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Response tests of a $LaCl_3$:Ce scintillation detector with low energy prompt gamma rays from boron and cadmium

A.A. Naqvi [a,*], M.S. Al-Anezi [a], Zameer Kalakada [b], Faris A. Al Matouq [a], M. Maslehuddin [d], M.A. Gondal [a], A.A. Isab [c], Khateeb-ur-Rehman [a], M. Dastageer [a]

[a] Department of Physics, King Fahd University of Petroleum and Minerals, Dhahran, Saudi Arabia
[b] Department of Civil Engineering, King Fahd University of Petroleum and Minerals, Dhahran, Saudi Arabia
[c] Department of Chemistry, King Fahd University of Petroleum and Minerals, Dhahran, Saudi Arabia
[d] Center for Engineering Research, King Fahd University of Petroleum and Minerals, Dhahran, Saudi Arabia

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ABSTRACT

The yield of 478 and 558 keV gamma-rays have been measured from water samples containing 0.031–0.500 wt. % boron and 0.0625–0.500 wt. % cadmium, respectively, using a cylindrical 76 mm × 76 mm (height × diameter) $LaCl_3$:Ce detector. Inspite of interferences between detector-associated and the sample-associated prompt gamma rays, the $LaCl_3$:Ce detector has excellent resolution for the low energy prompt gamma-rays. An excellent agreement has been observed between the experimental and calculated yield of boron and cadmium prompt gamma ray from water samples.

1. Introduction

Application of Prompt Gamma-ray Neutron Activation Analysis (PGNAA) technique is increasing in various industrial, environmental, homeland security, medical discipline due to continuous development in gamma ray detection capability such as improved energy resolution and detection efficiency (Atanackovic et al., 2007; Baechler et al., 2002; Eleon et al., 2011; Favalli et al., 2010; Grazman and Schweikert, 2005; Idiri et al., 2010; Khelifi et al., 2007; Naqvi et al., 2011; Paul and Lindstrom, 2000). Recently radiation hardened inorganic Lanthanum-halide $LaBr_3$:Ce and $LaCl_3$:Ce series gamma ray detectors, with higher density and light output than NaI and BGO detectors have been developed (Iltis et al., 2006; Alexiev et al., 2008; Ciema et al., 2009; Shah et al., 2003). The light output of $LaBr_3$:Ce, $LaCl_3$:Ce and NaI detectors are (63,000 Photons/MeV), (46,000 Photons/MeV) and (39,000 Photons/MeV), respectively, (Alexiev et al., 2008; Ciema et al., 2009; Iltis et al., 2006; Shah et al., 2003). Below 120 keV the energy resolution (FWHM) of the lanthanum halide detectors (42% for 5.9 keV x-rays) is comparable with NaI detector (39% for 5.9 keV x-rays) of same size. (Iltis et al., 2006; Balcerzyk et al., 2005) while above 120 keV lanthanum halide detectors have superior resolution energy (FWHM) than that of a NaI detector of the same size. For 662 keV gamma rays, the energy resolution (FWHM) of $LaBr_3$:Ce, $LaCl_3$:Ce and NaI detectors are measured to 3%, 4% and 6.9%, respectively, (Alexiev et al., 2008; Ciema et al., 2009). Due to their faster light decay time, lanthanum-halide detectors can operate over wide dynamic ranges of count rate with little variation in the energy resolution (Iltis et al., 2006; Alexiev et al., 2008; Ciema et al., 2009; Eleon et al., 2011). Although $LaCl_3$:Ce detector has slightly poorer energy resolution than $LaBr_3$:Ce detector but it is 20–30% cheaper than $LaBr_3$:Ce detector of the same size. Although price of a 76 mm × 76 mm (diameter × height) $LaCl_3$:Ce detector is 10–12 times more than that of a NaI detector of the same size, but it offers superior performance than that of the NaI detector(for photon energy in excess of 120 keV) in terms of energy resolution, higher detection efficiency, higher counting rate handling capabilities and higher speed of fast component of the light component (Balcerzyk et al., 2005).

