**Experience with Delayed- and Prompt-Gamma Neutron Activation Analysis using Accelerator-based neutrons at KFUPM: An overview**

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Abstract – *Neutron Activation Analysis (NAA) is one of the major nuclear applications using neutrons. These neutrons can originate from a variety of sources, such as nuclear reactors, isotopic neutron sources, or neutron generators. The nuclear group at KFUPM has gained extensive experience in the use of fast and thermal neutrons for NAA on a variety of solid and liquid samples, with delayed and prompt gamma rays, over the past 30+ years, using both open-end and portable neutron generators. The measured concentrations ranged from a low of 6 ppm for vanadium to a high of 2.3 wt% for strontium using delayed gamma rays from activated soil samples; and from a low of 24 ppm for boron in water samples to a high of 15.8 wt% for oxygen in bulk samples for prompt gamma rays. The present paper briefly describes the salient features of the NAA setups and some of the measurements and results obtained using them. Brief information is provided while details can be found in the relevant references listed.*

**Keywords:** TNC, NIS, NAA, PGNAA, Neutron Generators

I. Introduction

Neutron Activation Analysis (NAA) with neutron generators is a well-established and widely used nuclear analytical technique **[1]**. It is a non-destructive, multi-elemental, and bulk analysis method that requires minimum sample preparation. The process of neutron activation can be achieved with a variety of neutron sources. The sources that achieve the best sensitivities for most elements are high-flux nuclear reactors. These sensitivities can reach down to lower than ppb levels. This is due to the availability of very high thermal neutron fluxes inside the nuclear reactor for use in Instrumental Neutron Activation Analysis (INAA) with delayed gamma rays. Moreover, even the extracted neutron beams have very high fluxes for use in Prompt Gamma Neutron Activation Analysis (PGNAA), albeit many orders of magnitude lower than those inside the nuclear reactor.

The neutrons that are produced at the KFUPM 350 keV ion accelerator **[2]** are fast neutrons with approximately 2.5 MeV and 14 MeV energies. These neutrons were thermalized using a thick moderator as illustrated in **Fig. 1**. Commercial, portable neutron generators at KFUPM produce neutrons of 2.5 MeV energy **[3]**, while a 16 Ci Am-Be isotopic ion source produces neutrons in a wide energy range, with an average of about 4.2 MeV **[4]**. The experience gained in the many aspects of nuclear instrumentation including optimization as well as data acquisition and analysis using neutron generators is directly relevant to the case when the neutrons originate from nuclear reactors instead.

During the process of neutron irradiation of a target nucleus of mass number *A* and atomic number *Z*, , the following processes are likely to take place **[5,6]**:

1. For fast neutrons used in Neutron Inelastic Scattering (NIS), the following process takes place

where is a neutron of lower energy than that of the incident neutron , is an excited state of the target nucleus, and are prompt gamma rays emitted during the transition of to its ground state .

1. For thermalized neutrons used in Thermal Neutron Capture (TNC) reactions, the following two process may take place:

a.

where is an excited state of the compound nucleus, and are prompt gamma rays emitted during the transition of to its ground state . If the ground stateof the compound nucleus is stable, the process ends here.

1. However, if the ground state of the compound nucleus is unstable, a beta decay occurs with a specific half-life :

where is the daughter nucleus in an excited state, is an electron, is an anti-neutrino of the electron type, and are called delayed gamma rays. The beta decay process happens through weak interactions, and the associated half-lives span a very wide range, from seconds to years. The number of measured delayed gamma rays is governed by the half-life of the parent nucleus .

The prompt and delayed gamma rays are characteristic of the activated elements and are used to uniquely identify them, while their intensities are used to estimate the elemental concentrations in the activated samples.

**II. Experimental Setups**

**II.A. Delayed-Gamma NAA (NAA)**

**II.A. 1. Irradiation Station**

The Delayed-Gamma NAA Facility at KFUPM uses neutrons from the 350-keV ion accelerator or neutrons from the 16 Ci Am-Be isotopic source. Fast, monoenergetic neutrons are produced with about 2.5 MeV and 14 MeV energies in the D(d,n)3He and T(d,n)4He reactions, respectively.The neutrons from the Am-Be source are produced when an alpha particle emitted by the 241-Am radioisotope reacts with a 9Be nucleus to produce a neutron and a 12C nucleus in an excited state. The nominal output of such sources is 2.2 x 106 n/s for each 1 Ci of activity. Its half-life is 432 years and the average neutron energy is 4.2 MeV with a maximum of about 11 MeV **[4]**.

