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New generation non-stationary portable neutron generators for biophysical applications of Neutron Activation Analysis☆

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ABSTRACT

Background: Neutron sources are increasingly employed in a wide range of research fields. For some specific purposes an alternative to existing large-scale neutron scattering facilities, can be offered by the new generation of portable neutron devices.
Scope of review: This review reports an overview for such recently available neutron generators mainly addressed to biophysics applications with specific reference to portable non-stationary neutron generators applied in Neutron Activation Analysis (NAA).
Major conclusions: The review reports a description of a typical portable neutron generator set-up addressed to biophysics applications.
General significance: New generation portable neutron devices, for some specific applications, can constitute an alternative to existing large-scale neutron scattering facilities. Deuterium-Deuterium pulsed neutron sources able to generate 2.5 MeV neutrons, with a neutron yield of 1.0×10^6 n/s, a pulse rate of 250 Hz to 20 kHz and a duty factor varying from 5% to 100%, when combined with solid-state photon detectors, show that this kind of compact devices allow rapid and user-friendly elemental analysis. "This article is part of a Special Issue entitled "Science for Life" Guest Editor: Dr. Austen Angell, Dr. Salvatore Magazù and Dr. Federica Migliardo".

1. Background

1.1. A brief overview on neutron applications

Nowadays neutrons are used in different fields of science, including elemental analysis, art and archaeology, environmental and geological fields, electronics, medicine and especially in biophysics applications [1–15].

The use of neutrons in some of these fields has been limited by the large amount of sample required, however neutrons furnish many advantages in the study of the structure and dynamics of biological samples, since these systems have a high hydrogen content and so neutrons allow the visualization of hydrogen atoms [16–33]. For solution structures of macromolecular complexes that can often not be crystallized for structure determination, neutron scattering take advantage of contrast variation by changing the isotopic composition of both the solvent and the individual macromolecular components to clarify these structures at intermediate resolution [34–56]. More specifically, neutrons can be used as probe for biological systems for the atomic and molecular detail, to image processes at the organism level under realistic conditions. In this case, the advantage is the high penetrating power, in contrast between light elements and lack of radiation damage. Neutron imaging is useful to study the water uptake in plant roots, a process of obvious agricultural importance that is difficult to image with light microscopy.

Reactor technology, developed in 1940s reached a plateau in performance in 1970s with the construction of ILL (in France) and HFIR (in USA). The spallation process, developed in the 1970s, opened the opportunity to produce brighter beams. Currently operating high-flux spallation sources include ISIS (UK), SNS (USA) and the MLF at J-PARC (J).

In this framework, the European Spallation Source (ESS), in construction to Lund, Sweden, will have a long pulse, particularly well-suited for a number of neutron techniques relevant for biological systems [57–70]. In combination with modern isotope labelling techniques, it will allow experiments with a far larger number of biologically interesting samples. Detailed studies are dependent on how many neutrons can be produced by a neutron source. This is a significant limitation for existing sources based on nuclear reactors. As a result, scientists and engineers have developed a new generation of neutron sources based on particle accelerators and spallation technology, a

Abbreviations: NAA, Neutron Activation Analysis; ESS, European Spallation Source; NGs, Neutron Generators; D-D, Deuterium-Deuterium; D-T, Deuterium-Tritium; INAA, Instrumental Neutron Activation Analysis; PGNAA, Prompt-Gamma Neutron Activation Analysis; DNAA, Delayed Neutron Activation Analysis; PINS, Portable Isotopic Neutron Spectroscopy; HPGe, High-Purity Germanium.

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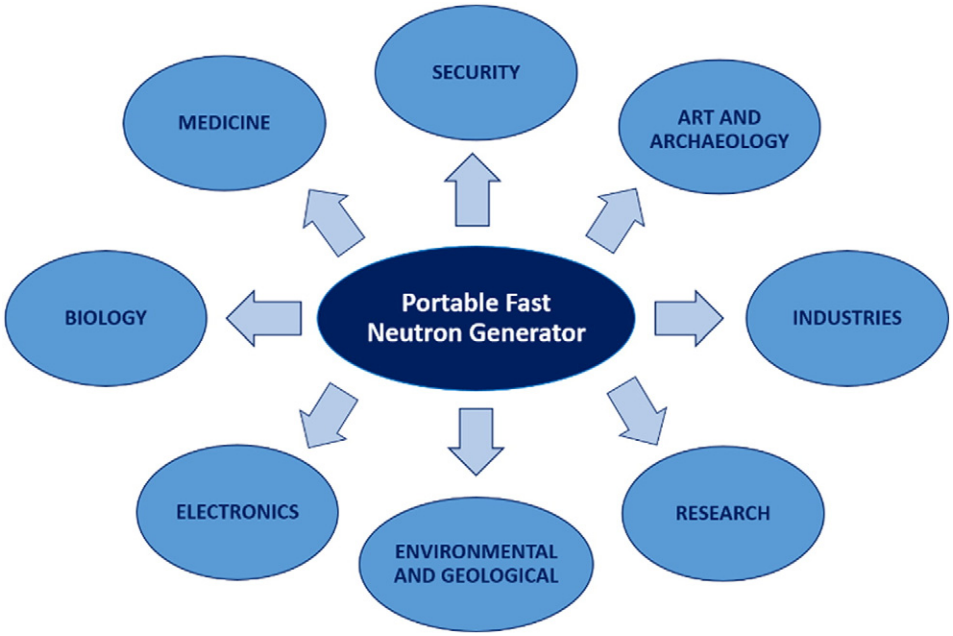

