Chapter 1

Overview of Neutron Activation Analysis

Lylia Alghem Hamidatou

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Abstract

This chapter provides a comprehensive overview of physical principles, procedures, proprieties, and some scientific achievements of neutron activation analysis. The most scientific events organized by the International Scientific Committees ICAA and k_0-ISC are also reported.

Keywords: neutron activation analysis, research reactor, gamma spectrometry systems, medicine and geology applications, advanced technologies, innovation

1. Principles

Neutron activation analysis is a physical technique that is based on nuclear reactions. The sample becomes radioactive when neutrons react with the nuclei of the elements' atoms. Radionuclides are formed and subsequently decay by emitting gamma rays that are unique in half-life and energy. Gamma-ray intensity is proportional to the element content in the sample. Instrumental neutron activation analysis (INAA) and k_0-INAA are the most sensitive analytical techniques used for the quantitative multielement analysis of major, minor, and trace elements in samples from almost every conceivable field of scientific or technical interest [1–9].

2. Procedures

The elements in a sample to be analyzed are made radioactive by irradiation with neutrons, and the induced radioactive samples can be identified and measured. The amount of a given neutron activation product that formed during neutron irradiation is proportional to the amount of its parent isotope and thus the concentration of the corresponding element [2].

3. Properties

The analytical techniques based on neutron activation are classified according to energy of incoming neutrons, elastic and inelastic neutron scattering, and neutron capture reactions as well as its combination such as thermal NAA (TNAA), epithermal NAA (ENAA), prompt gamma NAA (PGNAA), fast NAA (FNAA), and 14-MeV INAA a form of FNAA based on reactions with 14-MeV neutrons that are produced by neutron generators. These techniques have big application potential since they could provide data about large number of elements simultaneously. **Figure 1** presents more than 65 elements that may be analyzed using neutron activation analysis method. For most elements, NAA is an extremely sensitive method for analysis. The potential benefits of NAA include the multielement capability and the fact that non-destructive analyses can be performed.

4. Organization and scientific achievements

4.1. ICAA, k_0-ISC, and scientific events

The objectives of the International Committee on Activation analysis of ICAA are to improve and promote the application of nuclear methods of chemical analysis. The International Committee on Activation Analysis (ICAA) is organized exclusively for scientific and educational purposes, and specifically to coordinate the series of international conferences entitled Modern Trends in Activation Analysis (MTAA). The official ICAA website (http://www.icaa-mtaa.org) provides information on their activities, membership, etc. [10].

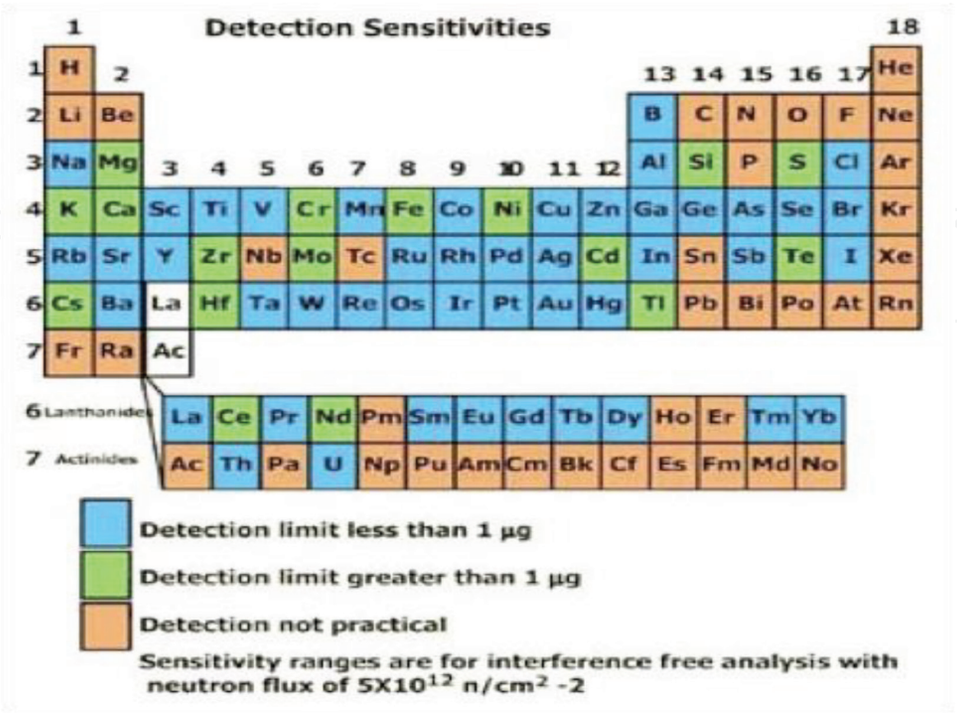

Figure 1. Detection sensitivities.

