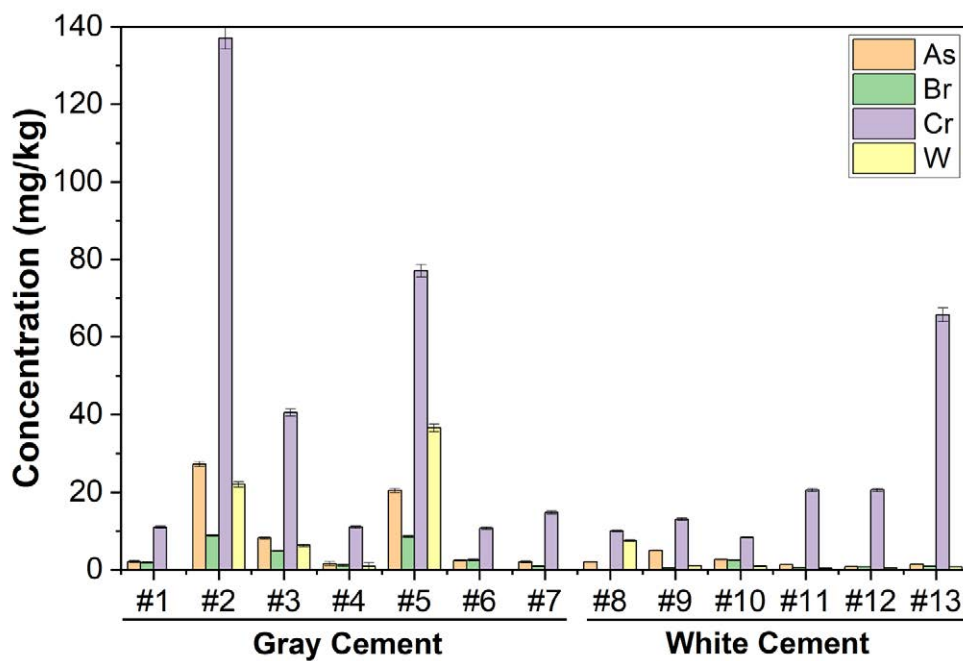


Fig. 1. Elemental concentrations (As, Br, Cr and W) of the cement samples

## Development of an organic coffee leaves reference material for foliar diagnosis

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**Keywords:** *Coffea Arabica*, mineral nutrition, elemental analysis

Brazil has been the world leader in the production of Arabica coffee for a century and has gained space in the specialty coffee market, with emphasis on organic coffees. To obtain quality coffees, it is necessary to meet all the nutritional demands of the coffee plant. Therefore, it is essential to carry out a foliar diagnosis before fruiting begins to assertively supplement nutrients. There are certified reference materials (CRM) for elemental analysis of coffee beans (KRISS CRM 108-10-023<sup>1</sup> and CRM-Agro C1007a<sup>2</sup>), however none produced from coffee leaves. In this context, the development of certified reference materials suitable for foliar diagnosis is of great relevance. The CRM-Agro C1009a Folhas de Café was produced in the Collaborating Center for Agricultural Defense CRM-Agro Reference Materials for Agriculture, Livestock and Toxicology at Radioisotopes Laboratory, CENA/USP. The species *Coffea arabica* cv Catuaí vermelho IAC 99 was selected on a certified organic farm, located in the municipality of Ibiraci, state of Minas Gerais, Brazil. A total of 30 kg of coffee leaves was collected, cleaned, sieved at 250 µm (100% passing), homogenized in a blender, radiation sterilized at 15 kGy to 20 kGy (<sup>60</sup>Co) dose, and then packaged in 210 amber polyethylene bottles each containing 25 g. Neutron activation analysis (NAA) was used to determine the chemical elements. The samples were irradiated in the IEA-R1 nuclear research reactor, Nuclear and Energy Research Institute of the National Nuclear Energy Commission (IPEN/CNEN), in São Paulo, SP, Brazil. The homogeneity of the material was assessed by analyzing triplicate samples of approximately 200 mg from ten bottles selected by random sampling. The stability of the material under transport and long-term conditions was also evaluated. For analytical quality control, SRM1515 Apple Leaves, IAEA 336 Trace and Minor Elements in Lichen and CRM-Agro C1005a Sugarcane Leaves were used. The mass fraction of chemical elements Ba, Br, Ca, Co, Fe, K, La, Na, Rb, Sc, Sm, Sr and Zn were quantified. No statistically significant differences ( $p > 0.05$ ) between-bottles and within-bottle were observed for the elements at the sample size used. No changes were observed in the mass fractions of chemical elements under transport conditions and over 12 months of storage ( $p > 0.05$ ). This innovative organic coffee leaf reference material is expected to be useful for assuring the quality of measurement procedures of elemental contents in leaf diagnosis.

### Acknowledgments

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## Developing an in-house quality control material of bovine tail hair for multi-elemental analysis

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**Keywords:** reference values, matrix matching, NAA, TQ-ICP-MS

Brazil is recognized as the world second-largest producer and largest exporter of beef. Beef constitutes an excellent source of protein and essential nutrients to human health. Since the nutritional quality of beef is directly related to its mineral composition, it is important to evaluate the nutritional status of the animals during the rearing and finishing phases, allowing adjustments to the diet. Blood analysis is commonly used for this purpose, but only reflects the body condition at the time of collection. Hair has the advantage of accumulating chemical elements throughout the animal life, providing a temporal record, in addition to being a non-invasive sampling easy to store. Certified reference materials for this matrix are not readily available for analytical quality control. Therefore, we decided to produce an in-house quality control material (QCM) from bovine tail hair, in accordance with the ISO Guide 80:2014 guidelines. The main function of in-house QCMs is to provide laboratories with a means of verifying the accuracy of routine methods, evaluating parameters such as repeatability, intermediate precision and reproducibility of measurement results. Tail hair were collected from 5 high performance animals from Angus breed, raised in a certified Brazilian farm that operates in a vertical production system, being responsible for the processes of insemination, breeding, rearing, fattening, slaughtering and marketing of the final product, facilitating the traceability of the production chain. The samples were cleaned, cut with titanium blade scissors, and homogenized. Neutron activation analysis and triple quadrupole inductively coupled plasma mass spectrometry (TQ-ICP-MS) were used to determine the elemental profile of the Tail Hair-QCM: Al, As, Ba, Br, Ca, Cd, Co, Cr, Cs, Cu, Fe, K, La, Na, Mg, Mn, Mo, P, Pb, Rb, Sc, Se, Sr, V and Zn. The measurement of all these elements in tail hair can be taken as a good indicator of its potential as a non-invasive alternative tool to assess the essential and toxic elements in beef cattle<sup>1,2</sup>.