The shortcoming of lanthanum halide scintillators is their intrinsic activity, which is due to unstable La isotope and U-series contaminant present in the detectors. U-series contaminant has

* Corresponding author.
E-mail address: aanaqvi@kfupm.edu.sa (A.A. Naqvi).

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been reduced in new versions of lanthanum halide detectors. The intrinsic activity of the lanthanum halide detector due to the unstable La isotope is proportional to the size The integrated counts for 1436+32 keV intrinsic gamma ray peak for cylindrical 25 mm × 25 mm, 38 mm × 38 mm and 51 mm × 76 mm (diameter × height) $LaBr_3$:Ce detectors were recorded to be 0.66 counts/s, 3.32 counts/s and 21.1 counts/s, respectively, (Menge et al., 2007). For 1436+32 keV γ line 50 counts/s (4.1 counts/cm^3 s) were recorded for a 25 mm × 25 mm (diameter × height) $LaCl_3$:Ce detector (Balcerzyk et al., 2005) For a large volume 4 in. × 6 in. (diameter × height) $LaCl_3$:Ce detector, the integral intrinsic count rate in the energy region 20–3 MeV was measured to be 1.8 counts/cm^3 s (Owens et al., 2007). The intrinsic activity is not a major concern with for fast lanthanum halide detectors.

Lanthanum halide detectors have been commercially available in relatively large sizes (Menge et al., 2007; Owens et al., 2007). PGNAA setup employing lanthanum halide detectors with better resolution and higher photopeak efficiency are expected to have better performance due to their higher counts in narrow peaks with less background under the peak as compared to those employing NaI and BGO detector. The lanthanum halide detector's intrinsic activity may limit their utilization in low-level counting (Alexiev et al., 2008). Although smaller size Lanthanum halide detectors crystal have less intrinsic activity but their pulse height spectra for high energy gamma rays become complex due to associated single and double escape peaks (Eleon et al., 2011). The escape peaks may interfere with full energy peaks of interest making the spectrum analysis complex. For testing the performance of medium size detectors low energy gamma are used because the resulting pulse spectrum of lanthanum detector is then free of escape peaks, less complex and easy to analyze (Eleon et al., 2011).

King Fahd University of Petroleum and Minerals (KFUM), Dhahran, Saudi Arabia has recently acquired a cylindrical 76 mm × 76 mm (height × diameter) $LaCl_3$:Ce detectors, model BrilLanCe 350 from Saint-Gobain Crystals, Europe for prompt gamma ray analysis of bulk samples. The response of the $LaCl_3$:Ce detector was tested for 478 and 558 keV prompt gamma-rays produced through thermal neutrons capture in boron and cadmium contaminated water samples, respectively. Boron and cadmium concentration analysis in water samples were chosen because of their importance for environmental studies (Baechler et al., 2002; Grazman and Schweikert, 2005; Idiri et al., 2010; Khelifi et al., 2007). Boron and cadmium contaminated waste water is hazardous to human health because consumption of crops grown in soil irrigated with B and Cd-contaminated sewage over extended periods of time can lead to a number of illnesses (Baechler et al., 2002; Cengiz et al., 2009; Garcia-Reyes et al., 2000; Grazman and Schweikert, 2005). The maximum concentration for cadmium in drinking water and waste water is 0.005 mg/liter (0.005 ppm) (Health Canada: www.hc-sc.gc.ca) and 0.01 mg/liter (0.01 ppm) (http://www.fao.org/), respectively. Also the maximum concentration of total boron in freshwater aquatic life (Marine) should not exceed 1.2 mg B/liter (1.2 ppm) (http://www.env.gov.bc.ca/) and in drinking water its upper limit is 5.0 mg/liter (5 ppm) (http://www.env.gov.bc.ca/). For waste water being used for irrigation total boron concentration limit varies from 0.5–6.0 mg/liter (0.5–6.0 ppm) (http://www.env.gov.bc.ca/).