The k_0-International Scientific Committee was originally mainly involved in the organization of the k_0-Workshops and the accepting and reviewing of the papers presented there. The mandate of the k_0-ISC is to promote the development and application of the k_0-method of neutron activation analysis, k_0-NAA. Examples of the k_0-ISC's promotion of the development and application of the k_0 method include the coordination of the k_0-Workshops, maintenance and improvement of the recommended k_0-Nuclear Database, through the k_0-Nuclear Data Subcommittee, and the maintenance of the k_0 website to give information and to allow k_0-users to contact members of the k_0-ISC.

George de Hevesy (1885–1966) received the Nobel Prize for Chemistry in 1943 for his work on the use of isotopes as tracers in the study of chemical processes. The Hevesy Medal as illustrated in **Figure 2** is the premier international award of excellence honoring outstanding achievements in radioanalytical and nuclear chemistry as illustrated. **Table 1** presents the list of the laureates of George de Hevesy award during the period 1968–2018 [10].

4.2. International k_0-Users Workshops

The development of k0-NAA method is one of the most remarkable advances in the history of neutron activation analysis (**Table 2**). The k_0-NAA method includes comprehensive and accurate models of the neutron activation, radionuclide decay, and gamma-ray detection processes. During the period 1970–1980, Prof F. De Corte (University of Gent, Belgium) and Dr. A. Simonits (KFKI-AEKI, Budapest, Hungary) and many co-workers developed the concepts and procedures of the k_0-NAA method [4, 11–18]. Currently, this method is an inactive use in numerous laboratories all over the world [19–29]. The official k_0-ISC website (http://www.kayzero.com/$k_0$naa/$k_0$naaorg/k_0-ISC.html) contains relevant information on the k_0-method and all associated events and database [24].

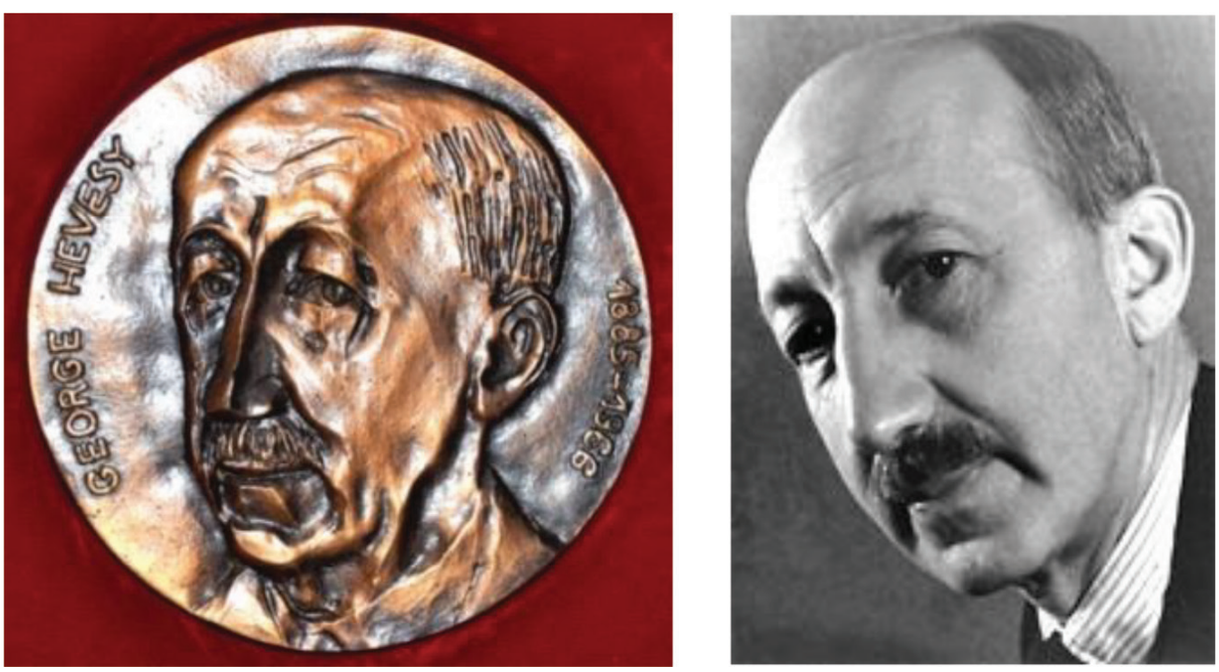

Figure 2. *George de Hevesy* (1885–1966) who received the Nobel Prize for Chemistry in 1943.