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### Acknowledgments

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## **The INAA facility in Delft: Fully automated, high capacity, high accuracy and opportunities for transnational access**

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**Keywords:** INAA, sample changers, automated, quality assurance, metrology, access

The INAA facility at the Reactor Institute of the Delft University of Technology is built on over 50 years of experience in automation of gamma-ray software for spectrum analysis and interpretation, automation of gamma-ray spectrometers by sample changers and integrating this all with quality assurance and quality (validity) control. The facility comprises 2 spectrometers with coaxial detectors and 3 spectrometers with well-type detectors, and a spectrometer with the fast rabbit system. The system allows for the simultaneous use of the various detectors by different projects which speeds up the turnaround time for each project; rather than having the measurements running sequential on one or two spectrometers.

The typical capacity is about 14 samples, 20 flux monitors and 2 quality control samples per spectrometer per day, resulting in ca. 100 samples/day if only 1 measurement is needed, and ca. 50 samples /day if 2 measurements (e.g., after 1 and 3 weeks decay time) are desired. The fast rabbit system is operated manually, and the number of samples to be analysed depends on the half-life dependent protocol but is typically also in the order of several tens per day.

Taking advantage of a research reactor with a reliable operating schedule of about 40 weeks/year at about 100 hours/week, the system provides a capacity of several thousands of samples for multi-element analysis a year.

The gamma-ray software can be run in the fully automated mode without operator intervention or, in and interactive mode, The software is largely similar to the IAEA - $k_0$  software but spectra can also be made available in other formats to be analysed by software such as GammaVision, Genie2000. These options facilitate irradiations and measurements at the facility and (remote) analysis by user-specific software.

The capacity of the INAA facility in Delft can thus be made available for projects from users worldwide, e.g. for other NAA facilities dealing with capacity limitations by lack of automation, limited reactor availability or technical difficulties. Sample preparation and spectrum analysis can done at the end-user's facility whereas the irradiations and measurements at contracted out to the Delft Facility at a nominal fee.

## Instrumental Neutron Activation Analysis of Chang'E-5 Lunar samples

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**Keywords:** lunar samples, neutron activation analysis, deep space exploration

The origin of the Moon is a core issue of lunar research. The data and samples returned by the lunar exploration missions have greatly improved the knowledge of human beings for the Earth-Moon system. However, some unsolved problems have been discovered and needed to be answered by future exploration tasks and scientific research. More than forty years after the Apollo and Luna missions, China's Chang'E-5 (CE-5) returned samples can further understand the Moon. Different China's research teams have revealed important information on the CE-5 lunar sample using advanced technologies for the basic properties, the formation age, and source characteristics of mare basalt and so on. In addition to precious lunar returned samples, lunar meteorites can provide important supplementary information on the composition and evolutionary history of the Moon. The non-destructive analysis of the elemental contents in the lunar soil sample is of great significance for understanding the lunar evolution and the development and utilization of lunar resources. Instrumental neutron activation analysis (INAA) technology can effectively solve the problem of non-destructive, high-precision measurement for the most elements in lunar sample, and provide a nuclear technology supporting for the China's deep-space exploration samples. In this paper, the multi-elemental composition of lunar samples (CE-5 samples and lunar meteorites) analysed by INAA and compare the data with the Apollo and Luna analysis. According to the basalt classification, the CE-5 lunar samples are classified as low-Ti, low-Al, and low-K mare basalts.

### Acknowledgments

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## Instrumentation for Activation Analysis

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### Calibration of a gamma ray Compton camera for radioactivity measurements

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**Keywords:** Compton imaging, radioactivity, gamma spectrometry

A second version of the dual-plane Compton imaging detector [1] previously developed for prompt gamma imaging [2] has been further tested and calibrated for quantitative radioactivity determination, specifically to assess radioactive debris from a reactor fuel element containing uranium isotopes and several long-lived fission products, including Cs-137. The detector consists of two pixelated planes of CZT crystals which record gamma ray events interacting within these volumes. In addition to the energy spectrum obtained by pulse height analysis, the time-stamped events from each detector pixel are sorted by location into 3 categories for post-processing: intra-plane, inter-plane, and coincidence. 2D and 3D imaging reconstruction software enables spatial localization of the select gamma ray peak(s) from the spectrum, thereby separating the gamma ray by specific isotopes. We used the camera to image a small piece of debris for demonstration purposes. Fig. 1 shows the spatial separation of the Cs-137 in the debris piece (A) from the Co-60 in the adjacent waste bin (B), along with their spectra (shifted vertically for clarity). A subsequent measurement was performed alongside a HPGe detector, which could determine the isotopes' absolute activities and be used as a reference for the camera. In addition, a known isotopic point source was used to calibrate the camera's energy detection efficiency in the same measurement geometry, correlating the image pixel intensity to the isotopic activity forming the basis for quantitative imaging. The results of these studies will aid prompt gamma imaging efforts when our neutron beam becomes available again.

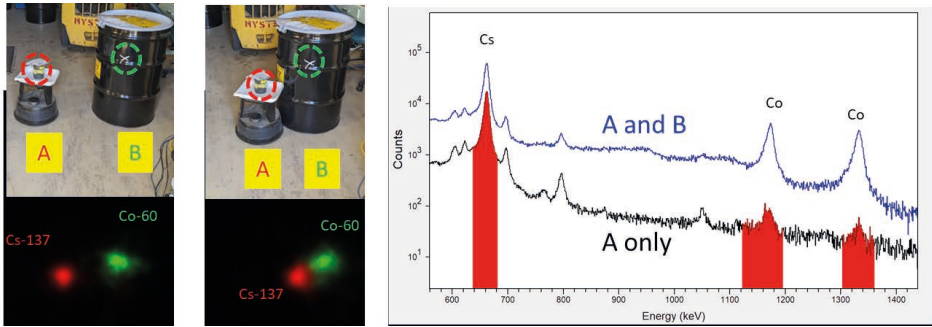


Fig. 1. Compton imaging of a debris piece (A) containing reactor fuel fission products including Cs-137, placed next to a waste bin (B) containing Co-60. The spectra are vertical shifted for display clarity.

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## **Advancements in instrumentation for activation analysis in nuclear research**

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Activation analysis is a powerful technique employed in nuclear research to determine the elemental composition of various materials. This study explores recent advancements in instrumentation applied to activation analysis, with a focus on enhancing precision, sensitivity, and efficiency in elemental analysis. The research conducted at the Federal Polytechnic Research Institute Ilaro aims to contribute to the optimization of activation analysis methodologies by incorporating state-of-the-art instrumentation.

The proposed research involves the development and utilization of novel detection systems, such as high-resolution gamma-ray spectrometers and advanced neutron sources. These innovations aim to overcome limitations associated with traditional activation analysis methods, providing researchers with improved capabilities for identifying and quantifying trace elements in diverse samples.

Additionally, this study investigates the integration of modern computational techniques, including artificial intelligence algorithms, for data analysis and interpretation. The combination of sophisticated instrumentation and advanced data processing methods is expected to yield more accurate and reliable results, thus enhancing the overall efficacy of activation analysis in nuclear research.