**II.A.2. Thermalization of fast neutrons**

Fast neutrons from the 350-keV ion accelerator first pass through a 1 cm-thick water layer, then through a 0.5 cm-thick stainless-steel water jacket casing, and are finally slowed down in a cylindrical moderator. The maximum thermal-neutron flux achieved with the D(d,n)3He reaction was 2.5 x 106 n/cm2-s and that achieved with the T(d,n)4He reaction was 3 x 107 n/cm2-s **[7]**. A drawing of a typical setup for Neutron Activation using Thermal Neutron Capture (TNC) with neutrons from the 350 keV ion accelerator. On the other hand, a paraffin sphere is used to moderate fast neutrons from the Am-Be source. The maximum thermal neutron flux achieved with the Am-Be neutron source was 2.7 x 104 n/cm2-s **[4]**.



*Fig. 1. Drawing of typical setup for Neutron Activation with delayed gamma rays in Thermal Neutron Capture using the 350 keV ion accelerator*

**II.A.3. Counting station**

The counting station was housed in a separate room that is about 60 m away from the irradiation station. It was based on a heavily shielded HPGe detector with its associated electronics and a PC-based data acquisition and analysis system with 4096 channels. The resolution of the detector is about 2 keV at the 1.33 MeV line of 60Co. The system is calibrated using standard γ-ray sources such as 22Na, 137Cs, and 60Co. The data acquisition and analysis were carried out using the MAESTRO software package provided by EG&G Ortec. More details on the counting station can be found in **[4]**.

**II.A.4. Applications**

***Vanadium determination in phosphate samples***

Economically viable phosphate deposits have been found in northwestern Saudi Arabia **[8,9]**. Phosphate deposits usually contain important trace elements such as vanadium and rare earth elements **[10]**. Such elements can be exploited, with existing technology, as by-products from the commercially viable phosphate deposits, and can help in the interpretation of the genesis and diagenesis of these phosphate deposits **[11]**. The accelerator-based thermal neutrons at the KFUPM NAA facility were used to analyze ten phosphorite samples collected from the phosphate deposits in northwestern Saudi Arabia. The nuclear data of vanadium relevant to the present analysis are listed in **Table 1**, along with the data of other studied elements, all of which were measured using delayed gamma rays.

*Table 1. Nuclear data of elements determined in accelerator-based thermal-neutron activation analysis.*

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| TargetIsotope | Abund.[%] | σth[barns] | Product Isotope (T1/2) | Eγ[keV] | Intensity[%] |
| 23Na | 100 | 0.513 | 24Na (15.0 h) | 1369 | 100 |
| 26Mg | 11.0 | 0.037 | 27Mg (9.46 m) | 1014 | 28.6 |
| 27Al | 100 | 0.226 | 28Al (2.24 m) | 1779 | 100 |
| 37Cl | 24.2 | 0.423 | 38Cl (37.2 m) | 1643 | 31.0 |
| 41K | 6.73 | 1.45 | 42K (12.4 h) | 1525 | 18.8 |
| 51V | 99.8 | 4.79 | 52V (3.75 m) | 1434 | 100 |
| 55Mn | 100 | 13.2 | 56Mn (2.58 h) | 847 | 98.9 |
| 86Sr | 9.86 | 0.770 | 87Sr (2.81 h) | 388 | 82.3 |
| 138Ba | 71.7 | 0.405 | 139Ba (84.6 m) | 166 | 22.1 |
| 164Dy | 28.2 | 2730 | 165Dy (2.33 h) | 94.7 | 3.58 |
| 185Re | 37.4 | 106 | 186Re (90.6 h) | 137 | 8.50 |
| 187Re | 62.6 | 73.2 | 188Re (17.0 h)188mRe (18.6 m) | 155106 | 14.910.8 |

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The results of the analysis are shown in **Table 2**. The errors were mainly due to counting statistics. The determination of vanadium concentration in the various phosphate samples was taken as the average of three independent measurements **[12]**.