Year	Laureates	Year	Laureates
1968	W. Weine Meinke	2003	Jeroen J.M. De Goeij
1969	Albert A. Smales	2004	Attila Vértes
1970	Ivan Pavlovich Alimarin	2005	Zhifang Chai, Gregory Choppin, and Nicholas M. Spyrou
1972	Philippe Albert and Julian Hoste	2006	Jan Kucera
1975	Tibor Braun and Juraj Tölgyessy	2007	Robert R. Greenberg
1976	Francesco Girardi	2008	Syed M. Qaim
1977	Saadia Amiel Richard E. Wainerdi	2009	Richard M. Lindstrom
1978	Robert E. Jervis	2010	Darleane C. Hoffman
1979	Vincent P. Guinn	2011	Peter Bode (TU Delft page)
1981	William S. Lyon Max Peisach	2012	Boris F. Myasoedov
1983	Edward V. Sayre and Garman Harbottle	2013	Rajmund S. Dybczyński
1984	Georges Amsel	2014	Heino Nitsche
1985	Nobuo Suzuki	2015	Kattesh V. Katti and Susanta Lahiri
1986	Emile A. Schweikert	2016	Tomoko M. Nakanishi
2000	Frans De Corte	2017	Pavel P. Povinec
2001	Amares Chatt and Eiliv Steinnes	2018	Rolf Zeisler
2002	Enrico Sabbioni		

Table 1. List of the laureates of George de Hevesy award 1968–2018 [10].

4.3. Nuclear data

The authors F. De Corte and A. Simonits published in 2003, the recommended nuclear data for use in the k_0-standardization of neutron activation analysis [3]. In 2010, De Corte described in his paper "Towards an international authoritative system for coordination and management of a unique recommended k_0-NAA database" the constitution of the nuclear data library [25]. As indicated in the paper, "the 2012 recommended k_0 database", R. Javimovic et al. established the k_0 database in the form of an accessible excel file freely downloadable at http://www.kayzero.com/k0naa/News/News.html [26].

4.4. Neutron activation systems

Database from the IAEA's Research Reactor Database (RRDB) provides information with respect to the status of the world's research reactors. More than half of the 241 operational RRs worldwide performing neutron activation analysis distributed over 59 member states [31]. The highest roles of NAA have been identified as the most suitable opportunity for research, education and training, and commercialization of RR services. For that, NAA groups focused their efforts on the development and modernization of neutron activation analysis process including irradiation devices, gamma-ray spectrometers, and data analyzing instruments [31–35].

Title	Date	Organizer	References
MTAA-01	14–15 December 1961	Texas A&M College	
MTAA-02	18–21 April 1965	Texas A&M College	
MTAA-03	06–10 June 1968	National Bureau of Standards	NBS Spec Pub 312, June 1969, 2 vols
MTAA-04	01–05 October 1972	CNRS, Saclay, France	JRC v15–16, 1973 (also 17–19)
MTAA-05	13–17 September 1976	Institute of Radiochemistry, Technical University Munich, Germany	J. Radioanal. Chem., v. 37–39
MTAA-06	12 December 1981	University of Toronto, Canada	J. Radioanal. Chem., v. 69–72
MTAA-07	23–27 June 1986	Risø National Laboratory, Copenhagen, Denmark	J. Radioanal. Chem., v. 112–114
MTAA-08	30 December 1991	Technical University, Vienna, Austria	J. Radioanal. Chem., v. 167–169
MTAA-09	24–28 September 1995	Seoul, Korea	J. Radioanal. Nucl. Chem., v. 215–217 (1997)
MTAA-10	18–22 April 1999	National Institute of Standards and Technology (NIST)	J. Radioanal. Nucl. Chem., v. 244–245 (2000)
MTAA-11	20–24 June 2004	University of Surrey, Guildford, UK	J. Radioanal. Nucl. Chem., v. 271, No. 1–4 (2007)
MTAA-12	15–20 September 2007	Tokyo Metropolitan University, Hachioji, Tokyo, Japan	J. Radioanal. Nucl. Chem., v. 278, Issue 3, December 2008
MTAA-13	13–18 March 2011	Center for Chemical Characterization and Analysis Texas ASM University, College Station, TX, USA	J. Radioanal. Nucl. Chem., v. 291, Issue 2, February 2012
MTAA-14 and NAMLS-11	23–28 August 2015	Delft University of Technology, Delft, The Netherlands	J. Radioanal. Nucl. Chem., v. 309, Issue 1, July 2016