Fig. 1.1. Possible application fields of a Portable Fast Neutron Generator, in particular in security, in art and archaeology, in industries, in research, in environmental and geological field, in electronics, biology and medicine.

much more efficient approach. ESS, for example, will provide up to 100 times brighter neutron beams than existing facilities today.

For some specific purposes an alternative to existing large-scale neutron scattering facilities, can be offered by the new generation of portable neutron devices [71–104]. In the following paper an overview of new generation portable neutron generators, mainly addressed to biophysics applications is presented [105–122]. In particular, the employment of portable non-stationary neutron generators in Neutron Activation Analysis (NAA) is discussed [123–155]. Finally some relevant examples of biophysics applications are reported, as obtained by a deuterium-deuterium pulsed neutron source able to generate 2.5 MeV neutrons, with a neutron yield of 1.0×10^6 n/s, a pulse rate of 250 Hz to 20 kHz and a duty factor varying from 5% to 100%; when combined with solid-state photon detectors it allows rapid and user-friendly elemental analysis and some examples of data analysis.

1.2. Portable neutron generators

In the last fifty years, neutron generators were developed and then employed in several fields of application.

The potentiality and the advantages of the portable neutron generators, based on fusion reactions, to produce easily fast neutrons, instead of spallation sources, radioactive decay neutron sources and spontaneous fission sources were well know already in 1961. In the last years, neutron generators were developed and then employed in several fields of application. Nowadays, neutron generators are diffusely employed, ranging from imaging techniques, to material characterization, from geological field to electronics (testing of devices), in non-destructive methods, in research activities thank to their simple structure and their simple functioning and most in biophysics. Among the various light-ion accelerators, compact devices designed as hermetic, sealed tubes that use Deuterium–Deuterium (D–D) and Deuterium–Tritium (D–T) reactions have found the most widespread use in industry:

$$D + D \rightarrow {}^{3}_{2}He + {}^{1}_{0}n + 3.270\,MeV$$

$$D + T \rightarrow {}^{4}_{2}He + {}^{1}_{0}n + 17.590\,MeV$$

These accelerators generate neutrons of ~2.5 and ~14.1 MeV, respectively. They consist of a source able to generate positively charged ions; one or more devices to accelerate the ions; a metal hydride target loaded with either Deuterium, Tritium, or a mixture of the two; and a gas-control reservoir, also made of a metal hydride material. The most common ion source used in neutron generators is a cold-cathode, or Penning

Table 1.1
Applications of Portable Fast Neutron Generator in different fields: security, art and archaeology, industries, research, environmental and geological field, electronics, biology and medicine.

Security	Detection of explosive material, illegal drugs, chemical and nuclear weapons, luggage or cargo inspections	[173–176]
Arts and archaeology	Art objects and archaeological artefacts typically not permitted to investigate with destructive techniques	[177–179]
Industries	On-line analyzers of raw materials transported on conveyor belts, especially in cement factories; analysis of minerals or materials with similar compositions (glass, cement, coal)	[180–182]
Research	Reaction cross-sections and interfering reactions	[183–185]
Educational	Training of young scientists in various aspects of neutron generation and diagnostics and a wide spectrum of modern technologies, such as those related to ion sources, particle acceleration techniques, beam handling and diagnostics, magnet technology, vacuum techniques, detector technology, nuclear electronics, data acquisition as well as processing techniques	[186–187]
Environmental and geological	Environmental studies focused on elements of interest such as Hg, Cd, As, Cu; recycled material (metals, plastics or mixtures thereof), waste rock piles, mine tailings, etc.…; in agriculture it is useful for detecting pesticide residues on crops	[188–190]
Electronics	Measure of impurities in silicon semiconductors and neutrons testing on electronic devices	[191–195]
Biology and medicine	Living material is composed mainly of carbon, hydrogen, oxygen, and nitrogen, the presence or absence of other elements can have a profound effect on the well-being of an organism. Thus, it is being used in several laboratories to measure the concentrations of magnesium, copper, zinc and other metals in human blood	[196–200]

ion source, which is a derivative of the Penning trap, used in Penning ion gauges.