PGNAA technique can be used to detect boron and cadmium concentration in environmental and biological samples using nuclear reactor as well as non-nuclear-reactor based PGNAA setups. For non-nuclear reactor based PGNAA setups B and Cd detection limits are relatively higher than those reported for nuclear reactor-based PGNAA setups. Best detection limits of cadmium (01–0.1 ppm) and boron (0.3 ppm) have been obtained utilizing nuclear-reactor based PGNAA setups (http://www.ncnr.nist.gov/instruments/pgaa/; Khokhlov et al., 2009; http://www.ncnr.nist.gov/instruments/pgaa/). For non-nuclear-reactor based PGNAA setups, a minimum detection limit of 13.6 ± 0.2 ppm for Cd (for a 125 mL phantom) has been reported for a ^{238}Pu-Be neutron source based PGNAA setup (Grinyer et al., 2007). For a 1 Ci Am-Be source based PGNAA setup (utilizing 2.2×10^6 n/s neutron source) Idiri et al. have reported a minimum detection limits for boron and cadmium 1.6 ppm and 6.7 ppm, respectively, for cylindrical water samples with 40 cm diameter and 20 cm height (Idiri et al., 2010). In short, PGNAA technique, when used in conjunction with nuclear reactors-based and non-reactor based neutron sources, has adequate detection sensitivity to measure boron and cadmium in waste water.

The response of the $LaCl_3$:Ce detector was tested for detection of low energy prompt gamma-rays from boron and cadmium-contaminated water samples using KFUPM portable neutron generator-based PGNAA setup (Naqvi et al., 2012). In the following text the detailed results of performance tests of $LaCl_3$:Ce detector for detection of low energy prompt gamma rays are described.

2. Experimental

The $LaCl_3$:Ce detector was tested newly designed KFUPM portable neutron generator based PGNAA setup (Naqvi et al., 2012). The new PGNAA setup mainly consists of a cylindrical specimen placed inside a central cylindrical cavity drilled in a cylindrical high density polyethylene moderator. The maximum diameter of the sample depends upon the external diameter of the polyethylene moderator. The longitudinal axis of the $LaCl_3$:Ce gamma-ray detector was aligned along the moderator and sample's major axis. In order to prevent undesired gamma-rays and neutrons from reaching the detector, 3 mm thick lead shielding and 50 mm thick paraffin shielding were provided around the gamma-ray detector. The neutron shielding is made of a mixture of paraffin and lithium carbonate mixed in equal weight proportion. Associated with each size of the cylindrical moderator is an optimum value of the specimen radius and length that can produce maximum yield of the prompt gamma-rays at the detector location. This size can be determined from the Monte Carlo calculations (Naqvi et al., 2011). The results of the Monte Carlo calculations showed that the optimum radius and length of the sample for high density polyethylene moderators with 250 mm outer diameter were 90 mm and 140 mm, respectively, (Naqvi et al., 2012). Fig. 1 shows the schematic of the PGNAA setup used in the portable neutron generator based PGNAA setup.

2.1. Measurement of intrinsic activity and activation spectrum of $LaCl_3$:Ce detector

In the present study intrinsic activity and activation spectra of $LaCl_3$:Ce detector were acquired using standard NIM electronics modules. The detector signal, which was routed through a preamplifier, was processed through a spectroscopy amplifier with shaping time of 1 micro seconds. Logical gate signal was generated for each signal processed by the amplifier using single channel analyzer and gate and delay generators modules. For dead time correction, one of the outputs of the gate and delay generator was used to gate Multichannel Buffer, while another output was used to calculate dead time correction. Fractional Dead time DTC, assuming due to Multichannel Buffer based data acquisition system only, was calculated at the end of each experimental run from the integrated count in the stored spectrum N_{tot} and total gate signals N_{gates} counted independently by a

