



Article Improving a PGNAA Technique to Detect Heavy Metals in Solid Samples

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Abstract: Prompt gamma-ray neutron activation analysis (PGNAA) is a useful approach for determining the concentrations of a variety of elements in natural materials, either online or in situ, without affecting their chemical forms in matter. The current research aimed to improve the yield of a portable PGNAA setup using a dc beam of 2.5 MeV neutrons and a CeBr₃ detector to record gamma rays from neutron inelastic scatterings. It is impossible to avoid the superimposition of heavy metal gamma rays and those from the detector's element. However, tests were carried out to improve the signal-to-background ratio. By assessing the minimum detectable concentrations (MDC) of chrome, titanium, and zinc in soil samples, the effectiveness of the new optimization was confirmed. The study shows an improvement in the MDC values.

Keywords: CeBr₃ detector; 2.5 MeV neutrons; neutron inelastic scattering; minimum detection concentrations

1. Introduction

Several analytical methods have been developed to determine the presence of hazardous metals and their concentration levels in environmental samples, including prompt gamma-ray neutron activation analysis (PGNAA). The PGNAA technique's key advantage was its capacity to accommodate relatively large bulk samples without incurring fees for chemical preparation, losing volatile components, or changing the materials' original phase. It has been used to determine the chemical composition of materials based on their neutron interaction probability. Only elements with large neutron cross-sections release visible gamma rays, which can happen through thermal neutron capture (TNC) or neutron inelastic scattering (NIS).

PGNAA approaches are now being developed to increase heavy metal detection performance in a variety of sectors [1], including anthropology [2], health sciences [3], mining industries [4], homeland security [5], and even landmine detection [6]. The rapid rise of modern lives and technology necessitates a high level of heavy metal consumption. Exposure to these metals in the soil, whether direct or indirect, can result in unintended discharging or deposition of pollutants [7], which can happen by volatilization, leaching, erosion, or suspension of soil particles [8]. As a result, pollutant exchange between soil, water, and air will spread pollution from agricultural soil to the food chain. The effects on mankind of eating food cultivated in contaminated soil are a topic of concern [9–14].

Although PGNAA is commonly used to identify and quantify chemical elements in aqueous samples or solid matrices such as soil and rocks [15–18], it suffers from a signal-to-background ratio problem, which affects its ability to recognize the components present in minute concentrations in a sample. This is usually the case when the neutron flux is



Citation: AlMisned, G.; Al-Abdullah, T.; Liadi, F.; Hawsawi, A. Improving a PGNAA Technique to Detect Heavy Metals in Solid Samples. *Appl. Sci.* 2022, *12*, 3714. https://doi.org/ 10.3390/app12083714

Academic Editors: Maria Gavrilescu and Jose Antonio Rodriguez Martin

Received: 7 March 2022 Accepted: 4 April 2022 Published: 7 April 2022

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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). modest or diffuse. Recent research revealed that identifying the silicon and phosphorus gamma-ray lines following the NIS reaction was difficult because their peaks were formed on wide continuum slopes [19]. Another issue arises from the activation of the detector's components, which add the dominant detector's peaks to the spectrum, sweeping the peaks from the sample under activation. Two recent studies [20,21] employing the portable neutron generator Genie 16 reported strong detector peaks following 2.5 MeV neutron irradiation of soil samples. Both investigations used two separate reaction processes to measure sulfur contents in the soil. For energies below 1.3 MeV, the first study used the NIS technique, and the acquired spectra using the CeBr₃ detector display multiple strong gamma-ray peaks from cerium (Ce) and bromine (Br) components [20]. Figure 1 depicts several of these peaks as well. Using a bismuth germanate (BGO) detector, the TNC reaction was used in the other investigation to observe the sulfur peak at 5.42 MeV. Similarly, the recorded spectrum revealed two strong BGO peaks in the 6–8 MeV energy range [21].



Figure 1. Four prompt gamma-ray spectra were collected from an empty sample container and a CeBr₃ detector. These spectra were related to the direct background (BkGd), background with a moderator before the detector (BkGd-Mod), background with angling the neutron generator tube (BkGd-Angle), and background with angling the neutron generator tube and adding a moderator directly before the detector (BkGd-Angle-Mod).