As far as the elemental analysis is concerned, a low neutron flux of $10^7 \div 10^8$ n/s is required, that furnishes the advantage to measure easily prompt gamma from (n,γ) and (n,n′,γ) reaction and allows to work safer, both in steady and in pulse mode, compared to radioactive neutron sources. In order to produce this neutron flux an ion source is used, such as Deuterium ions, of a few tens of microamperes and >80 keV energy, which produce neutrons of $10^6 \div 10^8$ n/s from D-D or D-T reactions.

In these last years, Portable Fast Neutron Generators (NG)s are becoming a valid alternative to nuclear reactors in many industrial applications, in fields of neutron science, medical research and various elemental analysis applications, ranging from art and archaeology to environmental and geological fields, from the research to electronics, medicine and especially in biophysics applications. Such a technological development has widened the horizons of Portable Fast Neutron Generators in a variety of fields. Fig. 1.1 and Table 1.1 resumes the most significant applications, while Table 1.2 reports some of commercial Neutron Generators.

Another relevant application of neutron portable sources is represented by Neutron Stimulated Emission Computed Tomography (NSECT) [156–172], which is being developed as a non-invasive imaging technique to determine element concentrations also in the living organisms. NSECT uses a neutron beam, which scatters inelastically from atomic nuclei, generating characteristic gamma photons that can be detected and identified. From the gamma photons energy, it is possible to infer the elemental composition of the target.

1.3. Elemental analysis

Elemental analysis is of basic importance in life sciences since neutrons allow determining of the different components of a sample resulting, in many cases, more sensitive in respect to other analytical methods [123–134]. One of the forms of interaction between neutrons and matter is the neutron capture; in this process (Fig. 1.3), a target,

Table 1.2
Characteristics of some commercial Portable Fast Neutron Generator, in particular Thermo Scientific, Sodern, Adelphi tech. and VNIIA.

Model	Applications	Maximum neutron yield [n/s]	Typical tube lifetime	Operating mode
Thermo Fisher Scientific, Inc.				
API 120 NG	Explosive detection, Buried land mines, Chemical weapons, UXO analysis, Drugs detection, In-Vivo body composition	2.00E + 07	1200 h @ 10^7 n/s	Continuous only
D 711 NG	Large object imaging in cargo, Radiation effects research, Fast neutron radiography, Neutron activation analysis	2.00E + 10	1000 h @ 10^{10} n/s	Continuous only
MP 320 NG	Explosive detection, Buried land mines, Chemical weapons, UXO analysis, Drug detection, In-Vivo body composition, Minerals mining and exploration, Bulk materials (coal, cement)	1.00E + 08	1200 h @ 10^8 n/s	Continuous and pulsed
P 211 NG	Transuranic waste assay, Fissionable Materials	1.00E + 08	Up to 500 h or greater	Continuous and pulsed
P 385 NG	Explosives detection, Bulk materials analysis, WMD detection, UXO analysis, Contraband detection, Vehicle inspection	5.00E + 08	4500 h @ 10^8 n/s	Continuous and pulsed
Sodern				
GENIE 16	Neutron analysis for scientific education, Neutron analysis for research, Industrial on line analysis	2.00E + 08	8000 h @ 5 × 107 n/s	Continuous and pulsed
GENIE 35	Neutron analysis for scientific education, Neutron analysis for research, Industrial on line analysis	1010/4π n/s/sr	2000 h @ 1010/4π n/s/sr	Continuous and pulsed
Adelphi Technology, Inc.				
DD108	Prompt Gamma Neutron Activation Analysis (PGNAA), Neutron Activation Analysis (NAA)	1.00E + 08	2000 h	Continuous and pulsed
DD109.1	Prompt Gamma Neutron Activation Analysis (PGNAA), Neutron Activation Analysis (NAA), fast neutron radiography.	1.00E + 09	2000 h	Continuous and pulsed
DD109.4	Prompt Gamma Neutron Activation Analysis (PGNAA), Neutron Activation Analysis (NAA), fast neutron radiography.	4.00E + 09	2000 h	Continuous and pulsed
DD-109 M	Prompt Gamma Neutron Activation Analysis (PGNAA), Neutron Activation Analysis (NAA), fast neutron radiography.	4.00E + 09	2000 h	Continuous
DD110MB	Prompt Gamma Neutron Activation Analysis (PGNAA), Neutron Activation Analysis (NAA), determining the concentrations of elements in many materials.	2.00E + 10		Continuous
DT108API	High resolution imaging	1.00E + 08		Continuous
DT110–14 MeV Neutron Generator	Security, baggage and container screening for the detection of explosives or contraband and for the detection of special nuclear materials, imaging and elemental analysis.	1.00E + 10		Continuous
All-Russia Research Institute of Automatics – VNIIA				
ING-013	Elemental analysis	5.00E + 09	1600 h @ 10^8 n/s	Pulsed
ING-03	Elemental analysis	3.00E + 09	1600 h @ 10^8 n/s	Pulsed
ING-031	Elemental analysis	2.00E + 10	1600 h @ 10^8 n/s	Pulsed
ING-07	Elemental analysis	1.00E + 09		Continuous and pulsed
ING-17	Elemental analysis	3.00E + 08		Continuous and pulsed
ING-27	Elemental analysis	1.00E + 08		Continuous
NG-14	Elemental analysis	2.00E + 10		Continuous
ING-10	Research activities	5.00E + 08		Pulsed
ING-12	Research activities	2.00E + 09		Pulsed

