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Hydrogen, carbon and oxygen determination in proxy material samples using a $LaBr_3$:Ce detector

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HIGHLIGHTS

- Hydrogen, carbon and oxygen concentration measurement in bulk samples using 14 MeV neutrons induced prompt gamma rays.
- Prompt gamma analysis of narcotics and explosive proxy materials e.g. ammonium acetate, caffeine, urea and melamine Bulk samples.
- Prompt gamma detection using large cylindrical 76 × 76 mm^2 (diameter *x* height) $LaBr_3$:Ce detector.
- Carbon/oxygen elemental ratio measurement from explosive and narcotics proxy material samples.

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ABSTRACT

Hydrogen, carbon and oxygen concentrations were measured in caffeine, urea, ammonium acetate and melamine bulk samples via 14 MeV neutron inelastic scattering using a $LaBr_3$:Ce detector. The samples tested herein represent drugs, explosives and benign materials, respectively. Despite its intrinsic activity, the $LaBr_3$:Ce detector performed well in detecting the hydrogen, carbon and oxygen elements. Because 5.1 MeV nitrogen gamma rays interfere with silicon and calcium prompt gamma rays from the room background, the nitrogen peak was not detected in the samples. An excellent agreement was observed between the experimental and theoretical yields of 2.22, 4.43 and 6.13 MeV gamma rays from the analyzed samples as a function of H, C and O concentrations, respectively. Within statistical errors, the minimum detectable concentration (MDC) of hydrogen, carbon and oxygen elements in the tested materials were consistent with previously reported MDC values for these elements measured in hydrocarbon samples.

1. Introduction

Recently, large-area radiation hardened $LaBr_3$:Ce and $LaCl_3$:Ce detectors have been manufactured with excellent energy resolution and light output (Alexiev et al., 2008; Ciema et al., 2009; Favalli et al., 2010; Menge et al., 2007; Owens et al., 2007). These devices have led to an increase in the applications for the Prompt Gamma-ray Neutron Activation Analysis (PGNAA) technique in in-situ elemental analysis of bulk samples in various disciplines, including environment (Paul and Lindstrom, 2000; Idiri et al., 2010; Seabury et al., 2007) and homeland security (Buffler and Tickner, 2010; Chichester et al., 2007; Eleon et al., 2011; Seabury, 2008; Strellis and Gozani, 2005). King Fahd University of Petroleum and Minerals, Dhahran, Saudi Arabia has acquired cylindrical $LaBr_3$:Ce (Naqvi et al., 2011) and $LaCl_3$:Ce (Naqvi et al., 2012a) detectors that are 76 mm in diameter and 76 mm high for prompt gamma ray studies for environmental and homeland security applications. Previously, $LaBr_3$:Ce detector responses were tested for hydrogen, carbon, and oxygen detection in bulk samples using a 14 MeV neutron-based PGNAA setup at KFUPM (Naqvi et al., 2012b). Herein, hydrogen, carbon, and oxygen were measured in explosive and narcotics-like bulk samples via 14 MeV neutron inelastic scattering studies using a $LaBr_3$:Ce detector. Explosives- and narcotics-like materials are distinctive compared with ordinary substances (benign substances) because they comprise characteristic C/O, N/O, Cl/O and Cl/H elemental ratios (Strellis and

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Gozani, 2005). As shown in Table 1, explosives have small C/O and N/O ratios, while narcotics have a high C/O ratio and small N/O ratio. Cocaine hydrochloride and heroine hydrochloride also have non-zero Cl/O and Cl/H ratios, which are typical signatures for chemical weapons (Buffler and Tickner, 2010; Seabury, 2008). Herein, C, O and H concentrations were measured in melamine, ammonium acetate, urea and caffeine bulk samples via the 14 MeV neutron inelastic scattering technique using a $LaBr_3$:Ce detector. These samples were used because their elemental composition ratios are similar to benign, explosive and narcotics materials, as shown in Table 2.