Furthermore, the research at the Federal Polytechnic Research Institute Ilaro explores the potential applications of activation analysis instrumentation in fields beyond traditional nuclear research, such as environmental monitoring, materials science, and industrial quality control. By expanding the scope of activation analysis, the study aims to demonstrate the versatility and broader societal impact of the developed instrumentation.

In summary, this research project at the Federal Polytechnic Research Institute Ilaro focuses on advancing the field of activation analysis through the incorporation of cutting-edge instrumentation and computational techniques. The outcomes of this study are anticipated to contribute significantly to the improvement of elemental analysis methodologies, fostering progress in nuclear research and its diverse applications.

## Specialty HPGe probe for neutron activation analysis

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**Keywords:** HPGe detector, field measurement, gamma-ray spectroscopy

This poster aims to introduce the Specialty HPGe Probes, which are HPGe detectors designed to withstand challenging measurement conditions. Designating an abstract concept rather than a product, Specialty HPGe Probes are new, innovative tools that are developed to suit specific end-users' constraints and needs, allowing to manufacture the ideal HPGe detector for their application. Some of these customization possibilities are of great interest in the field Instrumental Neutron Activation Analysis (INAA), Prompt Gamma-Ray Activation Analysis (PGAA), and Fast Neutron Activation Analysis (FNAA), as they allow to meet unavoidable constraints:

- Housing selection: dust-tight, sealed housing for use in contaminated environments (bringing decontamination capability); completely watertight housing for use in cooling pools, etc.
- Spatially constrained environments: adjustable HPGe crystal size and optimized choice of cooler; adapted shape (tubular shape...)
- Use in very highly radiative environments: use of proven, reliable technologies (MicroGe™, etc.); manufacture of customized shielding and collimators, etc.
- HPGe detector type selection: Small Anode Germanium Well (SAGeWell™) detectors; Standard Electrode Coaxial Germanium (SEGe™) detectors; Broad Energy Germanium (BEGe™) detectors, etc. depending on the application and on detection properties requirements.
- Industrial constraints: use of reliable components compatible with industrial constraints (heat, moisture, vibration, EMC, etc.).
- Possibility to anneal the HPGe crystal to recover an intrinsic energy resolution close to factory specifications.

Some examples of concrete, recent developments and application cases will be presented and discussed. In addition, some technical characterization results for these Specialty HPGe Probes will be presented, demonstrating their interest in the field of neutron activation analysis when HPGe spectroscopic performance are needed.

## k<sub>0</sub>-based activation analyses

### **Comparative study by k<sub>0</sub>-NAA method of two Algérien bentonites saturated and unsaturated with corrosion inhibitors**

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**Keywords:** bentonites, adsorption, treatment, k<sub>0</sub>-NAA method

Bentonite has been a stable natural geological material for millions of years. It has been chosen in the majority of highly radioactive waste storage studies to constitute the first barrier in contact with the waste package, which should last at least 10,000 years. The significant adsorption power of bentonite would block the passage of radionuclides to the biosphere. The low permeability makes it possible to delay the initiation of wet corrosion on container made of carbon steel. The corrosion products on the surface of container are able to modify the chemistry of the interstitial solution of the bentonite which affects the migration of the radionuclides. This leads to an alteration of the properties for which bentonite was chosen as a buffer for the storage of radioactive waste.

In aqueous solution, the carbon steel can protect itself against corrosion by the formation of a passive film of iron hydroxide which depends on the chemical nature of its environment. The influence of a chemical or physical factor can lead to an irreversible rupture of this passive film, and triggering of the dissolution of the steel in a uniform (generalized corrosion) or localized (pitting corrosion) manner. To inhibit these forms of aqueous corrosion many chemical inhibitors can be used, among them tungstates, which are inorganic and non-toxic oxyanions Boucherit et al.'s electrochemical analyses of 0.18% carbon steel have demonstrated a good inhibition efficiency of tungstate against localized corrosion. Notably, this effectiveness experiences a boost in the presence of iodates or other slight oxidants [1]. These oxyanions widely used in cooling circuits, they have an alkaline character which makes it possible to inhibit generalized corrosion, and the advantage of polymerizing at acidic pH to inhibit pitting corrosion.

The study of the estimation of the corrosion of carbon steel made by Zhang et al on 03 type of compacted bentonites showed that the corrosion rate is lower than those obtained in groundwater [2]. Studies of the electrochemical behaviour of carbon steel in contact with an Algerian bentonite, destabilizes the formation of the passive film on the surface of the steel due to its ability to adsorb iron ions released by the steel. Montmorillonite, which is the majority phase in bentonite, is responsible for this behaviour. Loading bentonite with a corrosion inhibitor such as tungstates successfully inhibits steel corrosion in the steel system surrounded by bentonite [3].

Given the established importance of loading bentonite with a corrosion inhibitor, the current study intends to measure the changes, including the chemical composition, of two Algerian bentonites from Maghnia (Mag) and Mostaganem (Mos) that have been treated with tungstates using the neutron activation k<sub>0</sub> analysis method.

The saturation ratios of Mag and Mos bentonites employing tungstates were determined to be 3.0E+04 and 2.9E+05, respectively. After rinsing, the retention rate of tungstates was 2.4% in Mag and 27.7% in Mos which is considered 12 times higher than Mag. This study helps to understand behaviour of Mag and Mos bentonites by examining the tungstate retention properties.

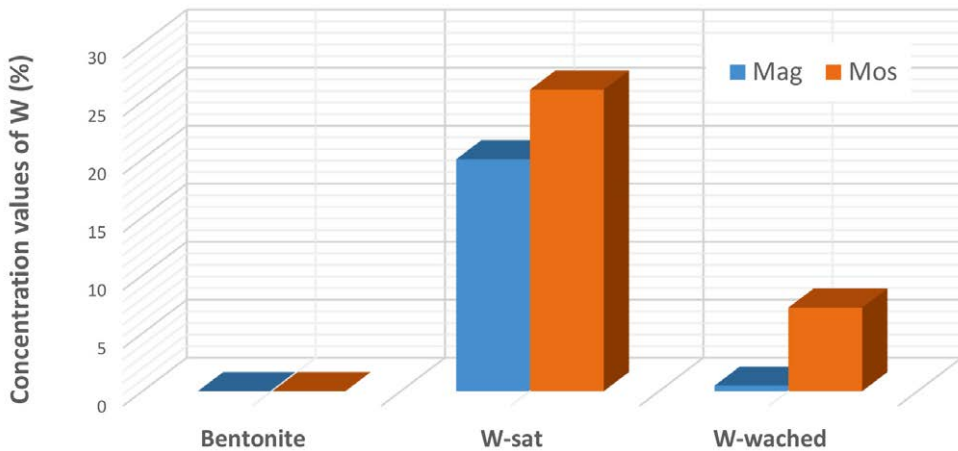


Fig 1: Comparison study of Mag and Mos bentonites treated with tungstates

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## Correction of uranium fission interferences for $k_0$ -NAA at the Dalat Research Reactor