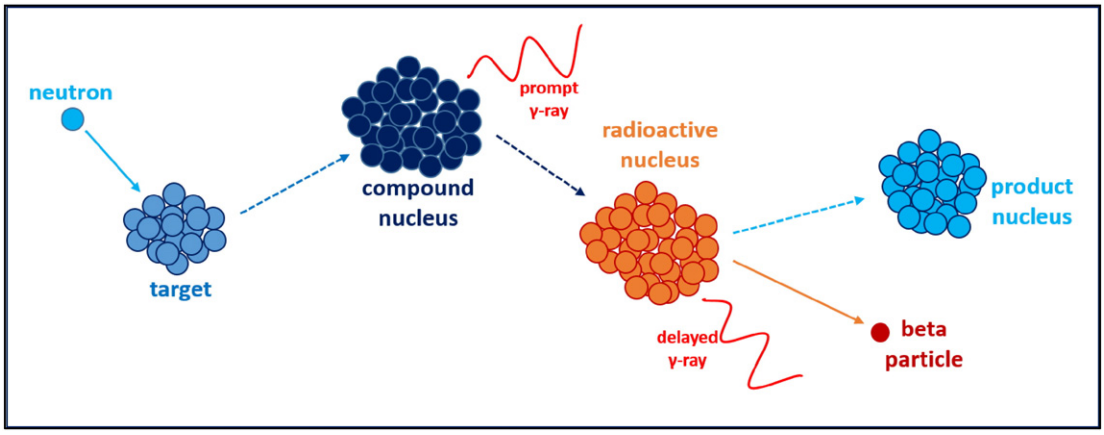

Fig. 1.3. Principle of function of NAA for elemental analysis. A target, via a non-elastic collision in the atomic nucleus, absorbs a neutron and a compound nucleus in an excited state is formed. Then the compound nucleus will de-excite into a more stable configuration through emission of one or more characteristic prompt γ-rays. The process ends, when the ground state formed during the de-excitation, is stable. This occurs, when an isotope of the same element, with a mass number increased by one, is produced.

via a non-elastic collision in the atomic nucleus, absorbs a neutron and a compound nucleus in an excited state is formed [135–146]. Then, the compound nucleus will de-excite into a more stable configuration through emission of one or more characteristic prompt γ-rays. The process ends when the ground state formed during the de-excitation, is stable; this occurs when an isotope of the same element, with a mass number increased by one, is produced. Generally, every chemical element has at least one isotope that produces a radioactive one in neutron capture. They typically emit β particles and γ radiation with a given half-life [147–155]. The energies of the prompt and delayed γ-rays allow identifying the emitter nuclide and their intensity is proportional to its amount. Neutron Activation Analysis (NAA), as also known as Instrumental Neutron Activation Analysis (INAA), Prompt-Gamma Neutron Activation Analysis (PGNAA) and Delayed Neutron Activation Analysis (DNAA), are analytical sensitive techniques useful for qualitative and quantitative multi-element analysis of major, minor, and trace elements [123–129].

