Chapter 2

Neutron Activation Analysis: Application in Geology and Medicine

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Additional information is available at the end of the chapter

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Abstract

Varied forms of neutron activation analysis (NAA), due to their high accuracy and reproducibility, are being used in geological studies and in medical application for the determination of concentration of elements down to the trace and ultra-trace level. Concentration of Cs, Sc, Fe, Ta, Co and Eu which may give rise to long-lived activity on neutron irradiation has been determined down to 0.1 ppm in rock samples from 11 geological formation in Karnataka, India, using NAA. NAA has been used by several authors to determine elemental concentration in biological shields, different geological formation around the world, thermal springs, archaeological objects and precious stones. NAA has been successfully employed by different groups to determine the concentration of Al, K, Na, Cl, Rb, Ca, Cu, Co, I, Mg, Se, Fe, Zn, Hg, Ba, Cr, etc. and their relative variation in breast cancer, skin cancer, colorectal cancer, dysfunction and malignancy of thyroid gland.

Keywords: neutron activation analysis (NAA), elemental concentration, geology, medicine, radioactivity

1. Introduction

Neutron activation analysis (NAA) is a state-of-the-art analytical technique used for identification of elements and determination of the elemental concentration in different applications. With the development of accurate and fast gamma spectrometric techniques, advanced electronics and automation in data analysis, the method has gained active application in diverse field of basic research. Non-destructive or instrumental NAA has also become popular for varied industrial application due to its ease in handling. With the development of high-power neutron source, precise and fast neutron detectors and sophisticated electronics, NAA has

achieved ultralow minimum detection limit (MDL). As a result the method has turned out to be one of the best choices for quantification of trace elements in a wide variety of samples—geological, archaeological, biological and environmental samples.

Varied analysis devices for neutron activation studies have made it a preferred choice for the analysis of construction material, coal, environmental, geological and archaeological samples and biological material, security monitoring and academic studies. Since its inception around the middle of last century, different variants of NAA such as prompt gamma neutron activation analysis (PGNAA), pulsed fast neutron analysis (PFNA), pulsed fast/thermal neutron analysis (PFTNA) and associated particle imaging (API) have been developed. Non-destructive NAA is one of the most preferred methods of sample analysis as it requires minimal processing of the activated sample. In the semiconductor industry, NAA is used to determine ultra-trace concentration of impurities, or dopant as impurity concentration even at 1 ppb can affect the performance of the semiconductor. NAA-induced associated particle imaging (API) efficiently used for security monitoring has several advantages over other conventional scanning methods.

Different forms of NAA offer good choice over other techniques for application in geology and medicine. NAA is used for quantitative analysis of trace elements in rock samples which serves as an important tool for modelling geochemical processes and also helps in sample selection for other applications. Pai et al. [1] have used instrumental NAA (INAA) for trace element analysis in different rock samples to determine its suitability for use in the coarse aggregate for concrete in shielding material. Over the last several years, NAA has made important inroads in the field of medical science. The method is used in production of radiotracers which are used in situ for evaluation of new pharmaceuticals for their distribution, time release, clearance, etc. Neutron-stimulated emission computed tomography (NSECT) is a newly developed imaging method that non-invasively maps the concentration of an isotope in the body [2]. Multiple pencil beams of fast neutrons are developed for early diagnosis of breast cancer through NAA using differential femto-oximetry (DFO) [3]. This chapter will discuss application of different forms of NAA in geology and medicine and related recent advancements.

2. Method

Neutron activation analysis (NAA) is a procedure employed for analysing the elemental composition of a material and for determining the concentrations of elements at the trace and ultra-trace level in a vast majority of samples. In this method the sample is irradiated with neutron from the reactor when nuclear reaction is induced in the sample. The gamma rays emitted from the isotope as a result of this reaction is detected and counted and the reaction analysed. The activity of the ith isotope induced in the sample is given by

$$A_i = N\left[\int \sigma_i(E)\varphi(E)dE\right]\left(1 - e^{\lambda_i t_l}\right) \tag{1}$$

where N is the number of target atoms available for interaction, σ_i is the cross section for production of the ith isotope, $\varphi(E)$ is the neutron fluence rate at energy E, λ_i is the decay constant for the ith isotope, t_I is the time of irradiation.