Table 2. List of the past international conferences on modern trends in activation analysis, MTAA (1961–2015) [10].

5. Innovation in NAA applications

Interesting paper published by P. Bode, the opportunities for innovation in NAA gives an overview by focused position-sensitive detection of elements in large samples, Monte-Carlo calculations replacing the use of standards, use of scintillator detectors, and new deconvolution techniques for increasing the sensitivity are examples of challenging new roads in NAA [30].

Menezes et al. reported in the paper "Advances in neutron activation analysis of large objects LSNAA with emphasis on archaeological examples", the recent developments and perspectives about the implementation of LSNAA and analyzing several kinds of matrices such as archeology, geology, and art objects [31]. During 2010–2015 period, the proficiency Tests and Inter-laboratory Comparisons have been carried out at the international level, by many NAA

laboratories, under the framework of IAEA projects using IPE (International Plant-analytical Exchange) and ISE (International Soil-analytical Exchange) samples provided by the accredited organism such WEPAL [32].

Neutron activation analysis has traditionally been used mainly for the characterization of geological, environmental, and biological materials. However, other analysis techniques have emerged to replace NAA for many of those applications, and NAA now thrives mainly because of its unique advantage, the high penetrating power of neutrons and gamma rays, leading to ease of use in many instances where no sample preparation is required. In addition, NAA practitioners have innovated to provide fast, accurate, and reliable analyses of various matrices posing great difficulties for other techniques. Some of the applications made possible by these innovations are listed here.

In the medical field, it has often been hypothesized that a lack of selenium in the body may lead to increased risk for certain cancers. To measure the body's selenium status, toenails have been found to be a good indicator of the selenium status over a period of several months prior to the taking of the sample. Several NAA laboratories [37–39] have measured Se in toenails using the very short-lived Se-77m, half-life 17.5 s. This required the development of fast pneumatic sample transfer systems with accurate control of irradiation and decay times as well as accurate correction of counting losses when the count-rate is changing during the counting period. The results of studies [37–39] using thousands of samples sometimes revealed an association between lower Se levels and increased cancer risk, while in others, no significant difference in Se level was observed between the controls and subjects who developed certain forms of cancer.

A knowledge of the levels of trace elements in plastics may be useful from several points of view, as it may reveal the presence of toxic elements such as Cd [36], or information on the production process through the presence of catalyst residues, it may permit differentiating new plastics from those containing recycled material with flame retardants Br and Sb, and it may help decide the suitability of plastics for insulation of high-voltage electrical cables [37] or cables used in the nuclear industry [38]. Plastic samples of 100 mg mass are appropriate for some applications but several NAA laboratories have innovated to be able to analyze routinely and quickly samples up to 4 g mass which, for certain trace elements, are more representative of the original material, and in special cases, even larger samples may be analyzed using large-sample NAA.

The problem of the growth of fungus or mold on wood or paper products used in humid environments, wood for windows, cardboard on wallboard used in basements, and wrappers for bars of soap stored in bathrooms, has been remediated by the application of fungicides containing heavy elements like copper and iodine. Regular quality control measurements are needed to ensure that the right amount of fungicide has been applied. NAA is ideally suited for this as there is no sample preparation and methods have been developed [39] for large representative samples, at least 6 cm^2 for wood samples cut from the surface of the board and 60 cm^2 for paper, and the NAA results are independent of penetration depth. To successfully provide a fast and reliable service for industry, the reactor and staff must be available when needed; the service must be given high priority.