Hevesy and Levi, who first noticed how samples exposed to a source of neutrons, became highly radioactive, discovered NAA in 1936 and so they apply neutron irradiation for the qualitative and quantitative

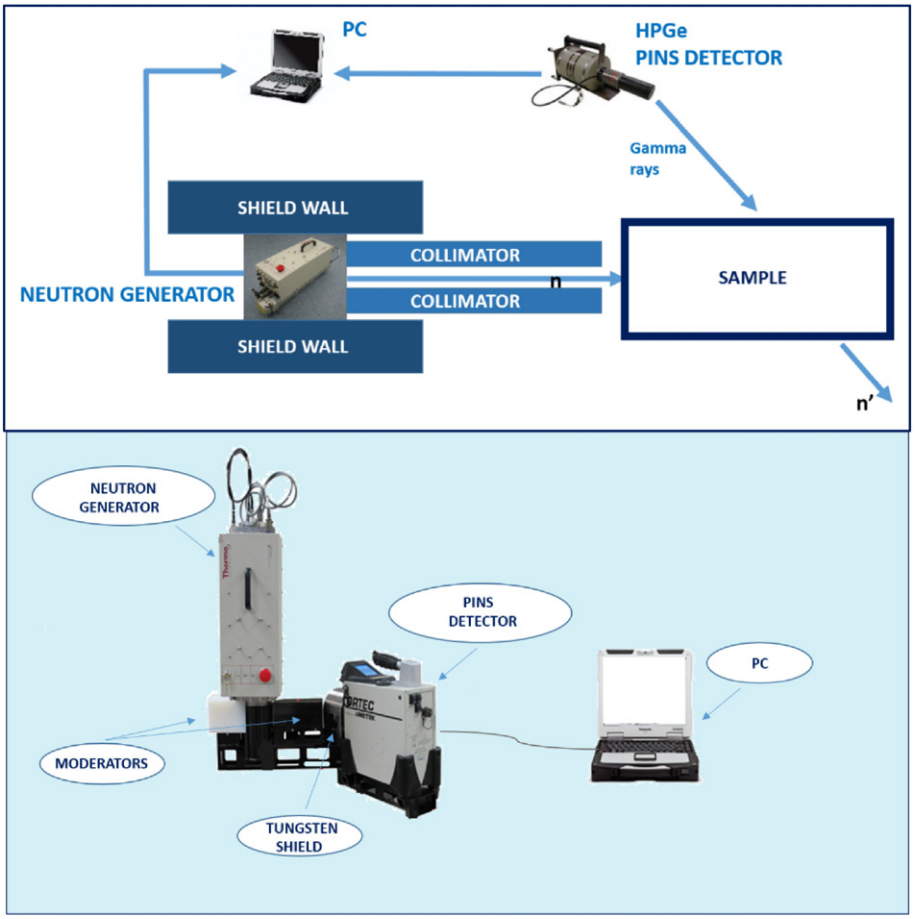

Fig. 2.1. Experimental set-up for NAA: a portable neutron source, a shield-moderator, a γ-ray spectrometer, and a computer form the experimental set-up, aligned by a fixed support.

identification of the elements present within a sample. NAA is used to determine the concentration of elements in a variety of matrices and is based upon the conversion of stable nuclei to other mostly radioactive nuclei via nuclear reactions. The nuclear reactions occur by irradiating the sample with neutrons. In particular, a neutron is captured by a target, transmuting it into an unstable nucleus, which then decays by fission or by the release of some particle or photon. NAA uses low-energy thermal neutrons to transmute a wide range of nuclei into unstable isotopes; irradiation can take many hours while measurement of the decay energies and rates of the unstable transmuted isotopes can require days. By analyzing the spectrum of prompt or delayed γ-rays and after comparing with tabulated nuclear data for γ-ray emission, one may trace back the elements of which the test sample consists. Fast NAA is also a possibility, requiring, however, considerably more energetic neutrons than a Deuterium filled DPF can produce.

The NAA reaches a detection limit as low as $1{:}10^{15}$. For this reason this technique is applied not only in a wide range of scientific applications covering as different fields of study as geology, biology or cultural heritage but it is equally interesting for quality control or environmental supervision in an industrial context. There are several types of neutron sources for NAA, such as accelerators, radio-isotopic neutron emitters, reactors and portable neutron generators.

2. Experimental set-up and data analysis for NAA with NGs

2.1. Neutron irradiation and spectroscopy set-up

A portable neutron source, a shield-moderator, a gamma-ray spectrometer, and a computer form the experimental set-up, aligned by a fixed support (Fig. 2.1) [71–87].

A neutron source is located at the front edge of a polyethylene moderator that slows down the neutrons. The source emits fast neutrons toward the sample. Fast neutrons excite nuclei by inelastic scattering, while some neutrons suffer capture reactions. A neutron generator produces one million neutrons per second omni-directionally.