Gamma spectrometric analysis of the irradiated sample helps one to determine the elemental composition and impurity of the sample. There are different types of NAA that can be used for the analysis of a sample.

In many applications the sample under study is irradiated in a reactor or by a neutron source, and the exposed sample is analysed for gamma emission. No chemical treatment of the sample is carried out between irradiation and counting. This type of NAA is called instrumental neutron activation analysis or INAA. This is also known as non-destructive neutron activation analysis. In the second method, the sample after irradiation with neutrons is treated chemically in order to separate different constituents depending on their solubility or other physical properties. The separated constituents are then counted separately for their characteristic gamma emission. This method is called radiochemical neutron activation analysis (RNAA). INAA can again be carried out in more than one way—prompt gamma neutron activation analysis (PGNAA) and delayed gamma neutron activation analysis (DGNAA). In the first method, gamma counting is done online when the sample is irradiated. The neutron interacts with the target nucleus, and the latter goes to an excited state which thereby decays to the lower energy states with the emission of gamma radiation. The reaction time is of the order of 10^{-15} s. Analysis of these gamma rays provides information on the incident neutron flux as well as the composition of the sample. In DGNAA gamma spectrometry of the irradiated sample is carried out after the irradiation is over. In this method the decay gammas from the product radioisotopes are counted. The k0-NAA is one of the most accepted standardisation methods for quantitative multielemental analysis of the sample through gamma spectrometry. In this method one standard or reference material with known amount of an isotope and a single comparator are irradiated and counted along with the sample under the identical conditions. The quantity of the different elements present in the sample is estimated using the k-factors determined from the analysis of the standard and the comparator. The varied forms of NAA are applied for quantitative analysis of different types of samples.

3. Neutron activation in geology

Different variant of NAA is employed for detection and quantification of the trace and ultra-trace level of elemental concentration in different geological formation, uranium ore, etc.

NDNAA and Compton-suppressed NAA are efficiently used for analysis of geological samples (i) for uranium content measurement in phosphate rocks and surrounding geology and (ii) in trace elemental analysis of rock samples. Trace elemental analysis of rock samples is particularly important as these rocks can find a novel use as the coarse aggregate in varieties of concrete that may be developed for shielding of accelerator and reactor facilities [1].

Analysis of rare earth and other trace elements in the rock samples helps geoscientists to understand the chemistry of rock formation [4]. NAA studies also suggest that extinction of the dinosaurs occurred soon after the impact of a large meteorite with the earth.

Neutron reaction cross section strongly depends on the energy and sharply increases as energy decreases below 0.5 eV (**Table 1**).

Phosphate rocks contain other elements adjacent in the periodic table. Of these are Na, Mn and Cl which may interfere with the detection of uranium. In the following portion, we shall have a look on the gamma spectrometric properties of the stable isotopes of these elements (**Table 2**).

Precise determination of the elemental content of the phosphate rocks is crucial in the understanding of geochemistry of the formation. INAA and delayed neutron activation analysis (DNAA) were employed to determine the concentration of uranium in some Egyptian environmental samples (Toshki soil, Aswan iron ore) and phosphate samples in the Red Sea coast area [4]. The study showed, in consonance with studies by other workers, that the phosphate rocks are rich in natural sources of uranium.

INAA coupled with gamma ray spectrometry was employed to determine the concentration of the three major elements U, Th and K collected from the lower Benue region of Nigeria [5]. The authors observed a higher abundance of Th than in the other regions. For some samples as the potassium contents were fairly normal, high values of the Th (ppm)/K ratios were obtained.

Shutdown of reactors, accelerators and other radiation facilities leads to generation of radioactive waste from shielding and other materials of the facility which got activated over the years

^{238}U	Thermal, 2.683 b; epithermal, ~1 b with sharp resonances
^{235}U	Thermal, 98.8 b; epithermal, ~10 b with sharp resonances

Table 1. Neutron capture cross sections for ^{238}U and ^{235}U.