A D–T neutron generator can be used to detect H, C, N, and O in concealed materials in cargo containers using the 14 MeV neutron inelastic scattering technique, however, prompt gamma rays corresponding to double escape peak of 6.1 MeV oxygen strongly interfere with 5.1 MeV prompt gamma rays from nitrogen, hence making nitrogen detection difficult. A D–T neutron generator-based pulsed fast and thermal neutron activation analysis setup was developed to detect explosive and contraband material using C/O elemental ratios (Vourvopoulos and Womble, 2001). Nitrogen can also be detected via thermal neutron capture (Nasrabadi et al., 2011). For the simultaneous detection of N and H (via neutron capture) along with C and O (via fast neutron inelastic scattering), one needs to produce a mixture of fast and thermal neutron fluxes in sufficient quantity to carry out neutron inelastic and thermal capture studies simultaneously. A D–T neutron generator cannot be used as an efficient thermal neutron source due to poor moderation of high energy 14 MeV neutrons (Seabury et al., 2007). A mixture of fast and thermal neutrons is generally produced using a ^{252}Cf source in conjunction with an external moderator (Seabury et al., 2006). Since the average neutron energy of a D–D reaction-based neutron generator and that of a ^{252}Cf neutron source are very close, a D–D neutron generator along with a moderator can also be used to produce thermal neutrons. Despite its lower neutron intensity, higher thermal neutron yield has been reported from a D–D neutron generator than that from a D–T neutron generator (Seabury et al., 2007). This may be due to the fact that prior to thermalization, one needs to soften 14 MeV neutrons spectrum from a D–T generator using high Z scatterers such as tungsten, bismuth and iron (Simpson and Chichester, 2011). This will result in loss of 14 MeV neutrons intensity and thereby yielding less thermal neutrons from a D–T neutron generator than that from a D–D neutron generator. Therefore, a combination of a D–D neutron generator (with a moderator) and a D–T neutron generator can provide a fast and thermal neutrons flux mixture like a ^{252}Cf source with a moderator setup. The combination of a D–D generator (with a moderator) and a D–T neutron generator may be used simultaneously to detect C, O, N, and H in concealed materials for national security applications.

In the present study C, O, and H concentrations were measured in melamine, ammonium acetate, urea and caffeine bulk samples via 14 MeV neutron inelastic scattering using a $LaBr_3$:Ce detector. These samples were chosen because their elemental composition ratios are similar to groups of benign, explosive and narcotics proxy materials, as shown in Table 2.

Table 1
Elemental Ratio of common substance, explosive, narcotics and chemical weapons (Strellis and Gozani, 2005).

Category	Substance	Elements signature			
		C/O	N/O	Cl/O	Cl/H
Benign	**Salt**				
	Sugar	0.8	0	0	0
	Wood	1.1	0	0	0
	Petroleum				0
	Cement		0	0	0
	PVC	0	0	1.5	11.5
	Fiberglass	3	1.3	0	0
	Sea water	0	0	0	0.03
Explosive	**PETN**	**0.3**	**0.3**	0	0
	TNT	**0.9**	**0.4**	0	0
	Dynamite	**0.2**	**0.3**	0	0
	C4	**0.6**	**0.9**	0	0
Narcotics	**Heroin**	**3.2**	**0.2**	0	0
	Cocaine	**3.2**	**0.2**	0	0
	Heroin hydrochloride	**3.2**	**0.2**	0.1	1.5
	Cocaine hydrochloride	**3.2**	**0.2**	0.1	1.6
Chemical weapons	**Hydrogen cyanide**	0	0	0	0
	Mustard gas	0	0	**1.5**	**8.9**
	Sarin	1.5	0	0	0

2. Prompt gamma analysis of the materials

The carbon, oxygen and hydrogen concentrations for melamine, ammonium acetate, urea, and caffeine bulk samples were measured using the 14 MeV neutron-based PGNAA setup previously described in detail (Naqvi et al., 2012b). For continuity, the setup is briefly described herein. It primarily comprised a cylindrical plastic container that was 90 mm × 140 mm (diameter *x* height) and filled with the sample at a 0° angle to the 14 MeV neutron beam. The sample center was 70 mm from the tritium target. The $LaBr_3$:Ce detector was placed at a center-to-center distance of 125 mm from the sample, and the sample is at a 90° angle to the 14 MeV neutron beam axis, as shown in Fig. 1. The detector is shielded against 14 MeV, thermal neutrons and gamma rays through tungsten, paraffin and lead shielding, respectively.

Table 2
Elemental composition of proxy material samples analyzed in this study.

