Research Article

Nitrogen Detection in Bulk Samples Using a D-D Reaction-Based Portable Neutron Generator

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Received 23 October 2012; Accepted 13 December 2012

Academic Editor: Paola Luches

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Nitrogen concentration was measured via 2.52 MeV nitrogen gamma ray from melamine, caffeine, urea, and disperse orange bulk samples using a newly designed D-D portable neutron generator-based prompt gamma ray setup. In spite of low flux of thermal neutrons produced by D-D reaction-based portable neutron generator and interference of 2.52 MeV gamma rays from nitrogen in bulk samples with 2.50 MeV gamma ray from bismuth in BGO detector material, an excellent agreement between the experimental and calculated yields of nitrogen gamma rays indicates satisfactory performance of the setup for detection of nitrogen in bulk samples.

1. Introduction

Prompt gamma-ray neutron activation analysis (PGNAA) technique is widely used for *in situ* elemental analysis of bulk samples in several scientific disciplines including environmental, industrial, and health sciences [1]. Its area of application ranges from quality-control tasks in mining and environmental [2–5] and building construction industries [6] to contraband detection for homeland security in concealed containers [7–11]. Prompt gamma rays can be excited in samples via thermal neutron capture (n_{th}, γ) reaction and fast neutron inelastic scattering ($n, n' \gamma$) reactions. Former reaction is used for elements with appreciable thermal neutrons capture cross-sections, while later reaction is used for elements with negligible thermal neutron capture cross-section. Therefore prompt gamma rays produced via 14 MeV neutron inelastic scattering from elements are used to measure C, N, and O concentrations in bulk samples [12–16]. Due to interference of weak intensity nitrogen prompt gamma rays with oxygen gamma rays, detection of nitrogen in bulk samples is a tedious task in 14 MeV neutron inelastic scattering studies. In spite of small thermal neutron capture cross-section, nitrogen can also be detected via prompt

gamma ray studies in thermal neutron capture reaction studies [17, 18]. Conventionally, nitrogen detection in bulk sample via thermal neutron capture is carried out using a ²⁵²Cf neutron source [17, 18]. In this study nitrogen has been detected in bulk samples via thermal neutron capture using a D-D reaction-based portable neutron generator. Thermal neutrons were produced in conjunction with 2.5 MeV fast neutrons from the portable neutron generator using the high-density polyethylene moderators. The study has been carried out first time about use of a D-D portable neutron generator in detection of nitrogen in bulk samples.

King Fahd University of Petroleum and Minerals (KFUPM), Dhahran, Saudi Arabia, has acquired a portable D-D reaction-based pulsed neutron generator model MP320, from Thermo-Fisher, USA, for the elemental analysis of bulk samples for prompt gamma studies. A thermal neutron capture-based prompt gamma ray neutron activation analysis setup has been developed utilizing the portable neutron generator. This setup was tested with nitrogen concentration measurement in bulk samples of explosive and narcotics proxy material. For a practical application of nitrogen detection for explosive detection concealed in cargo containers using PGNAA technique, this setup can

be modified. In this case, the cylindrical moderator can be replaced with a moderator-slab inserted between the neutron generator and the object under investigation. This will also allow to reduce the data acquisition time because sample size is much larger than the laboratory samples and detector covers much larger solid angle. In the following the study is described in detail.