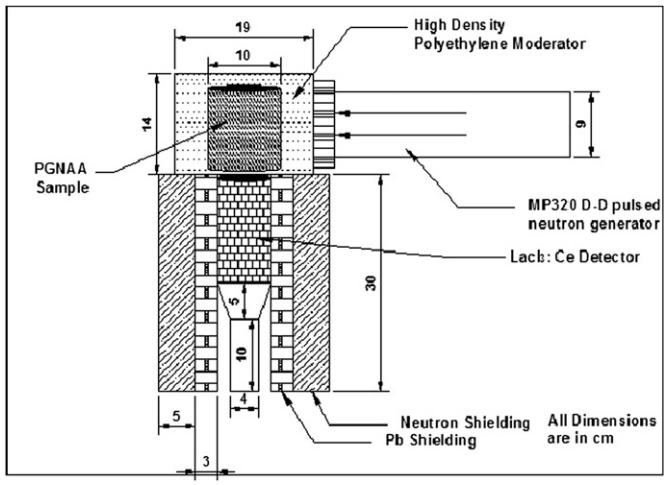

Fig. 1. KFUPM portable neutron generator based PGNAA setup used in the present study.

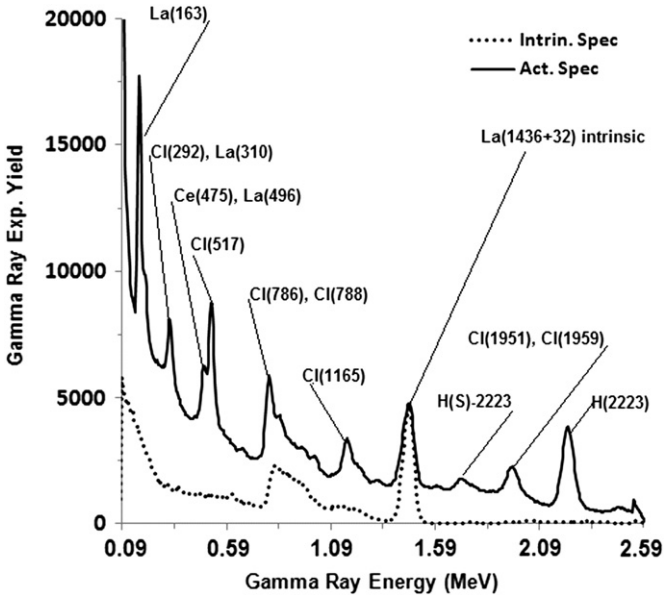

Fig. 2. $LaCl_3$:Ce detector activation spectrum superimposed upon the detector intrinsic activity pulse height spectrum.

high speed counter, using the relation

$$\mathrm{DTC} = [(N_{\mathrm{gates}} - N_{\mathrm{tot}})/N_{\mathrm{gates}}] \quad (1)$$

Then dead time corrected experimental yield of counts under a peak $Y_{\mathrm{DTC\text{-}Corr}}$ were obtained from experimental counts under the peak Y_{exp} using the relation:

$$Y_{\mathrm{DTC-Corr}} = Y_{\mathrm{exp}}[1 + \mathrm{DTC}] \quad (2)$$

Fig. 2 shows the detector's intrinsic activity pulse height spectrum superimposed upon activation spectrum of the $LaCl_3$:Ce detector. The detector has 4% energy resolution (FWHM) for 662 keV gamma-rays from ^{137}Cs. The 1436 keV gamma lines originating from the beta electron capture branches of La (Alexiev et al., 2008; Ciema et al., 2009; Eleon et al., 2011; Favalli et al., 2010) is shown in Fig. 1. The 1436 keV gamma line appears as 1468 keV gamma-ray peak (sum peak of 1436 keV gamma line and 32 keV X-ray fluorescence peak resulting from 32.2 keV K shell X-ray fluorescence of Ba, produced due to the electron capture of La). The abnormal width of the sum line of 32 keV X-ray fluorescence peak and 1436 keV gamma line may be due to overlapping of 1436 keV gamma line from La with 1460 keV line of ^{40}K, originating from the glass of the photomultiplier tube (Alexiev et al., 2008).