Stable isotope	Cross section (n, γ) (b)	Half-life (radioisotope)	Gamma energy (MeV) (intensity)
	Thermal		
^{23}Na	0.4	14.96 h (^{24}Na)	1.37 (99.99)
			2.75 (99.93)
^{37}Cl	0.4	37.23 min (^{38}Cl)	1.64 (33.3)
			2.16 (44.4)
^{55}Mn	15	2.58 h (^{56}Mn)	0.847 (98.85)
			1.81 (26.9)
			2.11 (14.2)
^{81}Br	0.25	1.27 min (^{82}Br)	0.511 (21.0)
			0.776 (99.0)

Table 2. Gamma spectrometric properties of isotopes relevant for INAA of phosphate rock.

of operation of the facility. Radioactive concrete is one of the major constituent of this waste. The concrete shield in reactors, low- and medium-energy accelerator facilities contain induced activity from neutron activation, while in high-energy accelerators, gamma activation also plays a significant part. Long-lived radioactivity in the concrete waste poses a serious problem for its management and disposal. This entails a detailed in-depth analysis of the elemental composition of the concrete to be used in the shielding of the accelerator facilities.

Coarse and fine aggregates are the primary components of concrete. In their work for development of self-compacting concrete (SCC), Pai et al. [1] had analysed rock samples from 11 different geological formation from the state of Karnataka, India. The different types of rocks analysed were gneiss, granite, trap, basalt, peninsular gneiss, dolomite rock, Deccan trap, sandstone, quartzite, limestone and laterite. Elemental composition of the samples was determined using non-destructive neutron activation followed by low-background gamma spectrometry. Concentration of the elements Cs, Sc, Fe, Ta, Co and Eu which may result in long-lived activity build-up was measured down to 0.1 ppm. A detailed analysis of the activated samples from all the 11 geological formation showed that quartzite has the lowest concentration of these elements and hence has the minimum activity build-up [1]. But it failed the fresh and hardened property test which determines the acceptance criteria for the SCC. Hence, dolomite rock which showed the second lowest activity build-up was inferred as the most suitable choice to be used as the coarse aggregate in developing the SCC. Some other types of rocks like Deccan trap and laterite showed a high concentration of neutron-induced activity and are not desirable during decommissioning or disposal. Hence, these are not recommended to be used as a constituent of the concrete shielding.

Medhat et al. [6] have assessed the contents of trace elements, in the biological shield of a decommissioned nuclear power reactor, which gives rise to significant amounts of radioactivity after long periods of operation. Several cement samples were studied through fast neutron irradiation followed by gamma spectrometry. The (n,γ) reaction was monitored to estimate the concentration of different elements like Ce, Co, Cs, Eu, Fe, Hf, Sb, Sc, Ta and Tb.

Nazarov et al. [7] studied the activity generation in the concrete of the shielding material of radiation facilities in order to understand the impact on their decommissioning procedure. Since the activity is produced mostly through (n,γ) reaction, analysis of the elemental composition of the raw materials plays a crucial role in the choice of the components of the cement and concrete. This elemental analysis was carried out by neutron activation and subsequent gamma spectrometric studies. The authors observed that the analysis of the elemental composition needs to be carried out at the design stage of the facility. This would give an idea of the radioactive waste generated at the decommissioning stage.

Alden et al. [8] has used INAA to determine the elemental composition of 157 archaeological samples and geological clay from Northern Chile. They observed that the major groups consisting of high chromium and low chromium were found in clays from within the region. The three minor groups containing low sodium comprised the vessels imported from northwest Argentina. The distribution pattern indicated that the constituent material differed for different groups of items. Compositional analyses also indicated that the Inka-style ceramics were produced in the region during the period of Inka domination.

Huckell et al. [9] has used INAA to determine the composition of microcrystalline sedimentary rock (chert) from the western part of North Dakota. The rocks came from a vast geological formation on the central and northern plains and were a part of the Eocene-age White River Group. They found that elemental composition of the rocks analysed was distinct and helped its separation from the other cherts.