2. Moderator Design of Portable Neutron Generator-Based PGNA Setup

Monte Carlo calculations were carried out using code MCNP4B2 [19] to design moderator of portable neutron generator-based PGNA setup. Since portable neutron generator provides limited flux of 2.5 MeV neutrons, its moderator should have optimum efficiency. This required optimization of source-moderator-sample geometry. In order to optimize source-moderator-sample geometry of the setup, thermal neutron intensity at the sample location was calculated for three different arrangements of source-moderator-sample geometry shown in Figure 1. Part (a) of the Figure 1 shows geometry for thermal neutron production at a cylindrical sample (90 mm in diameter and 145 mm in height) by inserting a cylindrical high-density polyethylene (HDP) slab between the portable neutron generator and the sample. The HDP slab has a 25 cm diameter and its thickness was varied in one cm steps and thermal neutron yield was calculated in sample volume for each slab thickness. In the next step, as shown in Figure 1, part (b), sample was enclosed in a HDP cylinder and was placed after the HDP slab with optimum thickness of 6 cm obtained from calculation of part (a). In this part of the study, the outer diameter of the cylinder was varied in equal steps and thermal neutron yield was calculated in sample volume for each outer diameter of the HDP cylinder. Finally, sample was enclosed in an HDP cylinder and was placed next to neutron generator, as shown in Figure 1 part (c). The thermal neutron yield was calculated in the sample volume as a function of HDP cylinder outer diameter.

Figure 2 shows the calculated yield of thermal neutron as a function of moderator thickness for the three different cases, superimposed upon each other for comparison purpose. For moderator cylinder, Figure 2 moderator thickness has been obtained by subtracting cylinder constant inner diameter from the variable outer diameter.

The thermal neutron yield for the slab case (Figure 1, part (a)) increases with moderator thickness and reaches an optimum value for 6 cm thick slab and then it drop-off primarily due to increasing distance from the neutron source. The drop-off may also be caused due to increasing thermal neutron flux consumption in thermal neutron capture reaction in the increasing moderator material. As shown in Figure 2, then by enclosing the sample in HDP cylinder, the thermal neutron yield further increases with increasing thickness of the moderator, that is, increasing outer diameter of the cylinder. The neutron yield then drops. As shown in Figure 2, the maximum yield of thermal neutron in slab+cylinder configuration was obtained with a combination of 6 cm thick slab and HDP cylinder with 17 cm outer diameter

FIGURE 1: Top schematic view of three different source-moderator-sample geometries of PGNA setup tested in the present study—(a) source-slab moderator, (b) source-slab-cylindrical moderator, and (c) source-cylindrical moderator.

FIGURE 2: Thermal neutron yield at the sample location from the three geometries study—(a) slab moderator, (b) slab-cylindrical moderator, and (c) cylindrical moderator plotted as a function of the effective moderator thickness.

(4 cm wall thickness of the HDP cylinder). Figure 2 shows thermal neutron yield for sample enclosed in cylindrical alone (Figure 1, part (c)). The yield increases with the cylinder outer diameter till it reaches a maximum value for HDP cylinder with 21 cm outer diameter (corresponding to 6 cm wall thickness of HDP cylinder). With further increase in wall thickness of the cylinder, the thermal neutron intensity decreases, a trend similar to the ones observed for Figure 1, parts (a) and (b). The thermal neutron intensity obtained in Figure 1 pt (c) configuration was two times higher than the optimum values achieved from Figure 1, parts (a) and (b),

FIGURE 3: Schematic side view of the portable neutron generator based PGNAA setup used in the study.

configurations. Therefore, Figure 1 part (c) configuration was chosen to design the moderator of the portable neutron generator-based PGNAA set.

Figure 3 shows the schematic of the portable neutron generator based PGNAA setup. The PGNAA setup consists of a cylindrical specimen container placed in a cylindrical cavity drilled through a cylindrical HDP moderator. A cylindrical gamma ray detector, with its longitudinal axis aligned along the sample's longitudinal axis, detects the prompt gamma rays from the sample. The moderator is placed adjacent to portable neutron generator. In order to prevent undesired gamma ray and neutrons from reaching the detector, 3 mm thick lead shielding and 50 mm thick neutron shielding are surrounding the gamma ray detector. The neutron shielding is made of a mixture of paraffin and lithium carbonate mixed in equal weight proportions. In the next step yield of nitrogen prompt gamma ray was calculated from proxy materials bulk sample, that is, melamine, caffeine, urea, and disperse orange material samples; elemental composition and masses are given in Table 1.