The intrinsic activity of the KFUPM $LaCl_3$:Ce detector was measured and intrinsic activity rate was determined through area integration under 1468 keV peak. The spectrum was acquired using 1 micro second shaping time of the amplifier for 1106 seconds (0.157 counts/cm^3 s) with 347.6 cm^3 $LaCl_3$:Ce detector. The measured intrinsic activity rate of the $LaCl_3$:Ce detector was 55.4 counts/s (0.157 counts/cm^3 s). Using the reported data of intrinsic activity count rate for various sizes of LaBr:Ce detectors (Menge et al., 2007) the photopeak efficiency of the $LaCl_3$:Ce detector was calculated from the ratio of measured intrinsic activity count rate of 55.4 counts/s and the calculated intrinsic activity of 332 Bq for 1436+32 keV photopeak of the detector. The calculated value 0.167 of the photopeak efficiency for our 76 mm × 76 mm $LaCl_3$:Ce detector is consistent with reported photopeak efficiency of 0.108 and 0.138 for smaller 41 mm × 76 and 51 mm × 76 mm $LaBr_3$:Ce detectors (Menge et al., 2007).

Activation spectrum of $LaCl_3$:Ce detector was measured using the KFUPM portable neutron generator based PGNAA setup (Naqvi et al., 2012). The activation spectrum of the $LaCl_3$Ce detector was recorded for 20 min runs. A pulsed beam of 2.5 MeV neutrons was produced via D(d,n) reaction using 70 μA beam of 70 keV deuteron. The deuteron pulse had a width of 800 micro seconds and a frequency of 250 Hz.

The activation spectrum of the LaC_{l3}:Ce detector contains prompt gamma-rays peaks due to capture of thermal neutrons in La, Cl and Ce element present in $LaCl_3$:Ce detector along with intrinsic activity peaks. Energies and intensities of prominent prompt gamma-rays due to capture of thermal neutrons in lanthanum, cerium and chlorine are listed in Table 1 (Choi et al., 2006). All these peaks are present in the sample spectra taken with the detector and needed to be subtracted from sample spectrum as the detector beam associated background. The activation spectrum of the detector shown in Fig. 2, shows most of the prompt gamma-ray lines of lanthanum, cerium and chlorine along with 2.22 MeV hydrogen capture peak in the moderator and neutron shielding of the detector, listed in Table 1, Due to short irradiation time delayed gamma-rays from ^{140}La (half life=40.3 h) could not be detected.

2.2. Prompt gamma-ray yield measurement from boron and cadmium contaminated water samples.

The prompt gamma-ray yield of boron and cadmium contaminated water samples was measured using the $LaCl_3$:Ce detector. The contaminated water samples were prepared by thoroughly mixing boron and cadmium compounds with water and filling it in cylindrical plastic bottles with 145 mm length and 90 mm internal diameter. Four cadmium samples with 0.0625, 0.125, 0.25 and 0.50 wt. % cadmium concentration and four boron samples with 0.031, 0.125, 0.250 and 0.50 wt. % boron concentration were prepared. The concentration of boron and cadmium in water samples was independently measured using Atomic Absorption Spectrometry Laboratory of Department of Chemistry, King Fahd University of Petroleum and Minerals, Dhahran, Saudi Arabia. The contaminated water samples were then irradiated in the portable neutron generator based PGNAA setup. The pulsed neutron beam was produced using the pulsed deuteron beam with specification given under subsection 2.1. Pulsed neutron beam improves signal to background ratio in the PGNAA studies. The prompt gamma-ray data from boron and cadmium containing water samples were acquired for preset number of time using Multichannel Buffer based data acquisition system.

Table 1
Energies and partial elemental cross section $\sigma_\gamma^z(E_\gamma)$-barns of prominent capture gamma-rays of boron and cadmium [Choi et al., 2006].