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**Keywords:**  $k_0$ -NAA, uranium fission interference; correction factors; certified reference materials

In the  $k_0$ -based neutron activation analysis ( $k_0$ -NAA), when the sample contains high uranium, there is the problem of interference from not directly produced only by the fission of uranium, but from the product of the decay of fission products, or because of the problem of spectral interferences uranium fission products. These formed radionuclides are same as the applied ones in  $k_0$ -NAA. Therefore, correction for fission interference is necessary when using  $k_0$ -NAA. This study presents the experimental determination of the correction factor for radionuclides of interest at the Dalat Research Reactor (DRR), i.e., Ce-141, Ce-143, La-140, Ba-131 and Sm-153. The obtained correction factors were compared with the values reported in the literature. The determined factors were integrated into the “k0-DALAT” program for  $k_0$ -NAA which is used to determine the mass fraction of elements of interest in the certified reference material (NIST-SRM-1633b). Mass fraction of elements obtained by  $k_0$ -NAA with fission interference correction applied give results a better agreement than the uncorrected case in comparison with the certified values in the range of 15-28%.

## An approach to apply the neutron activation analysis, $k_0$ method, on irregularly shaped samples

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**Keywords:**  $k_0$ -NAA, large samples, archaeological sherds

Typically, in Neutron Activation Analysis (NAA), the sample to be investigated must be small (about 200 mg), crushed into powder and sealed in polyethylene ampoule for further processing. However, there are samples that are not possible to withdraw an aliquot or even be pulverized. That is the case of sherds in archaeological studies. Usually, the ceramic fragments have different geometries and it is often impossible to remove an aliquot, either because it is a unique finding or because the size does not allow the removal of an aliquot. In such situation, a study was developed to verify the feasibility of establishing a methodology for analysing samples compounded by small sherds applying the  $k_0$ -standardization method of neutron activation analysis ( $k_0$ -NAA).

In this work, the methodology of a larger sample was also applied since the samples were 2-3 cm high. Two types of samples of the same ceramic were prepared: powder and sherds. Thin iron wires were inserted in the middle of each sample at the same height as the sample and sealed in polyethylene ampoule. For such prepared samples, an Al-0.1%Au standard was fixed on the top and bottom of each ampoule according to the  $k_0$ -NAA procedure. The samples and Al-0.1%Au standards were irradiated for 4 hours in the carousel of the TRIGA Mark I IPR-R1 research reactor. After irradiation, iron wires were removed and cut into pieces of 1 cm. The samples, Al-0.1%Au standards and pieces of iron were submitted to gamma spectrometry. Based on the comparator factor of Au ( $F_{c,Au}$ ) calculated with neutron monitors, the mass fractions of each sample were calculated. Based on  $F_{c,Au}$  and iron wires, the correction factors were determined for sample sherds related to powder. The results of the study will be presented and discussed.

### Acknowledgments

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## Machine learning and artificial intelligence in AA

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### Curse of dimensionality in NAA data: an investigation on classification and clustering analysis

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**Keywords:** machine learning, supervised and unsupervised task, feature extraction, data analysis

The Pareto principle states that, generally, 80% of effects can be attributed to 20% of causes. This principle applies to a wide variety of disciplines and aids computationally demanding data mining processes by reducing the amount of information to be processed. It is reasonable to consider that such a principle also applies to chemometric analysis. Neutron activation analysis (NAA) is a nuclear analytical technique that allows the simultaneous determination of multiple elements with accurate measurements. Although this is a positive characteristic that enables the characterization of various matrices, in forensic analysis tasks, geographic traceability, or exploratory analyses, some elements exert greater influence on the studied phenomenon. It is important to emphasize that an excess of information can impair the performance of data mining techniques by introducing noise and redundancy. This phenomenon of data mining performance impairment associated with information overload is referred to as the 'curse of dimensionality'. Given this scenario, we sought to evaluate the effect that attribute selection in chemometric datasets could have on the performance of classification algorithms and clustering methods. The datasets were obtained by NAA of matrices of animal and vegetable origin and they contain different number of samples, previously known labels and mass fraction of different chemical elements. Attribute selection was performed based on statistical tests such as PERMANOVA and Kruskal-Wallis test. The chemical elements that showed the greatest differences among labels were gradually added to the analyses. The assessment of the attribution selection effect was performed using accuracy and F1 score metrics for the classification algorithms and internal and external metrics for the clustering methods. The evaluated clustering methods showed greater sensitivity to the number of chemical elements used in the analysis than classification algorithms. As a supervised task, classification algorithms can deal better with the presence of noise or redundant information in reconstructing groups of interest. Internal metrics indicated that the larger the number of elements considered, the less well-formed the groups were. This occurs due to the rapid increase in distances in Euclidean space as the number of attributes considered increases, making the points sparse. External metrics showed great variation as the chemical elements were added to the analysis, and different datasets presented different behaviors. The addition of elements that resulted in smaller differences among labels not always led to a reduction in

the metric value, but most of the time resulted in a noticeable change. Regarding classification, overall performance increased considerably with the use of the chemical elements that yielded the greatest differences. The response of algorithms to the addition of more elements varied between stagnation and oscillation of metrics, with no apparent pattern. The curse of dimensionality proved to be a relevant phenomenon in chemometric analyses, indicating that the chemical elements used should be carefully chosen.

## Empowering classification algorithms with augmented data: A challenge for traceability studies using neutron activation analysis

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**Keywords:** data simulation, synthetic data, exploratory analysis

Neutron activation analysis (NAA) is a nuclear analytical technique offering multi-element capabilities for a plethora of matrices. The precision and low limit of detection achieved for many elements enable the identification of unique chemical signatures that, coupled with robust statistical methods, can be used for traceability and authenticity studies. Advanced data mining techniques, such as supervised machine learning tasks, have been extensively employed in chemometric analysis, aiming to explore the potential use of multi-elemental profiles as a source of information for such purposes. However, the number of samples analyzed by NAA is typically limited to a few tens or hundreds of observations due to the high cost and complexity of the involved processes. The low number of samples may compromise the performance of classification algorithms as many of them rely on iterative functions that may converge to local optima under limited training conditions, in a process known as underfitting. This is because the amount of information available for the algorithm to detect patterns present in the samples during the training and testing stages is directly related to the dimensions of the dataset. Increasing the number of iterations is an inefficient solution since repeated training with the same data makes classifiers susceptible to overfitting, where the solution found perfectly fits the training data but is not replicable in practical tests. In the face of these challenges, it was investigated whether expanding the dataset derived from experimental analyses through data augmentation could help improve the performance of classification algorithms. For this purpose, a dataset containing the mass fractions of nine chemical elements determined by NAA in beef from different geographical origins was used. The augmentation process aimed to create new artificial samples for model training while preserving the characteristics of the experimental samples such as mean, distribution, and covariances, later used in the model evaluation. The results were compared with the performance of classifiers trained with the experimental samples themselves using cross-validation. Superior performance of the algorithms was observed when trained with the artificial samples. The algorithms were able to capture the underlying patterns present in the experimental samples by training with the same patterns replicated in the artificial samples, without incurring in overfitting as the experimental samples were never actually seen, and neither incurring in underfitting since the training process was carried out with a satisfactory number of iterations and samples. Thus, the problem of training and testing with the same observations is overcome, yielding more cohesive results. It can be concluded that data augmentation has the potential to solve the limitations associated to small datasets in classification tasks of samples analyzed by NAA.