Michelsen and Steinnes [10] had used thermal neutron activation to estimate the relative abundance of copper in some geological samples. The activated samples were analysed by gamma spectrometry using the annihilation photons from ^{64}Cu which is a positron emitter. The coincidence counting of annihilation photons largely reduces the interference from ^{24}Na. ^{24}Na can be formed in the sample through neutron activation of the stable isotope ^{23}Na which is often present in significant amount in geological materials. Copper concentration to the level of 100–1000 ppm could be successfully determined in this method.

Ravisankar et al. [11, 12] used instrumental neutron activation analysis to detect and determine the elemental composition of some beach rock samples in the South East Coast of Tamilnadu, India. Along with other constituents, concentrations of the rare earth elements (REE) were measured. The authors used single comparator method in this study. They determined the fraction of 19 elements in the 15 samples collected from the area. High-resolution gamma spectrometry was used for the purpose. Irradiated Standard Reference Material (SRM 1646a Estuarine sediment) was measured for calibration. The geochemical behaviour of REE in beach rock was studied.

Natural emeralds, associated rocks obtained from Rajasthan, India, were analysed for contents of different elements [13]. The content ratio for 21 elements was estimated by instrumental neutron activation analysis (k0 INAA method) and high-resolution gamma ray spectrometry. Elemental analysis of the samples carried out by the authors showed that some elements from the trapped and host rocks were separated and preferentially combines with the mineral beryl forming the gemstones. For quantitative analysis of the samples, the results were compared with those for the reference rock standard of the US Geological Survey (USGS BCR-1).

Several works using INAA were carried out by El-Taher A. and his group to characterise the elemental composition of different geological formation around the desert in Egypt and the Egyptian Red Sea coast. INAA of 25 elements, As, Ba, Ca, Ce, Co, Cr, Cs, Eu, Fe, Hf, K, La, Lu, Mg, Mn, Na, Nd, Rb, Sc, Sm, Th, U, Yb, Zn and Zr, was carried out in quartz collected from the region [14]. Activation of the samples was done in the TRIGA Mainz research reactor and the activated samples were analysed using standard gamma spectrometric method. As claimed by the authors, these results are the first data for elemental composition of Egyptian quartz.

Detailed quantitative analysis was carried out by the same group also for granite samples collected from different locations in South Egypt [15, 16]. Elemental content for a total of 28 elements comprising of several rare earth elements (REEs) was estimated. INAA as a sensitive non-destructive analytical tool is a preferred choice for quantitative determination of REEs because strong similarity in chemical behaviour of these elements renders the chemical methods inefficient for identification and estimation of the elements. The elements quantified were Na, Mg, K, Fe, Mn, Sc, Cr, Ti, Co, Zn, Ga, Rb, Zr, Nb, Sn, Ba, Cs, La, Ce, Nd, Sm, Eu, Yb,

Lu, Hf, Ta, Th and U. The results were compared with those obtained from X-ray fluorescence of the same samples. Next, in this series of work by El-Taher et al., concentration of chromium and 15 trace elements was measured in the chromite rock samples collected from the Eastern Desert in Egypt [17]. Short periods of irradiation were used to measure the short-lived isotopes of Mg, Ti and Mn, while longer irradiation periods were used for quantification of Na, Ga, As, La, Sc, Cr, Fe, Co, Zn, Zr, Ce, Ce, Yb, Lu, Hf and Ta.

El-Taher et al. have used INAA to estimate the concentration of gold and 31 other elements in the 10 samples collected from two Egyptian gold ores in the Eastern Desert region [18]. They determined 42.4% and 25.7% abundance of gold for the samples collected from the two ores.

Schwedt et al. employed neutron activation to analyse ceramic vessels found during excavations of ancient cemeteries in different parts of Boeotia, Greece [19]. The analyses resulted in a clear separation between the samples obtained from the shores of the ancient lake Copais and those from eastern parts. Analysis of the elemental compositions revealed that for some of the samples the results were similar to those obtained among Bronze Age samples from the same region. On the other hand, composition of the samples from the Theban tombs indicated import from regions as distant as Asia Minor.