The current research proposed a new optimization for the PGNAA setup located at King Fahd University of Petroleum and Minerals (KFUPM). The optimization aimed to improve the signal-to-noise ratio by adjusting the setup to directly impact both the signal and background simultaneously. This achievement would allow us to observe the prompt gamma rays from specific elements upon neutron activation, assuming that the gamma-ray peaks overlap with the detector's peaks. As a result, we sought to improve the setup's measurement performance. Three groups of contaminated soil samples were created to demonstrate the quality of the new setup's adjustment. Each group represents a different element that has been blended with the soil in various amounts. Following the NIS reaction, chrome (Cr), zinc (Zn), and titanium (Ti) generate measurable gamma rays at 1.43 MeV, 1.005 MeV, and 0.984 MeV that coincide with Br peaks at 0.97 and 1.00 MeV and

Ce peaks at 1.35 MeV. These elements were chosen to demonstrate the effectiveness of the new optimization for the existing setup, as well as its suitability for determining the lowest level of elemental detection in solid samples.

Adopting PGNAA to detect Cr, Ti, and Zn in solid samples by applying fast neutron activation is rare. Available data were reported a few decades ago [22,23], and their achievements regarding the above elements will be reviewed in the results and discussion section below. Currently, exploring the resources near the earth's surface requires developing the PGNAA technique for borehole logging purposes, where information is needed to investigate the rocks or soils surrounding the hole. In this vein, the majority of recent articles [24,25] have focused on Monte Carlo simulations. Hence, measurements employing portable PGNAA setups will provide important information about the technique's quality.

2. Experimental

2.1. Optimization

The PGNAA setup used for the NIS measurements is well-described in Ref. [20]. In a nutshell, the setup consists of the Genie 16 portable neutron generator, which produces 2.5 MeV neutrons in a deuterium-deuterium (DD) reaction with a maximum intensity of 4.7×10^7 n/s. As suggested by the manufacturer, the dc deuteron beam is operated at 70 keV, and its current is set at 50 μ A [26]. The gamma rays were observed using a CeBr₃ detector. The signals from the detector were finally sent to a multi-channel analyzer coupled to a PC. The data were recorded, monitored, and analyzed using ScintiVission software.

In the previous work [20], the sample container was placed upright and in contact with the neutron target plane, and the detector faced the container such that its longitudinal axis was along the target plane of the neutron generator, and it was also touching the container. Hence, neutrons that traveled through the sample would hit the detector vertically at zero angle. Despite the fact that this increased the probability of the NIS reaction and the amount of observed gamma-ray lines, it also substantially activated the detector's elements since the alignment allowed neutrons to travel through the detector directly after leaving the sample. The sample volume inside the container was large enough to cover the front surface of the detector and the side surface of the generator tube surrounding the target plane to limit direct contact.

As illustrated in Figure 1, the new optimization began with an empty sample to capture the background spectrum, which reflects neutron activation of the detector's material. The neutrons were then delayed by sandwiching a thin (1.0 cm thick) moderator between the empty sample and the detector, which treats the spectrum as a background moderator. The background spectrum showed a significant drop, by over 25%. The system was then re-aligned, as shown in Figure 2, with just the neutron-generating tube tilted by 30° to reduce the number of neutrons penetrating the sample and arriving at the detector. Finally, two tests were run, one without the moderator (background-angle) and the other with the moderator in place (background-angle-moderator). Both cases demonstrated further reductions in the background, with just minor differences between them.

A pure titanium sample replaced the empty container in Figure 2 to explore the effects on the signal-to-noise ratio, and prompt gamma-ray spectra were obtained for each circumstance described above. Table 1 summarizes the study's findings, which reveal a nearly 100 percent increase in net counts when the neutron generator tube is tilted by 30 degrees and a moderator is introduced between the sample and the detector.

The thickness of the moderator and the orientation of the generating tube were also put to the test. The background was reduced, and the signal-to-noise ratio was improved by increasing the tube's angle. However, this required a lengthier period of measurement, almost a few hours, to obtain accurate statistics. Increasing the thickness of the moderator to more than 1.0 cm, on the other hand, lowers the net count rates. Considering the relatively small flux of Genie 16 in comparison with strong neutron sources such as Am-Be source, ²⁵⁶Cf source, or neutron accelerator, which could be at least two orders of magnitudes greater, in addition to the lifetime of the deuterium target for Genie 16, the most optimum



and appropriate situation for our PGNAA setup that provided the maximum net counts was chosen to perform several NIS reactions, which are discussed in the next section.

Figure 2. Schematic representation of the PGNAA setup used for neutron inelastic scattering reaction built around Genie 16. Figure is not drawn to scale.

Optimization	Background	Gross Counts	Net Counts
Zero Angle, No Moderator	140,057	144,589	4532
Zero Angle, Moderator	117,024	123,384	6360
30° Angle, No Moderator	113,087	119,939	6852
30° Angle, Moderator	107,019	116,674	9655

Table 1. Optimizations of Genie 16 setup in order to obtain the best net counts for a pure Ti sample.