The samples were contained in 90 mm diameter and 145 mm long plastic bottles. The intensity of a specific capture gamma ray of nitrogen is proportional to partial elemental cross-section due to capture of thermal neutrons in nitrogen, listed in Table 2 [20]. Figure 4 shows calculated yield of 1.68, 2.52, 3.68, 5.28, 6.32, and 10.83 MeV nitrogen gamma rays as a function of nitrogen concentration in melamine, caffeine, urea, and disperse orange bulk samples. The calculated yield of 2.52 MeV prompt gamma rays from nitrogen was later compared with the experimental yield of prompt gamma rays from the melamine, caffeine, urea, and disperse orange samples.

FIGURE 4: Gamma ray calculated yield plotted as a function of nitrogen concentration of caffeine, disperse orange, melamine, and urea samples obtained through Monte Carlo simulations.

3. Experimental

3.1. Prompt Gamma Analysis of Melamine, Caffeine, Urea and Disperse Orange Samples. Nitrogen concentration in melamine, caffeine, urea, and disperse orange material samples was determined through their irradiation in the newly designed MP320 generator-based PGNAA setup shown in Figure 3. A pulsed beam of 2.5 MeV neutrons was produced with 70 keV deuteron beam via $D(d, n)$ reaction using portable neutron generator. The portable neutron generator was operated with a pulsed deuteron beam with 5 milliseconds pulse width and 250 Hz beam frequency. The pulsed neutron beam improves the signal-to-background ratio in the PGNAA studies. The generator was operated at 70 μ A deuteron beam current. The nitrogen prompt gamma ray yield data from the samples were acquired using a newly purchased 100 mm $\times$ 100 mm (diameter $\times$ height) bismuth germinate (BGO) detector for a period of 3 to 4 hours per sample. BGO detector was chosen because it contains bismuth, which has smaller capture cross-section of thermal neutrons. Therefore, BGO detector is less sensitive to neutron radiation damage as compared to a NaI detector.

During the irradiation of the samples, the BGO detector, although well shielded, was also exposed to thermal neutrons and it registered the prompt gamma rays due to the capture of thermal neutrons in bismuth (Bi) and germanium (Ge) present in the BGO detector. The energies and intensities of prominent prompt gamma rays due to capture of thermal neutrons in detector and samples material are listed in Table 2 [20]. Figure 5 shows the pulse height spectra of the BGO detector from caffeine sample.

In this study, gamma ray spectra with energies less than 3.2 MeV energy were acquired. Nitrogen gamma rays with higher energies were not analyzed in this study because they

TABLE 1: Elemental composition of the bulk samples used in present study.

Compound	Formula	Sample mass (g)	H (wt.%)	C (wt.%)	N (wt.%)	O (wt.%)
Melamine	$C_3H_6N_6$	682	4.8	28.6	66.7	0
Urea	CH_4N_2O	816	6.7	20.0	46.7	26.7
Caffeine	$C_8H_{10}N_4O_2$	580	5.2	49.5	28.6	16.5
*Disperse orange	$C_{18}H_{14}N_4O_2$	534	4.4	67.9	17.6	10.1

*H, C, N, and O composition elements calculated for a sample containing 100% disperse orange.

TABLE 2: Capture gamma rays energies and partial elemental cross section in bismuth, germanium and nitrogen [20].

Element	Gamma ray energy (keV)	$\sigma_y^z(E_\gamma)$ -barns
Bi	162	0.008
	320	0.0115
	674	0.0026
	2505	0.0021
	2828	0.00179
	4054	0.0137
	4171	0.0171
Ge	175	0.164
	493	0.133
	500	0.162
	596	1.100
	608	0.250
	868	0.553
	961	0.129
	1101	0.134
	1204	0.141
	1472	0.083
N	1678	0.0063
	1885	0.0147
	2000	0.0032
	2520	0.0044
	3532	0.0071
	3678	0.0116
	4508	0.0132
	5269	0.0236
	5297	0.0168
	5533	0.0155
	5562	0.0084
	6322	0.0145
	7298	0.0075
	8310	0.0033
	10829	0.0113

were interfering with prompt gamma rays from the detector material. Further higher energies gamma rays had very poor statistics because photoabsorption cross-section of gamma ray above 3 MeV energy decreases drastically due to competing Compton scattering and pair production reactions. This reduces detection probability of high energy gamma