Material category	Compound	Formula	Elemental concentration (wt%)				$(C/O)_{calc}$	$(C/O)_{exp}$	N/O
			H	C	N	O			
Benign	**Melamine**	$C_3H_6N_6$	**4.8**	**28.6**	**66.7**				
Explosive	**Ammonium acetate**	$C_2H_7NO_2$	**9.1**	**31.2**	**18.2**	**41.6**	**0.75**	**0.92 ± 0.12**	**0.75**
	Urea	CH_4NO_2	**6.7**	**20.0**	**46.7**	**26.7**	**0.75**	**0.91 ± 0.11**	**1.75**
Narcotics	**Caffeine**	$C_8H_{10}N_4O_2$	**5.2**	**49.5**	**28.6**	**16.5**	**3.00**	**3.82 ± 0.38**	**1.73**

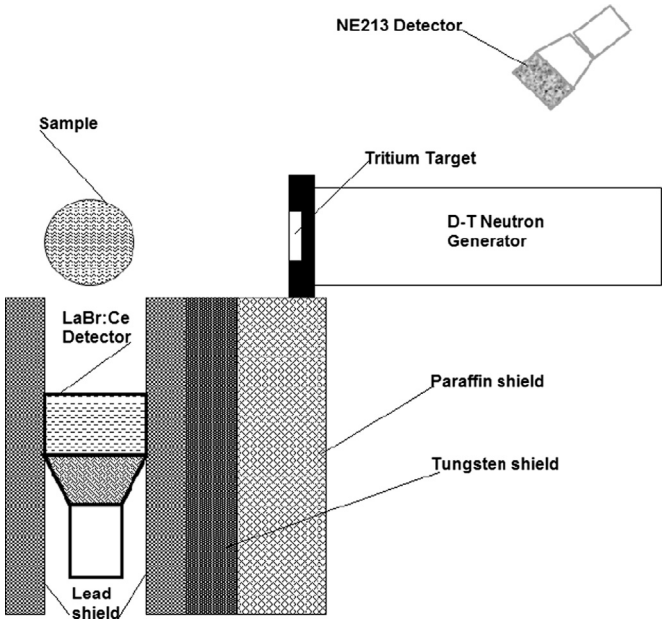

Fig. 1. Schematic of 14 MeV neutron-based setup used for detection of H, C and O concentration in bulk samples.

A pulsed beam comprising 14 MeV neutrons was produced via a T (d,n) reaction using a pulsed deuteron beam that was 200 ns wide and that had a 31 kHz frequency. The typical pulsed beam current for the accelerator was 60 μA. The fast neutron flux from the tritium target was monitored using a cylindrical 76 mm × 76 mm (diameter *x* height) NE213 fast neutron detector that was 1.8 m from the target and with a 130° angle to the beam. The prompt gamma ray spectra from the $LaBr_3$:Ce detector were recorded for a preset time. The 14.8 MeV neutron flux was measured at 10^6 n/cm^2/s, and the total gamma ray count rate for the $LaBr_3$:Ce detector with the sample was approximately 11–12 kHz.

The hydrogen, carbon and oxygen concentrations in the melamine, ammonium acetate, urea, and caffeine bulk samples were measured through element-specific prompt gamma rays produced via 14 MeV neutrons inelastic scattering. The element concentrations for the melamine, ammonium acetate, urea and caffeine bulk samples were independently verified using atomic absorption spectrometry in the Department of Chemistry, King Fahd University of Petroleum and Minerals, Dhahran, Saudi Arabia. For prompt gamma ray analysis, the samples were prepared by filling plastic containers with the sample materials. The containers were then irradiated in the 14 MeV neutron-based PGNAA setup. The prompt gamma-ray data from the samples were acquired for 20–30 min using a personal computer-based data acquisition system with a multichannel buffer module analog as the digital module. A neutron flux spectrum was recorded during each run using the NE213 detector, and the spectrum was later used for neutron flux normalization during data correction. The NE213 detector was operated at a half Cs-137 pulse height bias following the procedure previously described (Naqvi et al., 2012b).