A tungsten shadow shield is interposed between moderator and spectrometer, to protect germanium crystal from neutron damage. A collimator of annular bismuth surrounds the crystal, for excluding the background of gamma rays and for maximizing the signal/noise ratio of the gamma-ray spectrum.

The γ-ray detector is a Portable Isotopic Neutron Spectroscopy (PINS), a non-destructive assessment system that analyses and provides on-site identification information. The PINS spectrometer probes the investigated sample with neutrons; these excite the atomic nuclei within, producing γ-rays. The spectrum of the γ-rays energy intensity is characteristic of the chemical element. The PINS software permits to identify each element inside the sample and determines its composition. PINS uses neutrons produced by a portable deuterium-deuterium neutron generator, with a kinetic energy of approximately 2.5 MeV, to excite nuclei inside the testing sample [88–104].

The spectrometer includes a High-Purity Germanium (HPGe) detector, used to detect γ-rays emitted from the sample, a digital signal processing multichannel analyzer, an internal battery, and an electrical-powered refrigerator. The detector is connected to a computer, which acts as control panel, through an Ethernet. The PC allows the analysis of the instrument, start and stop data acquisition, such as display of the spectrum resulting and interlock for the neutron generator. The support aligns the experimental set-up in an optimal geometry for measurements. The system allows to identify chemicals inside a container without the need for disassembly, contact, physical sampling (Fig. 2.2).

2.2. NAA system

Neutrons, produced by the portable source, excite the nuclei they encounter by inelastic scattering and neutron capture; the excited nuclei decay to ground states because of γ-ray emission, and this rays

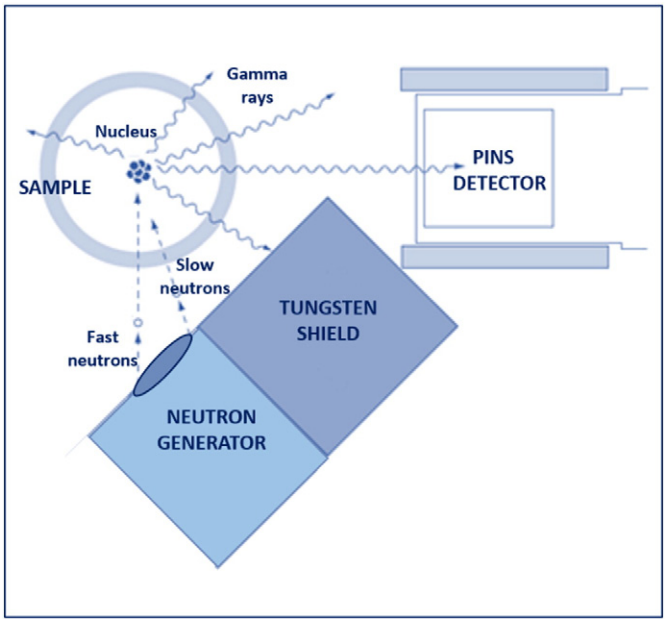

Fig. 2.2. The PINS moderator block and shadow shield aligned with the γ-ray detector.

are characteristic of the emitting nucleus. The Fig. 2.3 shows, as an example, the full energy-range PINS gamma-ray spectrum for the chlorine nucleus (Cl) with an energy of about 6 MeV. More precisely, a PINS spectrum contains many vertical peaks, which represent the identification of the chemical inside the investigated sample. The peaks are labelled with the emitting chemical element symbol and the gamma ray peak energy.

The Fig. 2.3 shows the data analysis. In general, γ-ray energy and intensity vary from one nucleus to another, such as in the case of hydrogen nucleus that emits a gamma ray at 2.2 MeV and phosphorus nucleus that emits at 1.3 MeV. The HPGe detector measures these γ-rays and displayed they in a spectrum, in which are identified the chemical elements excited by the neutrons. The PINS software records and displays the incoming spectra measurements, every 10 s, fitting the