Pure selenium used for photographic film must be very low in chlorine; an upper limit of 2 μg/g is tolerable. Achieving this sensitivity by INAA is difficult because Cl is detected by 37 min half-life Cl-38 with gamma rays at 1642 and 2167 keV which suffer interference from

the much more intense low-energy gamma rays emitted by several short-lived Se isotopes produced by neutron irradiation of Se. In order to reduce the detection efficiency for the interfering low-energy gamma rays relative to the high-energy Cl-38 gamma rays, discriminating gamma-ray spectrometry [40] has been used. A 10 mm thick lead plate was placed between sample and detector. To reduce the uncertainty of the calculated area of the barely visible 2167 keV peak, samples were counted for 30 m at the maximum tolerable count rate, which gave between 10 and 30% dead-time, and the peak fit was carried out using maximum predetermined information: the peak position and width were fixed relative to nearby strong Se peaks. A similar discriminating gamma-ray spectrometry technique was used [41] for the determination of vanadium in materials with high content of titanium, barium, and strontium.

To satisfy the increasing demand for rare-earth elements used in the electronics industry, new rare-earth mines are opening along with their associated refineries and the development of refining techniques. This development requires the measurement of rare-earth concentrations in the ore and products at all stages of refinement. The great sensitivity of NAA for most rare-earth elements has made it an excellent method for measuring rare-earths at low concentrations in rocks, sediments, soils, and plants. However, at the high concentrations found in ores and refinery products, the high neutron absorption cross-sections cause a severe neutron self-shielding problem even with small samples. This problem has recently been solved [42] with the development of an accurate neutron self-shielding model coupled with accurate gamma attenuation calculations for the low energy gamma rays of Ce, Nd, Sm, Dy, Ho, and Tm. The correction calculation is iterative, using the raw NAA-measured rare-earth concentrations in each sample as input and then simultaneously adjusting the neutron self-shielding factors and the gamma attenuation coefficients according to the models. For 100 mg samples with very high rare-earth concentrations, NAA results accurate to better than 5% were achieved for 13 rare-earth elements [43].

The use of nuclear analytical techniques is most practical to analyze a variety of samples in different fields related to life sciences in particular food product for humans. The objective of this research is to point out the research results of content and nutritional importance of individual essential elements that are present in various milks and dairy products. Recently, an extensively studied on nutrients and heavy metals concentration in food overall the world. In the last years, several brands of milk powder for categories child and adult have been studied by using the k_0-INAA technique [44]. As complementary work for dairy products, it is very important to evaluate the consumption of the commercial baby formula milk for the first and the second age in Algeria. The chemical element Ca, K, Na, Br, Rb, and Zn have been determined in six kinds of baby formula milk using k0-NAA technique. Results obtained in this work show a good agreement with concentration values given by producers [9].

Relevance of activation analysis is driven by its stakeholders. As an example of the existing stakeholder's needs are mainly to evaluate the whole blood and hair correlation factor of Na and K, Fe, and Zn of patients suffering from Alzheimer and psoriasis pathologies and normal controls using INAA and k_0-NAA techniques. According to the gender and age consideration, the results obtained in these studies have been discussed [45, 46]. For quality control and quality assurance, the biological matrix NIST1566b Ouster Tissue), IAEA A13 (animal blood) were analyzed simultaneously with the samples. Three statistical parameters Z-score, U-score, and bias were determined and discussed.

For more details about the basic theory of NAA, recent developments and technologies and potential applications of neutron activation analysis performed at the author's laboratory and nuclear facilities, the interested reader should refer to the works discussed in Chapters 2–6.

Chapter 2: Neutron Activation Analysis: Application in Geology Application in Geology and Medicine (*Maitreyee Nandy*).

Chapter 3: Colombian Neutron Activation Analysis Laboratory – CNAAL. Applications and Development using the Nuclear Research Reactor IAN-R1 (*Guillermo Parrado et al.*).

Chapter 4: Monte Carlo simulation of Correction Factors for Neutron Activation Foils (*Pham Ngoc Son*).

Chapter 5: Neutron Activation System for ITER Tokamak (*Vitaly Krasilnikov et al.*).

Chapter 6: An Overview of the Establishment of Methodology to Analyze up to 5 g-Sample by k_0-Instrumental Neutron Activation Analysis, at CDTN, Brazil (*Maria Menezes et al.*).

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Author details

Lylia Alghem Hamidatou

Address all correspondence to: lylia.h@hotmail.fr

Nuclear Research Centre of Birine, Algerian Atomic Energy Commission, CRNB/COMENA, Algeria

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