Srivastava et al. [20] has quantified the concentrations of selenium, arsenic and 13 other elements in soil samples rich in selenium from the state of Punjab, India. The samples were irradiated in research reactor, and the activated samples were counted using high-resolution γ ray spectrometry. Quantification was done using the comparator method using soil samples collected from a non-seleniferous region.

Quantitative analysis of the composition of pumice obtained from archaeological excavations throws light on important parameters like their origin, age, transport route, etc. Steinhauser et al. have used neutron activation on pumice samples from the Mediterranean regions Milos, Santorini, Kos, Gyali and Nisyros (Greece), Lipari (Italy) and Cappadocia (Turkey) [21]. Through gamma spectrometric analysis of the activated samples, they have determined the concentrations of the elements As, Ba, Ce, Co, Cr, Cs, Eu, Fe, Hf, K, La, Lu, Na, Nd, Rb, Sb, Sc, Sm, Ta, Tb, Th, U, Yb, Zn and Zr and inferred about the origin and ages of the pumice samples.

Wasserman et al. [22] have studied the geochemistry of Sepetiba Bay in four sediment cores. INAA was used to determine the total concentration of the metals Ba, Co, Cr, Cs, Fe, Hf, Rb, Sc and Zn; the rare earth elements Ce, Eu, La, Lu, Sm and Yb; the actinides Th and U, along with As and Br; organic carbon; and total sulphur. They found that the top layers had strong zinc contamination (1000 μg/g or higher).

Instrumental neutron activation analysis combined with pattern recognition techniques was applied by Watterson et al. to understand the mineralisation process in granites and classification of diamonds and to identify the sedimentary units in Witwatersrand, South Africa [23]. Classification of coals from the Witbank Coalfield was also carried out. The method proved to be an efficient tool for identification of sedimentary unit mapping of geological formation.

Limestone samples from different areas in the Longmen Grottoes, Henan province, China, were analysed by Zhu et al. [24]. Concentrations of major elements, trace elements and REEs

were determined. The authors identified three different groups of rocks—the samples from the northern part were made of dolomite rocks, and those from the middle and southern part are mainly made of limestone.

Contis [25] has measured the concentration of Se, V, As, Hg and Cd in the water from thermal springs using NAA. The study was carried out to understand the effect of thermal springs in the eastern Aegean Sea in Greece, Ikaria, on drinking water from sources near and far from the spring. The measured concentration of the elements did not show statistically significant variation from that due to natural background.

4. Neutron activation in medicine

NAA is used in different applications in medicine and has made inroads in the newer areas.

Trace elements have important bearing on the physiological and biochemical processes, and relative abundance and balance of different elements in trace quantities strongly influence the occurrence and advancement of many diseases. Estimation of trace element concentration in breast cancer, skin cancer, colorectal cancer, dysfunction and malignancy of thyroid gland has been done using neutron activation followed by high-resolution gamma spectrometric studies. NAA is one of the preferred choices for trace- and ultra-trace-level quantitative estimation as it is a highly accurate, precise and reproducible method even for measurements to the ppm and ppb level.

Concentration of Ca, Cu, Co, I, Mg, Se, Fe, Zn, Hg, Ba and Cr at the trace level in the malignant tissues of colorectal cancer was determined through NAA by H. Arriola et al. [26]. The study was carried out for patients from Mexican population. The results were compared with those obtained for normal tissues in the same population. It was observed that the amount of Co, Fe, I and Ba changes due to incidence of colorectal cancer.

NAA was used to estimate the concentration of Al, K, Na, CI, Rb, Co, Sc, Mn, Mg, Se, Zn, Cs, Fe and Cr in the patients of breast cancer in Sudanese population (Ammar Mubark Ebrahim A M, 2003) [27]. Though the number of patients studied was only 80, it was found that the amount of Al, Mg, Cr, Mn, Se and Zn is higher in the malignant tissues compared to the normal tissue. In this work the author has observed variation in the levels of K, Na, Fe, Co, Sc, Rb and Cs but to a much lesser extent.

Rees et al. [28] have used INAA to detect and measure the arsenic content in the toenail of the skin cancer patients. Tissues from both basal skin carcinoma (BCS) and squamous cell carcinoma (SCC) were examined from the patients of non-melanoma skin cancer.