*Table 2. Vanadium concentrations in the phosphate samples as determined by accelerator-based thermal-neutron activation analysis.*

|  |  |  |
| --- | --- | --- |
| SampleNo. | Mass[g] | Vanadium[ppm] |
| 1 | 21.8 | 457 ± 13 |
| 2 | 56.7 | 295 ± 6 |
| 3 | 89.6 | 197 ± 4 |
| 4 | 91.1 | 87 ± 3 |
| 5 | 109 | 69 ± 3 |
| 6 | 93.4 | 29 ± 2 |
| 7 | 42.1 | 28 ± 2 |
| 8 | 39.4 | 18 ± 2 |
| 9 | 8.69 | 77 ± 16 |
| 10 | 109 | 23 ± 1 |

***Determination of rhenium in Pt-Re/Alumina reforming catalysts***

In addition to platinum, rhenium is commonly used in reforming catalysts in the petroleum refining industry because of its high activity, selectivity, and long lifetime. Both calcined and uncalcined Pt-Re/Al catalyst samples should be analyzed and characterized using multiple analytical techniques to understand their physical characteristics. The accelerator-based thermal neutron flux at the KFUPM NAA facility was used to irradiate several calcined and uncalcined Pt-Re/Al catalyst samples of (0.5% Pt + 0.5% Re)/Al, 0.2% and 0.8% Re/Al. Table 1 shows the relevant nuclear data for the determination of Re. The minimum detection limit for Re was 35 µg [13].

 ***Elemental analysis of local soil samples***

Due to the importance of soil composition in agriculture, it was deemed useful to carry out an elemental analysis of a selected number of local agricultural soil samples in order to assess the capability of the KFUPM NAA facility for the elemental analysis of such samples. The soil samples used in this investigation were collected from Al-Hofuf area in eastern Saudi Arabia. The elements determined in the soil samples were: K, Mn, V, Al, Na, Cl, Ba, Sr, and Mg. **Table 1** lists the relevant nuclear data for the elements determined in this analysis. The minimum, maximum, average, and standard deviation of the absolute concentrations of the elements determined in the local agricultural soil samples are listed in **Table 3** **[14]**.

*Table 3. Minimum and standard deviation of absolute concentrations of the elements determined in soil samples using delayed gamma rays from accelerator-based thermal-neutron activation analysis.*

The table is added below

***II.B. Prompt-Gamma NAA (PGNAA)***

The Prompt-Gamma Neutron Activation Analysis (PGNAA) setups at KFUPM use either 2.5 MeV or 14 MeV neutrons from the 350 keV ion accelerator, or 2.5 MeV neutrons from the portable neutron generators for Neutron Inelastic Scattering (NIS). They also use polyethylene moderators to thermalize 2.5 MeV neutrons from the portable neutron generators for TNC studies. At one time or another, the scintillation detectors used in PGNAA measurements consisted of Sodium Iodide (NaI), Bismuth Germanate (BGO), Lanthanum Tri-Bromide (LaBr3), Lanthanum Tri-Chloride (LaCl3), or Cerium Tri-Bromide (CeBr3) cylindrical detectors. In all experiments the scintillation detectors were shielded against direct neutrons and scattered gamma rays. Nonetheless, they underwent some radiation damage. A typical arrangement of the activation setup is shown in Fig 2. An essential ingredient in the use of PGNAA is an up-to-date and accurate knowledge of the data tables of the multiple gamma ray lines of the various elements of interest **[15]**. Another important, if not essential, component for the efficient use of PGNAA are the Monte Carlo simulation codes that are used to optimize the setups, most notably MCNP **[16-19].**



*Fig. 2. Typical activation setup used in PGNAA using Neutron Inelastic Scattering with 2.5 MeV neutrons*

***II.B.1 Applications***

PGNAA has been extensively used at KFUPM for the detection and measurement of a large number of elements, particularly for environmental applications. The technique is useful for the measurement of major concentrations of most elements, including few light elements such as carbon, and oxygen. **Table 4** lists the studied elements using TNC-NAA. **Table 5** lists the elements studied using NIS-NAA with 2.5 MeV neutrons, and **Table 6** lists the elements studied using 14 MeV NIS-NAA. The performance of the PGNAA setup is indicated by the Minimum Detectable Concentrations (MDC) and their uncertainties (σMDC) for the studied elements.