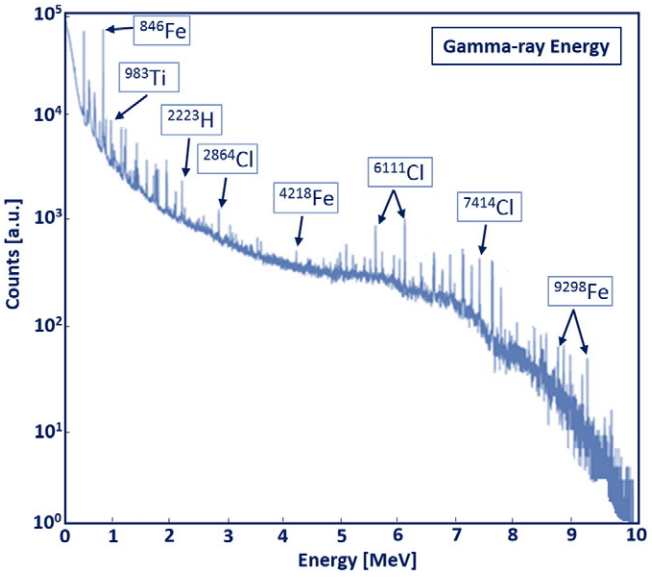

Fig. 2.3. Full energy-range PINS γ-ray spectrum for the chlorine nucleus (Cl) with an energy of about 6 MeV. The peaks, labelled with the emitting chemical element symbol and the gamma ray peak energy, represent the identification of the chemical inside the investigated sample.

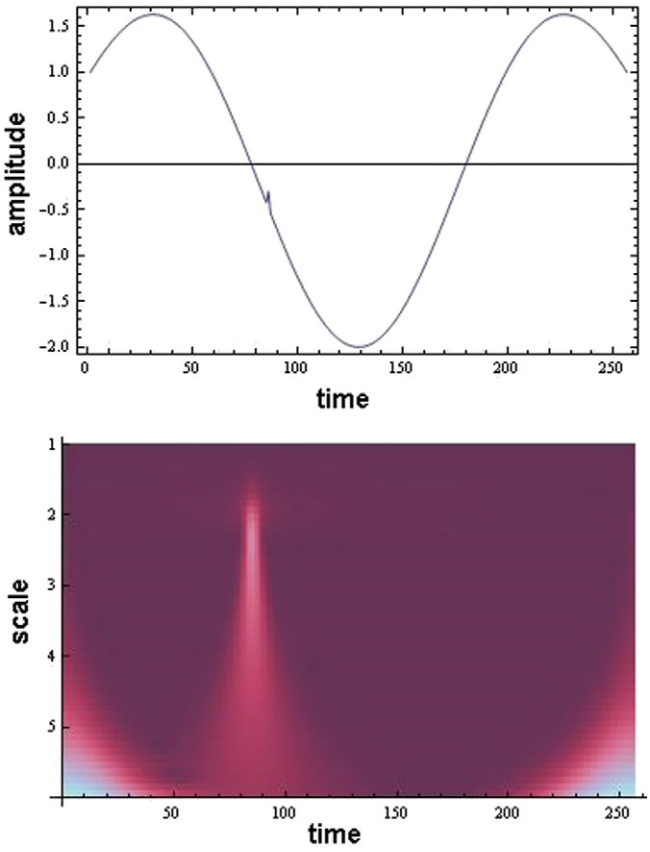

Fig. 2.4. An example of signal with a sudden spike (on the top) and its scalogram plot (on the bottom). The wavelet transform clearly reveals the spike of the signal.

peaks of interest, re-calibrating the energy scale, and performing a chemical analysis.

In order to determine the peaks in the full energy-range PINS γ-ray spectrum, i.e. the values for which "kinks" and/or anomalies in the trends occur, a wavelet method can be used.

It's well known that wavelet analysis represents an innovative and powerful tool to investigate non-stationary signal processing such as trends, drift, and, in particular, beginnings and ends of events, abrupt spectral changes and first order discontinuities which, in the framework of this work, identify with a great precision peak positions, especially in the presence of complex spectral features. Wavelet analysis has recently found a wide range of applications in various fields, such as, engineering, physics, mathematics and neutron scattering. In particular, wavelet analysis allows to perform a local analysis of the signal, to de-noise a signal without appreciable degradation and to highlight aspects of data such as aspects like trends, anomalies, discontinuities in higher derivatives, and self-similarity features.

Differently from what occurs for Fourier Transform (FT), which decomposes the signal into big waves, i.e. the sinusoids, Wavelet Transform(WT) decomposes the signal by means of shifted and scaled mother wavelet:

$$W(a,b)=\frac{1}{\sqrt{a}}\int_{-\infty}^{+\infty}(t)\psi^{*}\left(\frac{t-b}{a}\right)dt$$

where, $f(t)$ is the signal, a (being $a>0$) represents the scale or the scaling parameter that refers to the width of the wavelet; more precisely , if $|a|<1, \psi$ is compressed (high frequency), , if $|a|>1\,\psi$ is dilated (low frequencies). The parameter b is the shift or translation parameter, which take into account the time location of the wavelet, finally the term, ψ^{*} is the conjugate.

WT furnishes the *scalogram*

$$P_{W}(a,b)=|W(a,b)|^{2}$$

that represents the wavelet coefficients defining a local time–frequency energy density.