2.2. Sample Preparations

Soil samples contaminated with different amounts of Cr, Ti, and Zn elements were separately prepared to measure the prompt gamma rays emitted from each element following neutron activations with fast neutrons via NIS reactions. A pure powder of each element, with a grain size of around 0.149 mm, was mixed well with a dry sandy soil of size 0.255 mm and then stored dry in sealed polyethylene terephthalate (PET) bottles. In order to keep the whole mass of each sample at 650 g, the amount of the element in the combination was increased, and the amount of soil was reduced. Thus, the concentrations of the elements in the soil were as follows. Zn: 4.57, 7.62, 10.67, and 15.24 wt%; Ti: 3.66, 5.18, and 6.40 wt%; and Cr: 2.23, 3.71, 5.20, and 7.43 wt%. The soil itself contained mostly silicon dioxide with 88.0 wt% of Si concentration. The spectrum labeled BkGd-Angle-Mod in Figure 1, which is related to the pure soil sample, contributed only a silicon peak at 1.78 MeV as compared to the empty container (BkGd) spectrum. As a result, it was projected that the soil would be deficient in nutrients. The spectrum obtained from a pure soil sample was used as a background. Each sample was irradiated with neutrons in real time for 2000 s.

2.3. CeBr₃ Detector

A 75 mm diameter \times 75 mm height CeBr₃ gamma-ray detector was chosen to observe the gamma rays. The detector was calibrated using 570, 1063, and 1770 keV gammaray lines emitted from a ²⁰⁷Bi radioactive source. The energy resolution of the detector varies from 4.1% for 662 keV to 3.0% for 1770 keV, as determined in Ref. [27]. Although the moderator inserted before the detector was narrow, its finite efficiency partially thermalized the neutron flux after the sample. This explains the hydrogen peak at 2223 keV in Figure 1, as well as the additional gamma rays from bromine, cerium, and iron, the last of which is present in the table that supports the entire arrangement. Finally, lead bricks were used to shelter the detector from various background radiations.

3. Results and Discussions

Figures 3–5 show the normalized pulse height spectra of the CeBr₃ detector from heavy metals contained in soil samples. The characteristic gamma rays of Cr at 1.43 MeV were clearly observed, whereas those of Ti and Zn at around 1.00 MeV were being built upon the Br peaks at 0.97 and 1.022 MeV. The detector's resolution at 1.0 MeV is estimated to be around 30 keV. This clarifies the strong overlap between the two bromine peaks and the zinc or the titanium peak. Hence, the lack of CeBr₃ capabilities to differentiate between these peaks increases the statistical error in determining the minimum detection concentration (MDC). On the other hand, when the spectra of each element are superimposed as a function of their concentration in the soil, the elemental peak displays an increase in gross counts that is proportional to its abundance in the samples. Furthermore, as shown in Figure 3, the neutron flux that activated the Si and its measured gamma rays at 1.78 MeV showed a gradual decline in gross counts, which is indirectly proportional to the heavy metal concentration.



Figure 3. Prompt gamma-ray pulse height spectrum of soil samples containing 2.23, 3.71, 5.20, and 7.43 wt% Cr superimposed upon background spectrum using CeBr₃ detector. The spectrum of a 250 g pure Cr sample overlays the other spectra.

The integrated net counts under the chrome's peak at 1.43 MeV were determined by subtracting the uncontaminated soil background spectrum from the gross counts under each soil-Cr sample shown in Figure 3. The net counts under the 0.984 MeV titanium and 1.005 MeV zinc peak regions were obtained using a similar approach. The net and background counts were then used to calculate the MDC using the relation reported in [28,29];

$$MDC = 4.653 \frac{C}{P} \sqrt{B}$$
(1)

where *P*, *C*, and *B* were related to the net counts, element's concentration in (wt%), and the associated background counts, respectively, whereas the standard deviation (σ_{MDC}) is given by

$$\sigma_{\rm MDC} = \frac{C}{p} \sqrt{2B} \tag{2}$$

Figures 6–8 show the calibration curves for the analyzed elements and the MDC resulting from their characteristic gamma-ray energies using our PGNAA setup are presented in Table 2. In addition to the standard deviation error in the value of the MDCs, the error raised from fitting the data in order to compute the slope $\begin{pmatrix} C \\ P \end{pmatrix}$ was calculated using the inverse variance weighting scheme [30]. For the case of titanium, this scheme added around \pm 0.05 wt% to the standard error. Slightly larger amounts were found for Cr and Zn. Thus, the main error in determining the MDC is mostly due to the standard deviation error σ_{MDC} .



Figure 4. Prompt gamma-ray spectra of three soil samples containing 3.66, 5.18, and 6.40 wt% Ti superimposed upon background spectrum using CeBr₃ detector. The spectrum of a 100 g pure Tir sample overlays the other spectra.