Zaichick and Zaichick [29] have studied the changes in trace element concentration in the cancerous human prostate gland. They have used INAA to measure the concentration of 43 trace elements and compared the results for malignant, benign hypertrophic and normal prostate. Of the 43 elements measured, concentration of 33 elements in the malignant prostate is higher than that in the benign hypertrophic tissue. For the elements Co, Hg, Rb, Sc, Se and

Zn, a reverse trend was observed. When compared with the normal tissue, lower contents of Sc, Se, Zn, Rb and Cd were detected in the malignant prostate.

Zaichick et al. [30] have studied the role of trace elements in the induction and advancement of thyroid cancer. Contents of 11 elements, namely, Sc, Se, Zn, Co, Cr, Fe, Hg, I, Rb, Sb and Ag were determined in malignant and non-malignant thyroid nodules as well as in the non-affected paranodular thyroid tissue. Measured concentration of the elements mentioned was compared with the reference standard material H-4 of the International Atomic Energy Agency (IAEA). The results of this study showed that the level of Ag, Co, Hg, I and Rb is higher in the paranodular tissue. Selenium deficiency was also reported in this work for malignant thyroid. Zaichick and Zaichick [31] also studied the influence of different elements in the functional behaviour of thyroid and their dependence on sex and age.

Neutron activation analysis through monitoring of the delayed gamma radiation (DGNA) in combination with dual-energy X-ray absorption was used by Aloia et al. [32] to estimate the total body calcium. For the same population, the results obtained by the two methods vary more than 20%.

Gamma ray imaging plays a significant role in the pharmaceutical industry in development and progress of a drug delivery system. The standard form of radiolabelling of the drug molecules is done using some of the most commonly used medically important radioisotopes, like ^{99m}Tc or ^{111}In. But for complex drug molecules, radiolabel is produced through in situ neutron activation [33].

NSECT or neutron-stimulated emission computed tomography is one of the most advanced imaging techniques employed to study the isotope distribution in biological tissue [2, 34]. The method depends on irradiation of the sample by fast neutrons. The gamma rays emitted in the nuclear reaction induced in the isotopes in the tissue under study are monitored to construct tomographic images of each section of the sample. Though the instrumentation is expensive, high sensitivity of the technique has rendered it suitable to be used for cancer staging, detection of breast cancer.

Another important advancement in the realm of nuclear medicine is the early diagnosis of breast cancer with the help of the fast neutrons. Multiple pencil beams are developed to carry out NAA of the breast tissue. Since the oxygen content of the cancer tissue is different from that in the normal tissue, differential femto-oximetry is used in the diagnosis of the malignant tissue [3]. With the advancement in beam profile variation, the technique may be used for diagnosis of other types of cancer.

5. Conclusion

Neutron activation analysis is one of the preferred techniques for quantitative analysis of different types of samples and has thus found wide application. It is a highly accurate method and can reliably be applied for measurement of concentration of elements at the trace and ultra-trace level. We have discussed how NAA, particularly INAA, can be used for determination of

elemental concentration of rocks and other geological formation. The analysis provides information about the geological formation of the region. Moreover, rock samples may be used as coarse aggregates in cement or concrete in the biological shield of a nuclear installation. NAA of the rock samples helps the researcher to estimate the long-lived activity that may be developed in the shield due to long-term operation. This study helps to plan the management of radioactive waste after decommissioning of the facility. NAA of rock samples from Rajasthan, India, carried out by Acharya et al. has helped to understand the formation of natural emeralds. NAA has important contribution in medicine: biochemically significant elements in trace amount can be effectively quantified using INAA. Variation in elemental composition induced by various diseases, particularly for malignant tissues, can be quantified using INAA. NAA with associated particle imaging (API) is a prospective tool for elemental analysis to monitor the growth of an animal in response to new genetic, pharmacologic procedure. The inherent high accuracy of NAA will offer it as one of the preferred techniques for elemental analysis at the ultra-trace level in human body fluid to follow the biochemical baseline values or the changes therein due to diseases.

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