## Neutron activation analysis: extracting further information from gamma-ray spectra for machine learning approaches

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**Keywords:** support vector machine, cluster analysis, cat food

The elemental profile of samples can be used as markers for traceability and forensic analysis. The conventional approach consists of applying chemometric and data mining methods to the mass fraction values of chemical elements. In the context of neutron activation analysis (NAA), the photopeaks of detected radionuclides, while ignoring other information present in the gamma spectrum, such as the Compton effect, pair production and non-quantifiable photopeaks. In this study, it is proposed to use the counts of all channels in the gamma spectrum as a source of information for classification and grouping tasks. As a case study, spectra obtained from samples of dry cat food from 3 different market categories were used Standard, Premium and Super Premium<sup>1</sup>. The results using the complete spectra were compared with those obtained for the conventional approach, that is, only the photopeaks of the determined elements. The samples were irradiated in the IEA-R1 nuclear research reactor of the Nuclear Energy Research Institute of the Brazilian Nuclear Energy Commission (IPEN/CNEN), São Paulo, SP, Brazil, with thermal neutrons flux of  $1 \times 10^{13}$  cm for 4 h. High-resolution gamma spectrometry was performed on a hyperpure germanium coaxial detector, model GEM50P4 manufactured by ORTEC, with a relative efficiency of 50% for the photopeak 1332 keV of <sup>60</sup>Co. The induced activity was measured with decay time of 7 days, 30 min count and the spectra were adjusted for the sample mass and the incident thermal neutron flux. Differences were observed between the spectra of each market category in relation to the number of photopeak counts and continuous background of the low energy region. The Support Vector Machine algorithm, used to classify dry cat food samples, achieved an accuracy of 62% with specific photopeaks, and 81% when using counts from the 8192 channels of the gamma spectrum. In the grouping task, an improvement in homogeneity and completeness indices was observed when using the complete gamma spectrum. The use of the gamma spectrum as a source of elemental profile information resulted in advancements in the performance of classification and grouping tasks for the set of dry cat foods, suggesting that the proposed approach has advantages over the conventional one under appropriate conditions.

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## A novel approach for elemental identification of sealed cargo based on fast neutron activation analysis and artificial neural network

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**Keywords:** ENTRANCE project, Rapidly Relocatable Tagged Neutron Inspection System (RRTNIS), gamma-ray spectroscopy, artificial neural network, machine learning

Securing global trade requires efficient screening of containers for threat materials. This work demonstrates a novel approach combining fast neutron activation analysis and artificial neural network (ANN) to identify elemental composition of sealed cargo, in particular elements carbon, oxygen, and nitrogen, which are the main components of explosives.

The fast neutron induced prompt gamma-ray spectra were collected with the Rapidly Relocatable Tagged Neutron Inspection System (RRTNIS) as a part of the EU Horizon 2020 project ENTRANCE (Efficient Risk-based Inspection of freight Crossing borders without disrupting business). RRTNIS contains a DT-associated alpha particle neutron generator and twenty 5"×5"×10" NaI(Tl) gamma-ray detectors. The coincidence timing between the position-sensitive alpha detector and gamma detectors makes it possible to record gamma-rays originating from different depths of the container. The prompt gamma-ray spectra of twenty key elements (including carbon, oxygen, and nitrogen) and background obtained with the RRTNIS were used to generate 100,000 gamma-ray spectra of different unknown materials for training and testing a feed-forward ANN. In addition to the generated spectra, measured spectra of two simulants corresponding to the explosives Hexogen (RDX) and TATP, were used to test and validate the trained network. The confusion matrix, derived from the comparison between the predicted outputs and the actual targets of the neural network, demonstrates that the network achieves an accuracy of over 96% in identifying the predominant element in each spectrum. This demonstrates the robustness and efficiency of the trained network in accurately identifying the predominant elements in the spectra. Furthermore, the count contribution of carbon, nitrogen, and oxygen in the prompt gamma-ray spectra of the two simulants versus depth indicates that the explosives are correctly identified at their respective positions within the container.

This study thus shows that RRTNIS in combination with ANN is a potential promising solution for the inspection of sealed containers, allowing precise identification of elements and detection of potential threats without the need to open the containers. This enables quick security checks of suspicious containers while minimizing disruption to the regular flow of cargo.

### Acknowledgments

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## Prompt Gamma Activation Analysis

### Comparison of PGNAA simulation techniques for concrete analysis

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**Keywords:** PGNAA, concrete, PHITS and MCNPX

Prompt Gamma Neutron Activation Analysis (PGNAA) is an important technique for non-destructive elemental analysis of materials like concrete. Computer simulation can complement experimental PGNAA systems. This study compares the use of two Monte Carlo codes, MCNPX and PHITS, for simulating PGNAA on a concrete sample. The codes were modeled identically based on an experimental PGNAA system. Results showed that both codes identified the major elemental components of concrete, including Si, Ca, Cl, and Na. MCNPX identified more peaks overall compared to PHITS. Both codes provide reasonably accurate modeling of PGNAA for concrete analysis, with MCNPX offering more detailed spectral results. Further studies could optimize the codes for improved statistics.

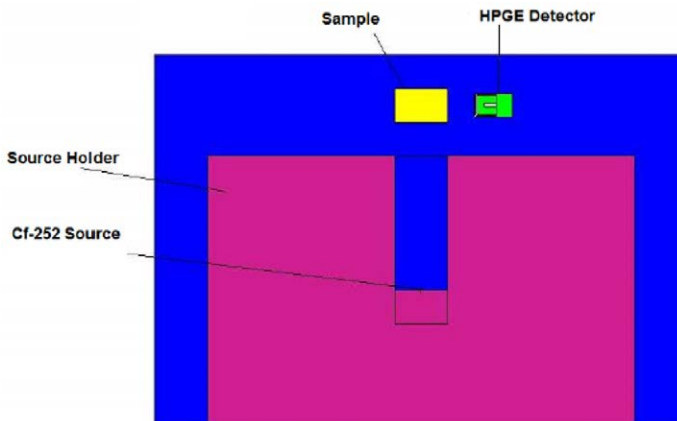


Figure 1, Basic schematic diagram of PGNAA experimental system from MCNP geometry code

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## Prompt vs delayed gamma rays for the detection of vanadium in solid samples