FIGURE 5: Prompt gamma ray experimental pulse height spectra from caffeine sample over 0–3.22 MeV taken with the BGO detector. Also superimposed is lower energy part suppressed caffeine sample pulse height spectrum.

rays above 3 MeV gamma rays. Although intensity of 10 MeV nitrogen line is 2.6 times higher than 2.52 MeV line but the photoelectric cross-section drops by a factor of 300 from 2 to 10 MeV gamma gamma rays. This results in net reduction in detection efficiency of 10 MeV gamma rays using the BGO detector.

3.2. Results and Discussions. Figure 5 shows BGO detector pulse height spectrum for caffeine sample over 0–3.22 MeV containing the 2.22 MeV hydrogen capture peak from the moderator material. Also shown in Figure 5 is single escape (SE) peak associated with hydrogen capture full energy peak at 2223 keV. Figure 5 further shows prompt gamma ray peaks at 596, 608, 868, 961, and 1472 due to thermal neutron capture in germanium in BGO detector material as was previously observed [5]. Also shown in Figure 5 are unresolved prompt gamma ray peaks from bismuth in BGO detector material and nitrogen in caffeine measured at 2.50 MeV and 2.52 MeV, respectively. Since the production cross-section of 2.52 MeV nitrogen gamma ray is two times higher than 2.50 MeV gamma ray of bismuth, nitrogen peak will still be measureable for samples containing nitrogen in quantities comparable with bismuth contents of the BGO detector.

The nitrogen prompt gamma ray peak was located at the higher energy end of the pulse height spectrum. The count rate in the lower energy part of the spectrum was high and it was desired to suppress count rate in lower energy part of the

spectrum to minimize detector dead time. The lower energy part of the pulse spectrum was suppressed (offset) using analogue standard NIM single-channel analyzer, gate and delay generator and linear gate circuitry shown in Figure 6. For data analysis purpose, each pulse height spectrum was converted into excel spectrum for later spectrum subtraction and peak integration. Also shown in Figure 5 is caffeine sample pulse height spectrum with solid line after lower energy part of the spectrum is suppressed. In order to resolve the bismuth-nitrogen peaks from hydrogen 2.22 MeV peak, detector operating voltage was raised by 50 volts above its optimum operating voltage. Due to this effect, hydrogen peak shown with solid line is broader than the peak shown with dotted line and also bismuth-nitrogen peaks are more resolved from hydrogen 2.22 MeV peak than in the dotted line spectrum.

Figure 7 shows lower energy suppressed pulse height spectra of melamine, urea, and caffeine samples over 1.76–2.81 MeV showing hydrogen capture peak at 2.22 MeV along with unresolved prompt gamma ray peaks from bismuth and nitrogen at 2.50 and 2.52 MeV, respectively. Figure 8 shows enlarged portion of Figure 7 over 2.39–2.84 MeV showing unresolved prompt gamma ray peaks from bismuth and nitrogen in melamine, urea, caffeine, and disperse orange superimposed upon background spectrum.

Since all samples have different matrices material, it was difficult to find a common background sample material. Therefore background spectrum was taken with the empty sample plastic container. Further water or polyethylene background sample are not suitable in this case because hydrogen contents of water or polyethylene background sample are higher than the actual samples and resulting background level around 2223 keV hydrogen capture peak in the background sample will be higher than actual spectrum. This will suppress the sample effects in the background subtracted spectra.

The nitrogen peaks have increasing trends depending upon their nitrogen concentration. The lowest nitrogen peak intensity has been measured for prompt gamma ray peak from disperse orange sample with 17.6 wt. % nitrogen and the highest nitrogen peak intensity has been observed for prompt gamma ray peak from melamine sample with 66.7 wt. % nitrogen concentration, as expected from data shown in Table 1. Finally, area under the normalized nitrogen peaks of melamine, urea, caffeine, and disperse orange samples was integrated and background was subtracted.