2.1. Gamma ray yield measurements for the bulk samples

Fig. 2 shows the prompt gamma-ray pulse height spectra from inelastic 14 MeV neutron scattering for hydrogen, carbon and oxygen in the caffeine and ammonium acetate bulk samples with shielding for the floor and walls comprising 0.46–8.30 MeV using the $LaBr_3$:Ce detector. The gamma ray energy values from inelastic 14 MeV neutron scattering in lead have previously been reported

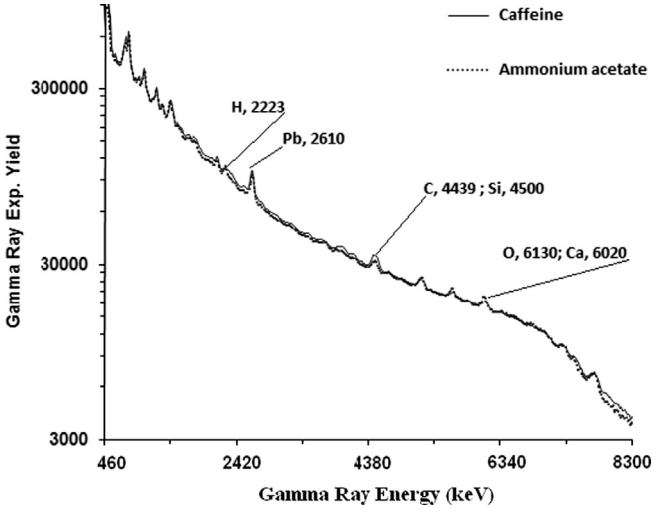

Fig. 2. Prompt gamma ray spectra of $LaBr_3$:Ce gamma ray detector from ammonium acetate and caffeine bulk samples plotted over 0.46–8.3 MeV energy range, superimposed upon each other.

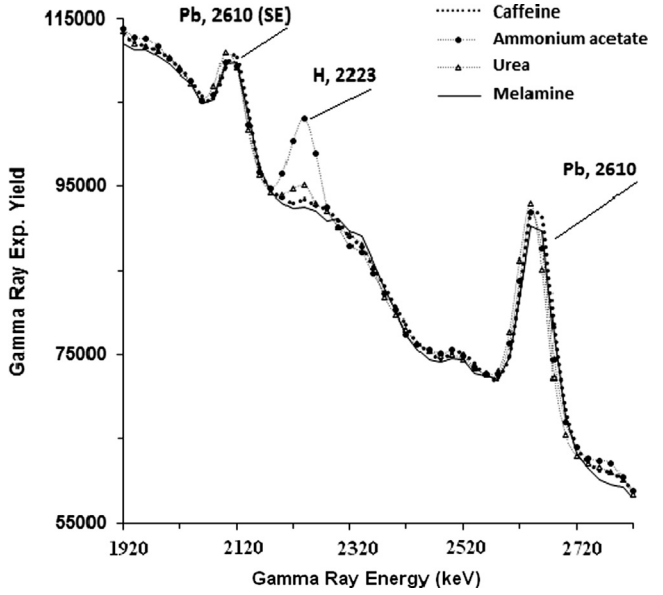

Fig. 3. $LaBr_3$:Ce detector prompt gamma ray spectra of from ammonium acetate, caffeine, urea and melamine bulk samples plotted over 1.92–2.82 MeV energy range showing hydrogen and lead shielding peaks.

(Naqvi et al., 2012b; Engesser and Thompson, 1967) and were observed at 570, 810, 1060 and 2620 keV herein.

Figs. 3–5 show the prompt gamma spectra for the caffeine, ammonium-acetate, urea, and melamine bulk samples with hydrogen, carbon and oxygen prompt gamma ray peaks in the 1.92–6.53 MeV range. Further, Figs. 3–5 show the background gamma rays produced through inelastic 14 MeV neutrons scattering for silicon and calcium from the walls and floor (Defense Nuclear Agency Report DNA # 2716, 1972). Such gamma rays can interfere with carbon and oxygen prompt gamma rays; the 4.50 MeV silicon peak interferes with the 4.44 MeV carbon peak, and the 6.02 MeV calcium gamma ray interferes with the 6.13 MeV oxygen peak (Simakov et al., 1998). Fig. 3 shows the full hydrogen energy peaks at 2.22 MeV and the Pb peak from the shielding material at 2.62 MeV. Also shown in the Fig. 3 is the single escape (SE) peak that corresponds to the full 2.62 MeV energy peak for Pb. The hydrogen peak increases intensity in the samples as the hydrogen concentration increases. The maximum peak intensity