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**Keywords:** TNC, PGNA, neutron generators, solid samples

Determining the presence of vanadium in the environment has attracted a lot of attention in the past few decades due to the expanding roles that various forms of vanadium are playing in the sciences. This work aims to measure the elemental contents in soil samples using the non-destructive Prompt gamma-ray activation analysis technique. The samples are exposed to a neutron beam, which causes elemental nuclei to thermally absorb neutrons and, upon deexcitation, emit prompt or delayed gamma rays. High-resolution detectors are then used to measure these gamma rays to recognize the neutron-capturing elements, while the concentrations of these elements are extracted from the intensities of their corresponding gamma peaks. The Genie 16 portable generator producing 2.5 MeV neutrons via a deuterium-deuterium (DD) reaction will be used to activate samples that have several concentrations of vanadium content. The generator is operated at 70 keV and its dc-current is set at 50  $\mu\text{A}$  to get a maximum intensity of  $4.7 \times 10^7$  n/s. The experiment is designed so that we can identify the prompt gamma rays during the activation phase and the delayed gammas after turning off the neutron source. To minimize the Compton background and fast neutrons from coming into direct contact with the detector, lead and polyethylene are also placed around the setup. The outcomes of this work will be presented.

### Acknowledgments

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## Identification of multielement using prompt gamma-ray technique: Comparison study

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**Keywords:** prompt gamma, PGNAA, isotopic neutron, elemental analysis

In this investigation, the fundamental of prompt gamma neutron activation analysis principle has been applied as a methodology to determine the element composition within examined sample. The prompt gamma-ray system consists of collimated isotopic neutron source, namely Cf-252, conjoined with High Purity Germanium (HPGe) detector and a Multichannel Analysis (MCA) software. Concrete samples with dimensions of 10x10x10 cm<sup>3</sup> and 15x15x15 cm<sup>3</sup> are subject to analysis. When neutrons enter and interact with elements in the concrete, the neutron capture reaction will occur and generating characteristic prompt gamma ray specific to each element. The result of this study demonstrates the identification of major element in the concrete was determined such as Si, Mg, Ca, Al, Fe and H, alongside other elements like Cl by analysis the gamma ray lines respectively. Comparative assessment of the results obtained were compared with computer simulation, NAA and XRF for a reference and validation purpose. The potential and the capability of neutron induced prompt gamma as a qualitative tool for multi elemental analysis to identify the elements present in the concrete sample discussed.

## QA/QC, Presentation of analytical results, Laboratory Information Management System

### QA/QC of Instrumental Neutron Activation Analysis Methods and applications towards preparation of In-house Reference Materials

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**Keywords:** QA/QC, INAA, CRM, radioanalytical techniques, In-house Reference Materials

Quality Assurance (QA) and Quality Control (QC) of any analytical method are very essential for authenticity of results and data validation. Thus, for data reporting as well as certification of a developed/procured material, it is necessary to evaluate the QA parameters like (i) Quality Control (QC) for method validation in order to evaluate accuracy, (ii) Cross validation of results using another reference technique(s), (iii) Reproducibility or precision of the results, (iv) total uncertainty measurement from individual uncertainty parameters and (v) Evaluation of detection limit from blank measurement. When the materials of interest are complex in nature and difficult for chemical dissolution, non-destructive analysis of solid samples or as received samples are preferred compared to wet-chemical atomic or mass spectrometric methods. In this respect, Nuclear and Radioanalytical methods like Instrumental Neutron Activation Analysis (NAA), Prompt Gamma-ray NAA (PGNAA), Particle Induced Gamma-ray/X-ray Emission (PIGE/PIXE) and Energy Dispersive XRF (ED-XRF) have several advantages for quantification of low to high Z elements starting from H to U in diverse matrices. INAA using relative and  $k_0$ -based conventional and internal monostandard NAA have been extensively utilized for major to trace elemental concentration determination in diverse matrices in fields like materials sciences, geological, biological and environmental samples and nuclear reactor technology. In each case, method has been validated using a suitable RM/CRM/SRM from agencies like NIST, IAEA, USGS and BCR and also QA parameters have been evaluated. As a cross validation method, ED-XRF and in situ current normalized PIXE and PIGE methods have been utilized. It was observed that  $k_0$ -based NAA/IM-NAA methods, though do not need multielemental standards, associated with higher uncertainties due to neutron flux parameters ( $\phi$  and  $\alpha$ ),  $k_0$ -factors and relative/absolute efficiency.

As QC is a must while reporting analytical results and its needs certified/standard reference materials (CRM/SRM) for method validation. CRMs/SRMs, high cost materials, should not be used as standards for routine analysis of many samples. In many cases, suitable or exact matrix matching standard is not available which in turn affects the accuracy of the method. In view of these, in-house reference material or standard is essential for an analytical lab for routine chemical analysis of samples. In the present work, INAA method using high flux reactor neutrons have been used to prepare in-house RMs of (i) sodalime glass using a large size original car wind-shield glass and (ii) sub-bituminous coal. Other methods namely

IBA (PIXE/PIGE) and ED-XRF have been used to cross validate the results as well as to get more information on number of elements using complementary techniques. External (in air) PIGE method was helpful low Z elements, whereas PIXE and ED-XRF were used for medium and high Z elements. About 20 elements in sodalime glass and 15 elements in coal were determined to be used as in-house reference materials. Detailed methodologies as well as QA/QC of INAA methods will be presented.

**Acknowledgments**

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## Proposal of a Laboratory Information Management System (LIMS) for the neutron activation analysis technique applied at Peruvian Institute of Nuclear Energy (IPEN)

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**Keywords:** LIMS, data management, GUI, neutron activation analysis, CSharp

In order to promote an efficient management of laboratory data, improving the productivity and integrity of the information produced in the analysis and research services, a LIMS is proposed, called Nuclear Center – INAA, based on the creation of an interactive platform that allows optimizing the management of data generated in the analysis by neutron activation – ksubzero method. The starting point was to identify the needs of the analysts, to continue with the conceptual design process, which allowed defining the appropriate architecture, which consists of five phases, from the reception and registration of a sample to the release of the results report. Key aspects considered were the user interface, the navigation logic and the integration of the phases generating databases. Safety was a priority, incorporating system access controls, user levels and session logging. These advanced features were implemented using the CSharp (C#) programming language which offers an extensive library of code packages. The user interface was developed using Windows Forms, providing a faster and more efficient environment, which are crucial elements for a laboratory management system.