Figure 5. Prompt gamma-ray spectra were acquired by the $CeBr_3$ detector, obtained from the background spectrum and superimposed upon four samples containing zinc with the following concentrations, 3.66, 7.62, 10.67, and 15.24 wt%.



Figure 6. The normalized net counts of 1.43 MeV prompt gamma rays of chrome powder added to pure soil samples in different concentrations. The solid line represents the best fit for the data using the inverse variance weighting scheme.



Figure 7. The yields of titanium prompt gamma-ray at 0.984 MeV as a function of its concentration in soil samples. The solid line represents the best fit for the data using the inverse variance weighting scheme.

In this study, the MDCs for Cr, Ti, and Zn were determined to be 0.85 ± 0.26 wt%, 0.68 ± 0.18 wt%, and 1.53 ± 0.47 wt%, respectively. When compared to the previously discussed values in Jiggins [22] and Yates [23], the following can be summarized: 3.29 MeV neutrons were created by the DD reaction in Jiggins and Habbani's experiment, which used a 400 kV Van de Graaff machine and a NaI detector to measure the prompt gamma rays [22]. The high beam current of 100 A and the activation time of 1430 s were the two key advantages of their work. This current intensity was two times that of Genie 16's beam current, having a considerable impact on the NIS cross-section. On the other hand, in Yates et al.'s work, fast neutrons at 2.5 MeV were produced using a DD reaction, and the prompt

gamma rays were recorded using a Ge(Li) detector [23]. The high resolution of the detector allowed the gamma rays of the heavy metals to be well-characterized in their spectra. Our MDC results indicated a better value for Cr, a fairly comparable value for Zn, and a somewhat worse value for Ti, all within the uncertainty of the Genie 16 setup. Overall, the current findings show that the new setup's optimization resulted in a significant increase in MDC values.



Figure 8. The yields of zinc prompt gamma ray at 1.01 MeV as a function of its concentration in soil samples. The solid line represents the best fit for the data using the inverse variance weighting scheme.

Table 2. The measured MDC values for Cr, Ti, and Zn in soil samples using 2.5 MeV neutron inelastic scattering prompt gamma rays.

Element	E_{γ} [MeV]	(MDC $\pm\sigma_{ m MDC}$) [wt%]		
		Current Study	Jiggins [22]	Yates [23]
Cr	1.430	0.85 ± 0.26	0.99	1.20
Ti	0.984	0.68 ± 0.18	0.47	0.54
Zn	1.005	1.53 ± 0.47	1.21	1.70

4. Conclusions

The signal-to-noise ratio in gamma-ray spectra has been improved using a new optimization for a PGNAA setup consisting of the portable neutron generator Genie 16 and the CeBr₃ detector. The improvement was achieved by rotating the generator 30° with respect to the detector's axis of symmetry and then adding a 1 cm-thick moderator in the front of the detector. The new setup minimizes the flux of fast neutrons arriving at the detector after passing through the material, which has two major benefits. In the first, we were able to cut the sample size and lower the neutron activation period to nearly half of what we had previously utilized. As a result, we used less heavy metal powder, and Genie 16's life was extended. The other major benefit was improved sensitivity for identifying low concentrations of heavy metals in solid samples, especially when the heavy metals' prompt gamma rays were superimposed on those from the detector. The MDC values for chrome, titanium, and zinc in soil samples were measured to demonstrate this advantage. The MDC $\pm \sigma_{\text{MDC}}$ values obtained for Cr, Ti, and Zn are, respectively, 0.85 \pm 0.26, 0.68 \pm 0.18, and 1.53 \pm 0.47 wt% and are in good agreement with the previously reported values. These data demonstrate the reliability of the PGNAA technique in borehole logging, particularly when drilling and recovering material from the ground to the surface may cause a material disturbance.

Author Contributions: Conceptualization, T.A.-A. and G.A.; methodology, T.A.-A.; validation, T.A.-A. and G.A.; formal analysis, T.A.-A. and F.L.; investigation, T.A.-A. and G.A.; resources, G.A. and A.H.; data curation, T.A.-A., F.L. and A.H.; writing—original draft preparation, T.A.-A.; writing—review and editing, T.A.-A.; supervision, T.A.-A.; project administration, T.A.-A. All authors have read and agreed to the published version of the manuscript.

Funding: This research project was funded by the Deanship of Scientific Research, Princess Nourah bint Abdulrahman University, through the Research Funding Program, Grant No (FRP-1440-18).

Institutional Review Board Statement: This study did not require ethical approval.

Informed Consent Statement: This study did not involve human.

Data Availability Statement: The data of this study will be provided upon request.

Conflicts of Interest: The authors declare that there are no conflict of interest.

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