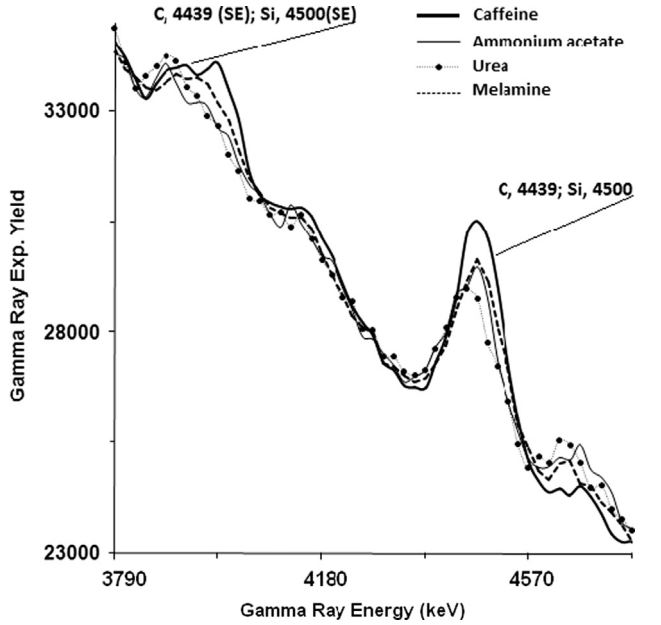

Fig. 4. $LaBr_3$:Ce detector prompt gamma ray spectra of from ammonium acetate, caffeine, urea and melamine bulk samples plotted over 3.79–4.76 MeV energy range showing carbon full energy peak along with carbon and silicon single escape peaks.

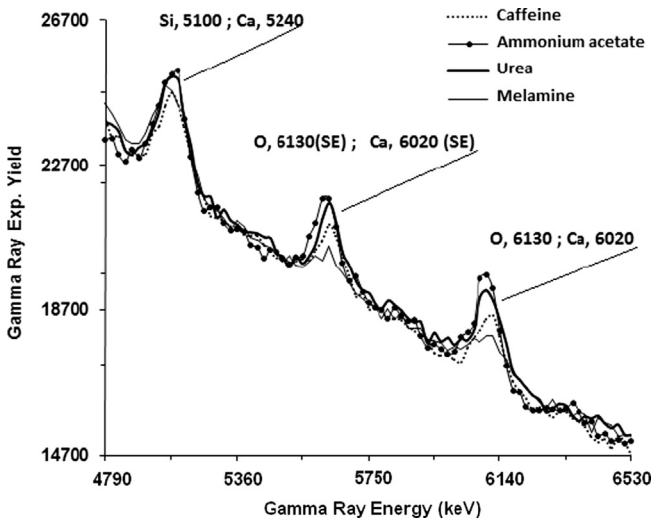

Fig. 5. $LaBr_3$:Ce detector prompt gamma ray spectra of from ammonium acetate, caffeine, urea and melamine bulk samples plotted over 4.97–6.53 MeV energy range showing oxygen peaks along with calcium and silicon peaks from the room background.

for hydrogen was observed in the ammonium acetate sample with a 9.1 wt% hydrogen concentration, while the minimum peak intensity for hydrogen was observed for melamine, which has a 4.8 wt% hydrogen concentration.

Fig. 4 shows the pulse height spectra for the caffeine, ammonium-acetate, urea and melamine bulk samples over a 3.79–4.76 MeV energy range with single escape (SE) and full energy peaks at 4.439 MeV prompt gamma rays via inelastic 14 MeV neutron scattering for carbon. The carbon peak intensity in Fig. 4 increases with carbon concentration in the samples (i.e., the maximum peak intensity for the carbon peak was observed in caffeine with 49.5 wt% carbon, and the minimum peak intensity was observed for the urea bulk sample containing 20.0 wt% carbon). Also shown in Fig. 5 is the SE peak that corresponds to a 5.10 MeV silicon full energy peak.