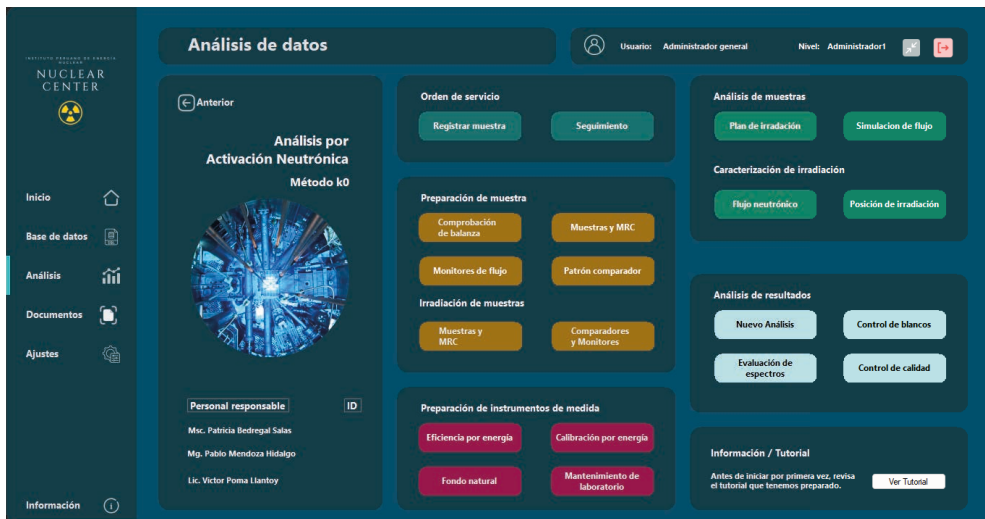


Figure Graphical user interface (GUI)

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**Related techniques (Charged-Particle AA, Photon-induced AA, ...)**

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**Flux correction for photon activation analysis**

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**Keywords:** photon activation analysis, flux correction, secondary neutron

**Introduction**

We have determined elemental concentrations in various samples, mainly geochemical and cosmochemical samples, by photon activation analysis (PAA) using bremsstrahlung produced by an electron linear accelerator. In our case samples are enclosed in a quartz tube in series, thus intensity of bremsstrahlung at each sample position decreases downstream of the beam. Therefore, photon flux correction is essential to determine accurately. In the early days, gold foils were used as photon flux monitor, using  $^{197}\text{Au}(g, n)^{196}\text{Au}$  reaction. And NaCl monitors were also used for secondary neutron flux correction because  $^{23}\text{Na}(n, g)^{24}\text{Na}$  reaction interferes determination of Mg using  $^{25}\text{Mg}(g, p)^{24}\text{Na}$ . The evaluation of their corrections was reported previously [1]. Recently, Ni foils have been used as photon flux monitor as well as a comparator of single comparator method. On the other hand, the secondary neutron flux have been measured using NaCl as before, but at present secondary neutron flux fluctuation is different from that of the past. Therefore, we evaluated the correction of photon flux and secondary neutron flux again.

**Experimental**

Some various kinds of metallic foils, powder reagents, and geochemical standards as well as Ni foils were enclosed in quartz tubes and were irradiated with bremsstrahlung at the maximum energy ( $E_0$ ) of 22 MeV or 33 MeV for 30 minutes or 4 hours in the electron linear accelerator at the Research Center for Electron Photon Science (ELPH), Tohoku University. After irradiation, these samples were measured with a Ge detector. The sample holder box and water-cooling system for irradiation at ELPH are renewed and are different from ones in the report in 2011 [1]. Open-type sample box with water cooling was replaced with closed-type one.

**Results and Discussion**

For accurate determination, specific radioactivity of  $^{57}\text{Ni}$  in Ni monitors and that of radionuclide in samples used for elemental determination must decrease at the same rate from upstream to downstream of the beam.  $^{57}\text{Ni}$  in nickel foils and  $^{57}\text{Ni}$  in the other samples were found to decrease at the same rate. The radionuclides produced in the (g, n) reaction as well as  $^{57}\text{Ni}$  showed similar decreasing rates with some variations. It is unclear that these variations are systematic or experimental chance error at the present. At least, no clear correlation with Q values has been found. When standard sample for comparison method is placed in a

quartz tube at the most upstream, the determination values differ by up to  $\pm 20\%$  at the most downstream. If the standard is placed in the middle of all samples, the overall uncertainty can be reduced.

$^{24}\text{Na}$  induced by  $^{23}\text{Na}(n, g)$  reaction was found to increase downstream. This observation is the opposite of the trend reported previously [2]. The other  $(n, g)$  products showed same trends as  $^{24}\text{Na}$ . However, their increasing rates varied greatly for each radionuclide, which is not like  $(g, n)$  products. The differences among nuclides may reflect  $(n, g)$  excitation function and the distribution of neutron energy, but no systematics has been found so far.

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## Sample preparation

### Testing 3D printing raw materials as sample holders in Neutron Activation Analysis

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**Keywords:** PEI, PEEK, high-temperature resin, reactor irradiation, NAA

Neutron Activation Analytical (NAA) community is seeking for radiation resistant, cost effective, easy to prepare sample holder. The well proven, commonly used high purity quartz glass is expensive and requires expertise in glasswork. 3D printing has invaded the industry and become widespread in the production of various devices and components. Additive manufacturing could offer an obvious solution in the field of NAA, if providing radiation-resistant raw materials, which are easy to decontaminate, because they are supposed to be acid-proof and heat-resistant. There are many types of resins, and manufacturers provide us with all kinds of information about their physical capabilities, while missing quite a few essential chemical data and radiochemical characteristics.

Experts of the 3D printing companies proposed PEEK, PEKK, PEI, and other ultra-high temperature plastics for testing, as these materials have the properties of the highest heat resistance, as have high strength and stability at high temperatures, and very low coefficient of thermal expansion. Next to that, they also have good chemical resistance and flame retardance. Six different raw materials were irradiated at BNC, in the Budapest Research Reactor and NAA were applied after the short and the long irradiations. Activation properties were measured, colour changes were visualized, and elemental concentrations causing higher activities were also filtered, to find applicable material for NAA.

The purest material is Stratasys ULTEM 1010 PEI resin, in which all detectable elements are below mg/kg level, however it changes colour, and gets a bit darker after long irradiation. It is the most suitable as a sample holder for NAA. Stratasys Antero PEKK samples were highly active after irradiation, as well as Antero 840 CN 03, which is less suitable for irradiation due to its relatively high Cl, Fe, and Co content. Antero 800 NA is more suitable candidate but rather for long irradiation. Two other HT (250 and 300) samples changed colour due to irradiation effects, but HT 250 would be a reasonable sample holder for short (max 10 minutes) irradiations! However, EvonikM40 shows no visible changes due to irradiation, it gets highly active, as it has high Na content and Mg, so it found to be inadequate.

Further testing of these materials is essential to be widely used in various radiation environments, nuclear research, and applications in nuclear industry.

## Spectrometry (gamma-ray, charged particle)

### Mössbauer spectroscopy technique: How to characterize Fe oxides in archaeology

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**Keywords:** Mössbauer spectroscopy, cultural heritage, iron oxides, nuclear gamma resonance

Mössbauer spectroscopy is nuclear gamma resonance analytical technique used to characterize Fe oxides in cultural heritage. The Mössbauer technique provides valuable insight into the oxidation state, and coordination environment of Fe atom in matrix. And it makes a significant contribution to the study of potential information, such as types of Fe oxides,  $Fe^{3+}/Fe_{Tot}$  ratio, firing environment and temperature.