Element	Gamma-ray energy (keV)	$\sigma_\gamma^z(E_\gamma)$-barns
B(n,α)	478	716
Cl[a]	517	7.58
	786	3.42
	788	5.42
	1164	8.90
	1601	1.21
	1951	6.33
	1959	4.10
	2863	1.82
	3061	1.13
Cd	245	274
	558	1860
	651	359
Ce	475	0.082
	662	0.241
	1107	0.040
La	163	0.489
	272	0.502
	288	0.73
	567	0.335
	595	0.103
	789	intrinsic
	1436	intrinsic

[a] Energies and intensities of Cl prompt gamma rays are given for energies below 3 MeV.

Dead time was calculated for prompt gamma-ray spectra of boron and cadmium water samples using Eqs. (1) and (2) given in sub-section 2.1. As expected dead time was smaller for lower concentration samples and increased with increasing concentration of boron and cadmium in water. For four boron samples with 0.031, 0.125, 0.250 and 0.50 wt. % boron the dead time correction was calculated to be 3.8, 5.0, 8.8 and 25%, respectively. For four cadmium samples with 0.0625, 0.125, 0.25 and 0.50 wt. % cadmium concentration, the dead time correction was measured to be 2.5, 5.0, 8.8 and 21%, respectively. Since the capture cross section of cadmium is almost 2.5 times that of (n,α) cross section of boron, we operated neutron generator in cadmium runs with half of the beam current as compared to boron run.

For each boron and cadmium run the data were acquired for 25 minutes. Neutron flux during each run was monitored using a cylindrical 76 mm × 76 mm (height × diameter) NE213 detector placed at a distance of 1.0 m from the neutron generator. Neutron monitor spectrum was recorded for each concentration of boron and cadmium and was used for neutron flux normalization during data correction.

Fig. 3(a) shows pulse height spectra of prompt gamma-rays from water samples containing 0.031, 0.125, 0.250 and 0.5 wt. % boron superimposed upon each other along with background spectrum taken with pure water sample. In order to show effect of increasing concentration of boron on the pulse height spectrum, pulse height spectra for different boron concentration are plotted with a constant vertical offset. The 478 keV boron gamma-ray peak along with intrinsic 1436+32 keV intrinsic activity peak are quite prominent. Fig. 3(b) shows 478 keV boron peak on enlarged scale to show its interference with 475 keV peak from activation of cerium in $LaCl_3$:Ce detector. Since boron peaks contain the contribution of Ce(475) peak, difference spectra of boron peaks for 0.031, 0.125, 0.250 and 0.500 wt. % concentration were generated by subtracting the background spectrum from each of them. Fig. 3(c) shows the difference spectra of boron peaks for 0.031, 0.125, 0.250 and 0.500 wt % boron concentrations. Finally, the