Fig. 5 shows the pulse height spectrum for the caffeine, ammonium-acetate, urea and melamine bulk samples over a 4.97–6.53 MeV energy range including the full energy peak at the 6.13 MeV prompt gamma ray via inelastic 14 MeV neutron scattering for oxygen. Also shown in Fig. 5 is the full energy peak for 5.10 MeV gamma rays through 14 MeV neutrons inelastic scattering from silicon in the walls and floor of the room. The full energy peak intensity for silicon is higher than the oxygen single escape peak because the silicon content is higher in the walls and floor. The full energy and single escape peaks at 6.13 MeV for oxygen from the caffeine, ammonium-acetate, urea and melamine bulk samples demonstrated an increasing trend proportional to the oxygen content in the sample. The maximum intensity for an oxygen peak was observed in the ammonium acetate sample, with a 41.6 wt% oxygen concentration, while the minimum intensity in an oxygen peak was observed for melamine, with a 0.0 wt% oxygen concentration. Because the silicon and calcium prompt gamma rays from the room background interfere with the 5.1 MeV nitrogen gamma rays, the nitrogen peak was not detected in the samples.

3. Results and discussion

In this study, data from a $LaBr_3$:Ce detector for hydrogen, carbon and oxygen peaks in the respective pulse height spectra for the caffeine, ammonium acetate, urea, and melamine bulk samples were analyzed. The net content for the H, C and O peaks were extracted by subtracting the floor and walls background spectra from the sample spectra. The net content was then corrected for dead time and neutron flux variations. Finally, the gamma rays yield curves as a function of H, C and O concentration in the bulk samples were generated.

Figs. 6 and 7 show the gamma ray yields as a function of the O, C and H concentrations for the caffeine, ammonium acetate, urea and melamine bulk samples measured using the $LaBr_3$:Ce detector. The lines in Figs. 6 and 7 are the theoretical O, C and H prompt gamma ray yields for the caffeine, ammonium-acetate, urea and melamine bulk samples calculated by Monte Carlo using the MCNP4C code (Briesmeister, 1997) following a procedure previously described (Naqvi et al., 2012b). The theoretical and experimental yields were consistent for the H, C and O prompt gamma-ray analysis as a function of the respective concentrations, as assessed by the $LaBr_3$:Ce detector, which demonstrated that the detector is appropriate for measuring H, C and O in contraband and explosive detection.

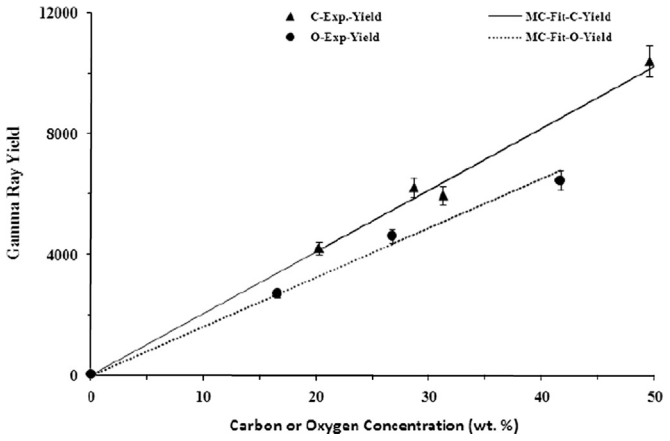

Fig. 6. Integrated normalized experimental yield of carbon and oxygen prompt gamma rays taken with the $LaBr_3$:Ce detector, plotted as a function of oxygen concentration in ammonium acetate, caffeine and urea bulk samples. The solid lines are Monte Carlo fits to the experimental data.

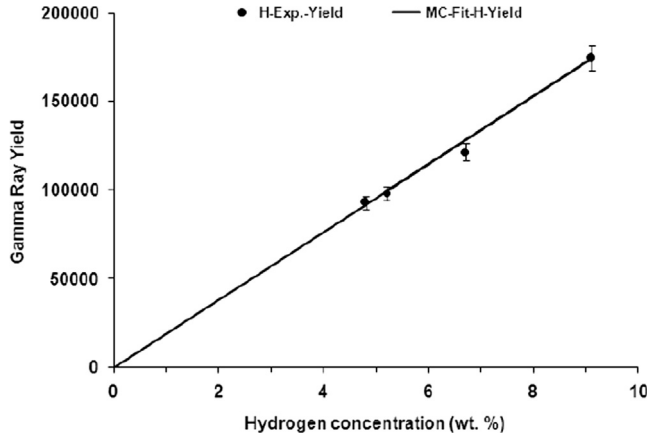

Fig. 7. Integrated normalized experimental yield of hydrogen prompt gamma rays taken with the $LaBr_3$:Ce detector, plotted as a function of hydrogen concentration in ammonium acetate, caffeine, urea and melamine bulk samples. The solid line is a Monte Carlo fit to the experimental data.