*Table 4. Elements studied with PGNAA using TNC.*

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Element | sample | Energy[keV] | MDC ± σMDC[wt%] | Ref. |
| Ni | Soil | 8553 | 0.79 ± 0.24 | [20] |
| Ni | Soil | 8998 | 1.27 ± 0.39 | [20] |
| S | Soil | 5420 | 1.3 ± 0.4 | [23] |
| Cl | Water | 517 | 0.08 ± 0.02 | [24] |
| Cl | Water | 786-788 | 0.07 ± 0.02 | [24] |
| Cl | Water | 1165 | 0.18 ± 0.05 | [24] |
| Cl | Water | 1951/1959 | 0.11 ± 0.03 | [24] |
| Cl | Water | 2863 | 0.17 ± 0.05 | [24] |
| Cl | Water | 6111 | 0.13 ± 0.04 | [24] |
| Cl | Water | 6619 | 0.18 ± 0.05 | [24] |
| Cl | Water | 8578 | 0.8 ± 0.24 | [24] |
| B | Water | 478 | 24.4 ± 7.43 ppm | [25] |
| Cd | Water | 245-651 | 95.6 ± 29.1 ppm | [25] |
| Hg | Water | 368 | 0.15 ± 0.05 | [25] |

*Table 5. Elements studied with PGNAA using NIS of 2.5 MeV neutrons.*

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Element | sample | Energy[keV] | MDC ± σMDC[wt%] | Refe |
| Ni | Soil | 1331 | 2.00 ± 0.70 | [20] |
| Ni | Soil | 1454 | 1.79 ± 0.55 | [20] |
| Cr | Soil | 1430 | 0.85 ± 0.26 | [21] |
| Ti | Soil | 984 | 0.68 ± 0.18 | [21] |
| Zn | Soil | 1005 | 1.53 ± 0.47 | [21] |
| P | TSP | 2230 | 0.55 ± 0.17 | [22] |
| Na | TSP | 438 | 0.79 ± 0.24 | [22] |
| Na | NaCl | 438 | 0.73 ± 0.22 | [22] |
| P | TSP | 2230 | 0.37 ± 0.11 | [22] |
| Na | TSP | 438 | 0.31 ± 0.09 | [22] |
| Na | NaCl | 438 | 0.35 ± 0.11 | [22] |
| S | Mg-SO4 | 2240 | 0.56 ± 0.17 | [22] |
| S | Soil | 2240 | 0.68 ± 0.21 | [22] |

*Table 6. Elements studied with PGNAA using NIS of 14 MeV neutrons.*

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Element | sample | Energy[keV] | MDC[wt%] | σMDC[wt%] | Ref. |
| C | bulk | 4439 | 12.2/3.2 | 3.8/1.0 | [26] |
| O | bulk | 6130 | 15.8/x | 4.8/x | [26] |

The first number for the MDC and σMDC corresponds to the LaBr3 detector while the second number after the slash sign corresponds to the BGO detector. Notice that Oxygen cannot be analyzed using the BGO detector because of the large presence of Oxygen in the detector material.

III. Conclusions

The experience gained at KFUPM in NAA and PGNAA using TNC and NIS with 2.5 MeV and 14 MeV neutrons from the 350 keV ion accelerator and the portable neutron generators shows that, even though the available neutron fluxes are relatively low, they nonetheless can be advantageously used to carry out useful analyses of interest to academic research, various industries, as well as environmental studies, when coupled with scintillation detectors with high resolution and good efficiency. The analyses can be optimized by judicious use of simulation codes and data reduction software. The results can definitely be much improved by having higher neutron fluxes and using appropriate electronics to suppress the continuous Compton background in the scintillation detectors at low gamma energies. The valuable experience gained at KFUPM in NAA with both delayed and prompt gamma rays using neutron generators over the past 30+ years is very relevant to the applications program of nuclear reactors.

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NAA: Neutron Activation Analysis

TNC: Thermal Neutron Capture

NIS: Neutron Inelastic Scattering

PGNAA:Prompt Gamma Neutron Activation Analysis

TSP: Sodium triphosphate

MDC: Minimum Detectable Concentration

σMDC: Error on MDC

σth: Thermal Capture Cross Section.

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| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Element→ | K[wt%] | Mn[ppm] | V[ppm] | Al[wt%] | Na[ppm] | Cl[ppm] | Ba[ppm] | Sr[wt%] | Mg[ppm] |
| Minimum | 0.6 | 126 | 6.0 | 1.2 | 2490 | 352 | 349 | 0.4 | 3130 |
| Maximum | 1.1 | 165 | 26 | 2.1 | 3900 | 5090 | 534 | 2.3 | 6876 |
| Average | 0.8 | 151 | 11 | 1.6 | 3090 | 1490 | 436 | 1.5 | 5327 |
| Std. Dev. | 0.2 | 18.0 | 10 | 0.4 | 603 | 1640 | 63.0 | 0.7 | 1195 |

Table 3