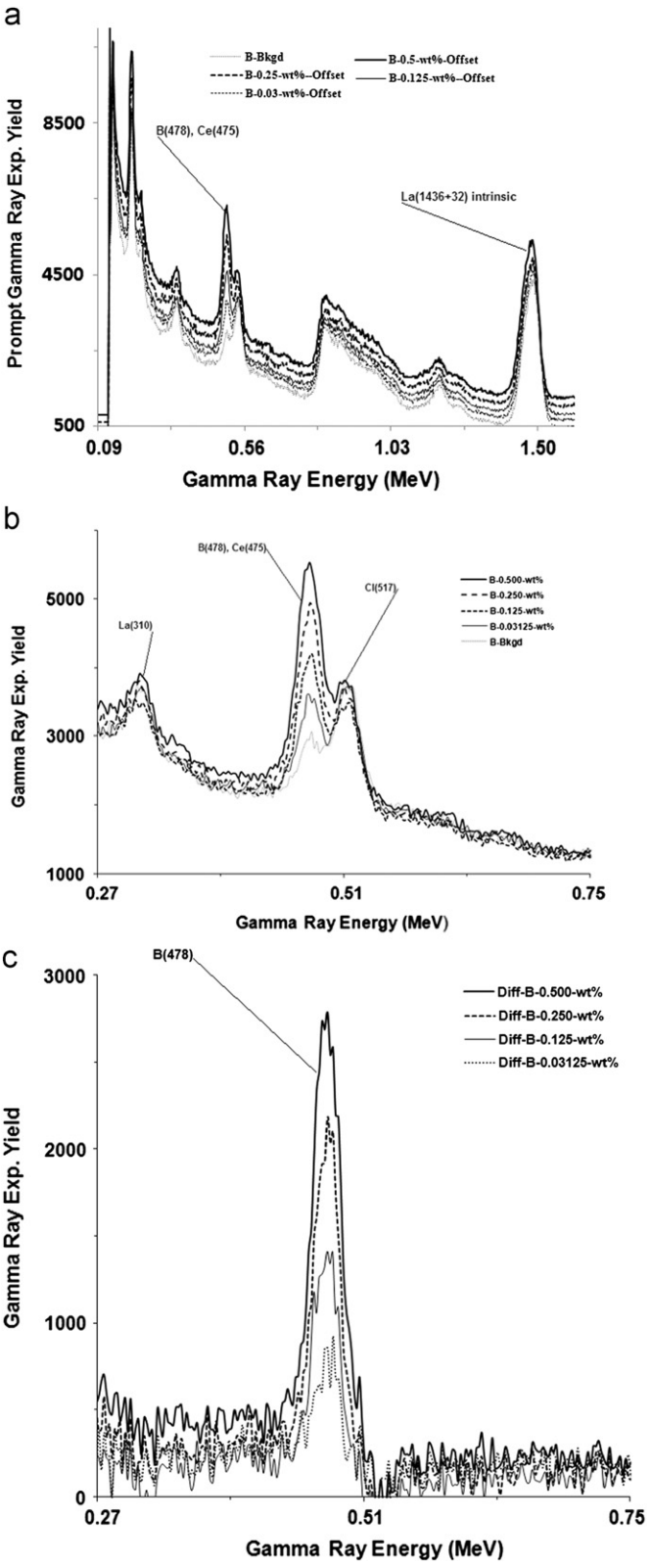

Fig. 3. (a) Prompt gamma-rays pulse height spectra of four boron contaminated water samples containing 0.031, 0.125, 0.250 and 0.50 wt. % boron, along with background spectrum taken with pure water sample, plotted with a constant vertical offset. (b) Enlarged prompt gamma-ray experimental pulse height spectra of water samples containing 0.031, 0.125, 0.250 and 0.5 wt % boron, along with background pure water sample, showing interference of 478 keV boron peak with 475 keV Ce peak. (c) Enlarged prompt gamma-ray experimental pulse height spectra after background subtraction from the four boron-contaminated water samples.

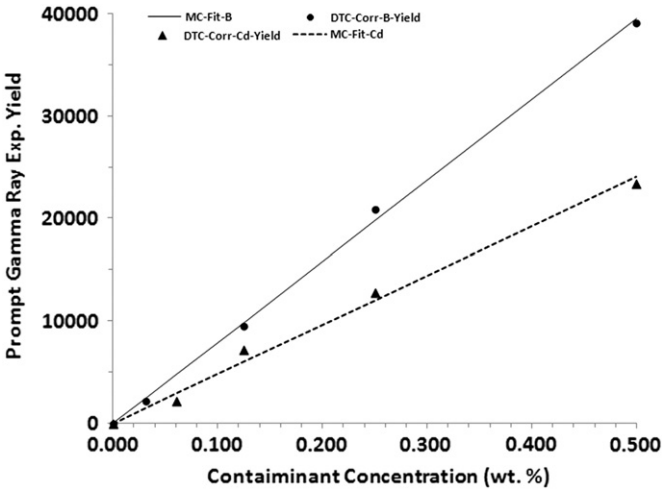

Fig. 4. Dead time corrected integrated yield of 478 and 558 keV prompt gamma-ray of boron and cadmium, respectively, from four contaminated water samples plotted as a function of contaminant concentration. The solid line shows normalized-calculated yield of the gamma-rays obtained through Monte Carlo calculations..

peaks of the difference boron spectra were integrated to generate integrated boron gamma yield as a function of boron concentration. Similarly the pulse height spectra of prompt gamma rays from water samples containing 0.0625, 0.125, 0.250 and 0.500 wt. % cadmium were analyzed through difference spectra and were integrated to generate integrated cadmium gamma yield as a function of boron concentration.