The integrated yield of 2.52 MeV prompt gamma rays peak of nitrogen as a function of nitrogen concentration is plotted in Figure 9. The solid line in Figure 9 represents the calculated yield of prompt gamma ray obtained from Monte Carlo calculations following the procedure described elsewhere [4, 5, 15]. There is an excellent agreement between the theoretical yield and the experimental yield of nitrogen prompt gamma ray measured from melamine, urea, caffeine and disperse orange bulk samples using a portable neutron generator based PGNA setup utilizing a 100 mm × 100 mm (diameter × height) BGO detector.

Finally the minimum detection limit of nitrogen concentration in proxy material samples MDC was calculated for the portable neutron-based PGNA setup using the equation

FIGURE 6: Electronics block diagram used to suppress the lower energy part of the BGO detector spectrum.

FIGURE 7: Enlarged lower energy suppressed prompt gamma ray experimental pulse height spectra of BGO detector from caffeine, melamine, and urea samples over 1.76–2.81 MeV showing hydrogen capture peak along with unresolved 2.50 and 2.52 MeV bismuth and nitrogen peaks, respectively.

described elsewhere in detail [21]. The detection limit for an elemental concentration MDC measured under a peak with net counts N_p and associated background counts N_B under the peak is defined by the following:

$$\text{MDC} = 4.653 * \left(\frac{C}{N_p} \right) * \sqrt{N_B}, \quad (1)$$

where C is the element's concentration in the peak corresponding to net counts N_p . The corresponding error in MDC, that is, $\sigma_{\text{MDC}} = (C/N_p) * [\sqrt{(2 * N_B)}]$.

The MDC of nitrogen in proxy material samples for the KFUPM portable neutron generator was calculated to be

FIGURE 8: Enlarged unresolved 2.50 and 2.52 MeV bismuth and nitrogen prompt gamma ray experimental pulse height spectra of BGO detector from caffeine, disperse orange, melamine, and urea samples over 2.39–2.84 MeV.

FIGURE 9: Experimental integrated yields of 2.52 MeV prompt gamma rays from nitrogen plotted as a function of nitrogen concentration in caffeine, disperse orange, melamine, and urea samples. Line represents the results of the Monte Carlo simulations.

8.2 ± 2.5 wt%. In spite of low thermal neutron flux of the portable neutron generator and low thermal capture cross-section of nitrogen, the value of MDC for nitrogen detection using a portable neutron generator is reasonable. The study has provided useful data for application of a portable D-D neutron generator in detection of nitrogen in bulk material samples.

4. Conclusion

In this study, a D-D portable neutron generator-based PGNA setup has been designed and tested for nitrogen concentration measurements in the bulk samples. The PGNA set up response was tested through 2.52 MeV nitrogen prompt gamma ray yield measurements from melamine, urea, caffeine, and disperse, orange bulk samples containing nitrogen over 17.6–66.7 wt%. In spite of low flux of thermal neutrons produced by D-D portable neutron generator and interference of 2.52 MeV gamma rays from nitrogen in bulk samples with 2.50 MeV gamma ray from bismuth in BGO detector material, an excellent agreement has been observed between the experimental and the calculated yield of nitrogen prompt gamma rays as a function of nitrogen concentration in melamine, urea, caffeine, and disperse orange bulk samples. This indicates the excellent performance of the portable neutron generator-based PGNA setup for detection of elements with low thermal neutron capture cross-section.

Acknowledgments

This study is part of projects no. IN090033 and RG1201 funded by the King Fahd University of Petroleum and Minerals, Dhahran, Saudi Arabia. The support provided by the Department of Physics and Department of Chemistry, King Fahd University of Petroleum and Minerals, Dhahran, Saudi Arabia, is also acknowledged.

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