Table 3
Intercomparison of MDC of hydrogen, carbon and oxygen for the proxy material samples (present study) and hydrocarbon samples (Naqvi et al., 2012b) for KFUPM 14 MeV neutron-based PGNAA setup using $LaBr_3$:Ce.

Samples type	MDC_H (wt%)	MDC_C (wt%)	MDC_O (wt%)
Hydrocarbons	0.5 ± 0.1	12.2 ± 3.8	15.8 ± 4.8
Proxy materials	0.2 ± 0.1	11.9 ± 3.6	16.8 ± 5.1

The carbon to oxygen (C/O) element ratio for the samples, which differentiates between explosive and narcotics, was calculated from the experimental data for ammonium-acetate, urea and caffeine. The C/O ratios for the ammonium-acetate and urea were 0.92 ± 0.12 and 0.91 ± 0.11, respectively, while for caffeine, the C/O was 3.82 ± 0.38. As expected, the explosive-like materials (ammonium-acetate and urea) produced smaller C/O ratios, while the narcotics-like material (caffeine) generated a high C/O ratio. Within experimental error, the C/O ratios for ammonium-acetate and urea was consistent with the calculated value, while the C/O ratio for caffeine was approximately 15% higher than the calculated value.

Finally, the minimum detectable concentration, MDC, and its associated error, σ_{MDC}, were calculated (Naqvi et al., 2012b) for hydrogen and carbon in the caffeine, ammonium-acetate, urea and melamine bulk samples using the $LaBr_3$:Ce detector in the KFUPM 14 MeV-based PGNAA setup. The MDC and its associated error, σ_{MDC}, were calculated for an elemental concentration C measured for a peak with the net content P and associated background content B (under the peak) using the following equations (Naqvi et al., 2012b):

$$MDC = 4.653\left(\frac{C}{P}\right)\sqrt{B} \quad ; \quad \sigma_{MDC} = \left(\frac{C}{P}\right)\sqrt{2\,B} \quad ;$$

where P and B integrations were performed for the same time and channel width. For 90 mm × 140 mm (diameter x height) cylindrical bulk samples comprising caffeine, ammonium-acetate, urea and melamine, the MDC for hydrogen, carbon and oxygen using the KFUPM 14 MeV neutron-based PGNAA setup with the $LaBr_3$:Ce detector are listed in Table 3. For comparison, the MDC for hydrogen, carbon and oxygen in the bulk hydrocarbon samples measured using a $LaBr_3$:Ce detector (Naqvi et al., 2012b) are also included in Table 3. Within statistical error, the *MDC* values for H, C and O in the samples using the $LaBr_3$:Ce detector are consistent with the *MDC* from the $LaBr_3$:Ce detector for H, C and O in the hydrocarbon bulk samples previously reported (Naqvi et al., 2012b).

4. Conclusion

The $LaBr_3$:Ce detector was tested for detection of H, C and O in caffeine, ammonium acetate, urea and melamine bulk samples using a 14 MeV neutron-based PGNAA setup. An excellent agreement was observed between the experimental and theoretical yields of the 2.22, 4.43 and 6.13 MeV gamma rays as a function of H, C and O concentrations, respectively, in caffeine, ammonium acetate, urea and melamine samples. Within statistical error, the MDC values from the $LaBr_3$:Ce detector for H, C and O detection in the samples was consistent with the MDC from the $LaBr_3$:Ce detector for H, C and O in the hydrocarbon bulk samples previously reported.

The carbon to oxygen element ratios for the explosive-like materials (ammonium-acetate and urea) and narcotics-like material (caffeine) were determined from the experimental gamma-ray yields. As expected, the explosive-like materials produced smaller C/O ratios, while the narcotics-like material generated a high C/O ratio, demonstrates that the detector can reliably measure H, C and O concentrations in explosive- and narcotics-like materials.

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