The integrated boron and cadmium gamma ray yield data was corrected for dead time correction and neutron flux fluctuation using neutron monitor count for each boron and cadmium concentration peak. Fig. 4 shows dead time corrected and background subtracted counts of four cadmium and boron samples as a function of cadmium and boron concentration in water samples. The lines in Fig. 4 represent results of calculated yield of boron and cadmium prompt gamma-ray obtained from Monte Carlo calculation following the procedure described elsewhere (Naqvi et al., 2011). There is an excellent agreement between the theoretical yield and experimental yield of prompt gamma-ray from boron and cadmium water samples measured as a function of boron and cadmium concentration, respectively, as measured by LaCl:Ce_3 detector.

2.3. Minimum detection limit of boron and cadmium in water samples

Finally the minimum detection limit of boron and cadmium concentration in water MDC was calculated for the KFUPM portable neutron generator based PGNAA setup using the procedure described elsewhere in detail (Gedcke, 2001). The detection limit MDC for an elemental concentration C measured under a peak with net counts P and associated background counts B (under the peak) where the P count and B counts integration is carried out for same channel width and for same time, is defined by (Gedcke, 2001)

$$\mathrm{MDC} = 4.653 \times (C/P) \times \sqrt{B} \quad (3)$$

where C/P is concentration (wt.%)/counts for a specific gamma ray peak.

$$\text{The error in MDCi.e. } \sigma_{\mathrm{MDC}} = (C/P) \times [\sqrt{(2{*}B)}] \quad (4)$$

For 90 mm × 140 mm (diameter × height) cylindrical water sample; the minimum detection limit of KFUPM portable neutron generator based PGNAA setup for boron and cadmium in water samples was determined from $LaCl_3$:Ce detector tests data. The minimum detection limit of boron $\mathrm{MDC_B} = 104 \pm 32$ ppm was calculated from 0.5 wt % B data with net counts $P_B = 70915$, background counts $B_B = 100270$ and using Eq. (3). Similarly minimum detection limit of cadmium $\mathrm{MDC_{Cd}} = 130 \pm 32$ ppm was calculated from 0.5 wt % Cd data with $P_{Cd} = 34417$, $B_{Cd} = 36942$ and using Eq. (3). Within statistical uncertainty MDC values of boron and cadmium agree with each other.

Idiri et al.(2010) have reported a minimum detection limits for boron $\mathrm{MDC_B} = 1.6$ ppm and cadmium $\mathrm{MDC_{Cd}} = 6.7$ ppm for a cylindrical (with 40 cm diameter and 20 cm height) water samples using a 1 Ci Am-Be source (2.2×10^6 n/s source) based PGNAA setup utilizing a portable high purity 20% efficiency HpGe gamma ray detector. Since the MDC data of the LaCl3:Ce detector based PGNAA setup has been obtained from tests results of a PGNAA setup, whose design was not optimized to obtain optimum value of MDC, there is room for improvement in presently reported MDC for boron and cadmium for a $LaCl_3$:Ce based PGNAA setup.

3. Conclusions

In this study, the response of a $LaCl_3$:Ce detector was tested for the detection of low energy prompt gamma-rays from boron and cadmium contaminated water samples using a portable neutron generator spectrum. The boron concentration was varied over 0.031, 0.125, 0.250 and 0.500 wt. % in water samples while cadmium concentration was varied over 0.0625, 0.125, 0.250 and 0.500 wt. %. An excellent agreement has been observed between the experimental yield of prompt gamma-rays and the calculated yield of prompt gamma-rays for the given concentration. Inspite of interferences of the detector background gamma rays with the sample associated prompt gamma rays, the $LaCl_3$:Ce detector has excellent response for low energy prompt gamma-rays from boron and cadmium.

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