The NAA spectra can be analyzed by means of a wavelet analysis that allows to de-noising the registered signal and to identify the peak positions [201–205].

In Fig. 2.4, as an example, a signal with a sudden spike, at top, is shown; at bottom the wavelet analysis, WT reveals the spike of the signal. It is possible to notice that wavelet analysis allows decomposing the signal into shifted and scaled versions of the mother wavelet.

The Fig. 2.5 reports some particular attributes of the ^{137}Cs γ-ray peak: the centroid, the height, the width and the area. The centroid is the midpoint of count distribution of peaks; which represents the energy peak of the γ-ray, and identifies each chemical element. The peak area is calculated from the sum between the number of counts and the edge of the lower peak energy to the upper energy edge, while the net peak area from the difference between the peak area and the background area under the peak. The net peak areas are directly proportional to the mass of the chemical element present in the investigated sample. About the determination of the areas, the related uncertainties are the most critical information of a spectrum: there is a background called the Compton continuum. The blue part highlights the background area under a peak. It can be subtracted from the gross peak area to determine the net peak area. Alternatively, a Gaussian fit for the peak atop a line segment that represents the average Compton background is possible. An example is for the 137Cs that presents a peak centroid energy of 0.662 MeV. The uncertainty ratio of the net peak area is t = (826.9/45.1) = 18.3 that is certainly a real peak. In fact, as a rule, a

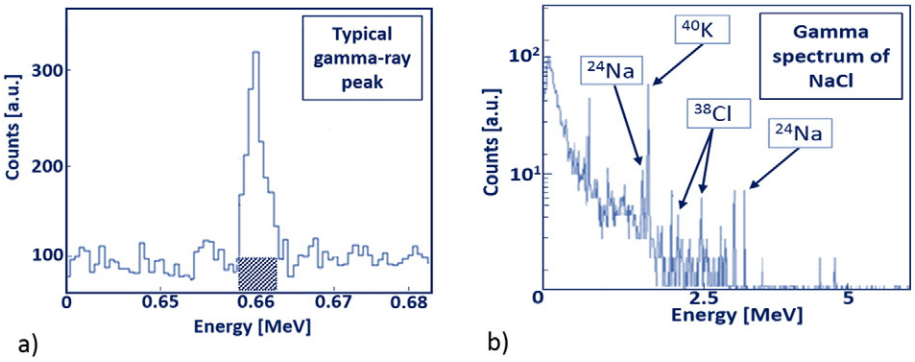

Fig. 2.5. Some relevant examples of NAA are reported. a) Typical γ-ray peak. The PINS instrument analyzes γ-ray peaks individually or in small groups. In the figure, some particular attributes of the 137Cs γ-ray peak: the centroid, the height, the width and the area, are reported. The peak centroid energy is at 0.662 MeV. b) A γ-spectrum obtained from the D-D NG with a NaCl sample irradiated by a neutron yield of ~10^{6} n/s from the D-D NG. In particular, the energy peak reports the presence of ^{24}Na, ^{40}K, ^{38}Cl in the NaCl investigated sample.

peak with a ratio of value 3 have a high statistical, while peak value major to 5 are certainly true peaks. Candidate peaks with t values <1 are likely fluctuations in the Compton background. By this test, the small spike near 0.651 MeV is a fluctuation.

Another example, that demonstrate the validity of a simple and intuitive system of elemental analysis, as well as portable too, is the gamma spectrum obtained from the D-D generator with a NaCl sample. The Fig. 2.5 b) shows the result of a γ spectra obtained with NaCl sample irradiated by a neutron yield of ~10^6 n/s from the D-D NG. In particular, the energy peak reports the presence of ^{24}Na, ^{40}K, ^{38}Cl in the NaCl investigated sample.

3. Conclusions

This work reports an overview of the new generation of portable neutron sources specifically addressed to biophysics applications. After an introduction on the importance of neutrons for the study of biophysical samples and a description of the typical experimental set-up for the analysis of biophysics samples, an overview of applications with specific reference to elementals analysis is furnished. More specifically some example of NAA results obtained by using a portable non-stationary Deuterium-Deuterium pulsed neutron generator are presented. Such device is able to generate 2.5 MeV neutrons with a neutron yield of 1.0×10^6 n/s, with a pulse rate of 250 Hz to 20 kHz, and a duty factor varying from 5% to 100%; in combination with a solid-state photon detector, such as PINS spectrometer, allows a rapid and user-friendly elemental analysis. Finally, some examples are reported.

Transparency document

The Transparency document associated with this article can be found, in online version.

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