The excavated iron relics presumed to be from the Joseon Dynasty (South Korea) and specimens of black glazed ware were subjected to spectroscopic studies. Samples were collected with a little damage to the artefact as possible. The iron relics was an iron pot specimen, and many traces of rust were observed on the surface, and red-brown colour corrosion was confirmed. From the Mössbauer spectrum of the iron relics, the hematite and magnetite, which are caused by corrosion of iron, were analysed the most, and goethite and lepidocrocite were also identified. Mössbauer experiment was measured at low temperature (4.2 K) to distinguish the Fe phases of the obtained doublet spectrum. In addition, to infer the production process of black glazed ware, the ceramics of black ware were produced and its characteristics were investigated. The black glazed ware varies in colour from black to brown depending on the iron oxide content of the glaze layer and firing conditions. The ceramics were produced according to component ratio (Fe oxide, oak ash, calcite, and feldspar), and firing environment and temperature. Based on the obtained ceramic glaze data, we have investigated how conditions (firing environment, temperature and manufacturing component) affect colour expression.

Mössbauer spectroscopy is a powerful technique for studying the chemical and physical states of Fe and many other elements in solids, and its archaeological applications have mainly been applied to clay-based ceramics and may continue to have valuable potential applications in the future.

**Acknowledgments**

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## Physics-informed neural network as an unfolding approach to gamma spectroscopy

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**Keywords:** unfolding method, physics-informed neural network, gamma spectroscopy, NaI(Tl) scintillation detector

Radioactive isotopes inevitably go through several decay processes, along with emitting a gamma ray with specific energy to each isotope. This characteristic allows for the identification and quantification of isotopes by measuring energy-resolved counting data, a process known as gamma spectroscopy. Thallium-doped sodium iodide (NaI(Tl)) scintillation detectors are known for their portability and high detection efficiency and have been widely used as in-situ gamma spectrometers. However, their main drawback is poor energy resolution, which makes it challenging to distinguish between overlapping peaks with similar energies or to detect weak peaks. To address this issue, various unfolding methods have been introduced to decompose Gaussian peaks in the measured spectrum into delta functions and infer their intensities. Here, we present a physics-informed neural network as an unfolding method for converting a Gaussian-broadened spectrum into a high energy-resolved spectrum. By incorporating the physical principles of gamma spectrum measurement into the neural network's parameter optimization process, this approach does not require the preparation of large numbers of training datasets. To verify its performance, we simulated some spectra contributed by gamma rays with two or more energies and different relative intensities, and measured experimental spectra with different combinations of four radioisotopes. For both the simulated and measured spectra, the proposed method was proven to accurately identify the energies of gamma rays and determine their relative intensities with acceptable accuracy.

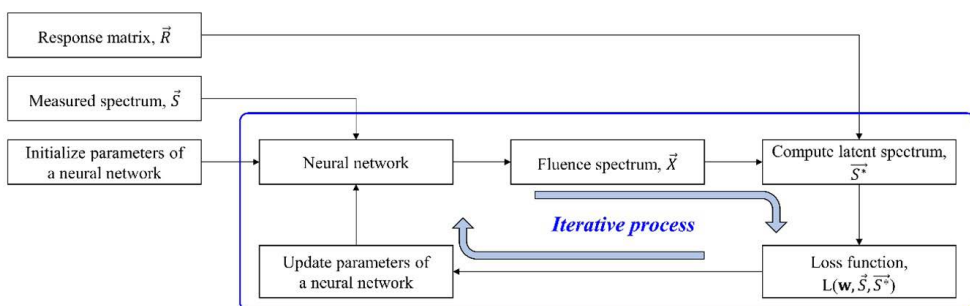


Fig. 1. Scheme of the physics-informed neural network-based unfolding method

**Acknowledgments**

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## Spectrum evaluation and analysis softwares

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### Optimization of baseline removal and peak area integration in gamma spectrometry

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**Keywords:** NAA, reference spectra, peak integration method

Gamma spectrometry softwares offer features for peak area calculation and baseline removal. Some specialized k0-NAA software packages have built-in tools for performing these procedures. In this process, a source of analytical uncertainty is included in the neutron activation analysis (NAA), associated with the integration of the emission peak. This bias is caused by the peak area integration method, which is relevant in peaks with low counts or complex background radiation. The baseline in the gamma spectrum represents the level of background radiation, that is, detected radiation not directly related to the analytes under investigation. Incomplete or inadequate baseline removal and integration method directly affect the calculated peak area. At the Radioisotopes Laboratory, CENA/USP, the Quantu package<sup>1</sup>, composed by Quantu INAA and Quantu MCA, developed in house, is used to perform the analysis of gamma spectra and calculations related to the method  $k_0$ , since 2003. There is currently a new software package for k0-NAA being developed to update the Quantu package, which uses machine learning for process automation and optimization. This work aims to evaluate baseline removal and peak integration methods for implementation in the new software. Consolidated methods in the literature were used to remove the baseline, such as polynomial fit, asymmetric least squares (ALS), adaptive iterative reweight penalized least squares (airPLS) and morphological weighted penalized least squares (MPLS), and machine learning algorithms such as support vector machine (SVM) and regression tree (DT). To integrate the peak, the integration method by total peak area (TPA), Wasson-Sterlinski and Gaussian adjustment were used. The tests were carried out with reference spectra from the International Atomic Energy Agency (IAEA)<sup>2</sup>, searching for close or overlapping peaks. The results indicated variations in the final area calculated by the different baseline removal methods, especially in the continuous region of the spectrum caused by the Compton effect, indicating that none is ideal for all peaks. The integration methods showed greater variations for low energy peaks and lower number of counts. The Wasson-Sterlinski and Gaussian adjustment methods proved to be better options for peaks with interference, in which TPA was not effective. Therefore, an optimal combination of baseline removal and peak integration methods was not found for all energies, suggesting that the software under development should implement different methods for use in specific situations.

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## User facilities

### Neutron Activation Analysis at MLZ

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**Keywords:** INAA, user facility

The FRM II research neutron source provides outstanding irradiation possibilities. For NAA applications, a maximum thermal neutron flux of in the order of  $10^{14}\text{cm}^{-2}\text{s}^{-1}$  can be used. Most positions are well thermalized with  $f$ -values in the range  $10^3$ - $10^4$ . A rabbit system connects the laboratory with the irradiation facilities. Three counting stations with HPGe detectors and digital spectrometers are available for the measurement of the activation products. The analysis is based on the standardless  $k_0$  approach. Beam time at the NAA instrument is accessible for scientific and industrial users. The GHOST interface streamlines the application process for NAA and all other MLZ instruments. We will present the current status of the instrument, discuss future developments, and provide an overview of the wide range of applications.