Scintillation properties of CsBa₂I₅ activated with monovalent ions Tl⁺, Na⁺ and In⁺

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Abstract

We have previously reported the scintillation properties of CsBa₂I₅ activated with Eu²⁺, which exhibits excellent behavior [1, 2]. The presence of Cs in the lattice makes CsBa₂I₅ a good candidate for activation with monovalent ions. We grew single crystals of CsBa₂I₅ with monovalent ions Tl, Na and In as activators. We performed a series of luminescence and scintillation measurements, including pulse height measurements using two different photosensors, on these crystals. We show not only that the monovalent ions are suitable activators in the chosen host lattice but also that the system has light yields between 33,000 and 40,000 ph/MeV, lower self absorption than observed in Eu doped samples and excellent proportionality. The measured energy resolution on our initial samples of 7.1% FWHM for 662 keV gamma rays using avalanche photodiodes is very promising.

Keywords: scintillators, crystals, gamma-ray detection, activators, cesium, barium, iodine, thallium, indium, sodium, energy resolution, halides, non-proportionality

1. Introduction

In recent years many high performing new scintillators have been found which are activated with Eu²⁺ and Ce³⁺, including SrI₂:Eu, BaBrI:Eu and LaBr₃:Ce [3, 4, 5]. These compounds exhibit high light output, excellent energy resolution, and for Ce doped compounds fast decay times. Among this group is CsBa₂I₅:Eu, which we have reported to exhibit a light output greater than 100,000 ph/MeV and energy resolution of 2.5% [1, 2]. However, Eu²⁺ and Ce³⁺ have some significant disadvantages as activator ions. With these ions there is typically a small Stokes shift between the excitation band and the emission band, leading to significant self absorption which limits performance in large crystals. Additionally, the rare earth elements are expensive to obtain and difficult to purify. This motivates searching for alternative activator ions which can achieve the same performance in these excellent scintillators without the disadvantages of Eu²⁺ and Ce³⁺.

Tl⁺ and Na⁺ are used as activators in two of the most widely available commercial scintillators, NaI and CsI [6], respectively. These activators are monovalent and thus substitute onto lattices with monovalent sites. Tl⁺ and In⁺ are ions with the *ns*² configuration in the ground-state [7], the luminescence of which has been studied in a considerable number of hosts (e.g. CsI, NaI, etc.). In both of them, it is agreed that the optical emission is related to ³P₁–¹S₀ transition [8]. In the case of Na⁺, the origin of the luminescence is caused by the

radiative decay of singlet and triplet halide excitons perturbed by Na⁺ ions [9]. CsBa₂I₅, in addition to the divalent Ba site onto which the Eu²⁺ activator can be substituted, has a single monovalent Cs⁺ site, as shown in Figure 1. The Cs⁺ ions are coordinated by 9 iodide ions. Thus CsBa₂I₅ is an excellent candidate for activation with alternative ions. In this work we present the first studies of CsBa₂I₅ activated with Tl, Na and In. Single crystal samples were grown and extensive luminescence and scintillation measurements were performed. Pulse height measurements, using two different photosensors, revealed decent light yields and promising energy resolutions. Therefore this system may be a high performing alternative to the Eu and Ce activated compounds.

2. Experimental

Single crystal samples were grown following a similar procedure as was previously done for Eu doped samples [1]. High-purity reactants (99.9%) of CsI, BaI₂, TlI, NaI, InI and EuI₂ obtained as beads from Sigma Aldrich were used without additional purification. The reactants and products are sensitive to moisture and were handled in an argon-filled glovebox maintained below 0.1 ppm of O₂ and H₂O. Single crystals were grown by the Bridgman-Stockbarger method. Stoichiometric mixtures of starting materials were sealed in quartz ampoules and heated to roughly 650°C, 40 degrees above the melting point of CsBa₂I₅ of 610°C [10]. The ampoule was then slowly translated through a gradient of 10 C/cm at 0.75 mm/hr. Single crystals were grown with nominal concentrations in the melt of 0.5%, 0.08%, 1%, and 0.2% of Eu, Tl, Na and In, respectively. Dopant concentrations were varied for Tl and Na but here we present only data for the optimized values. Phase identification

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Figure 1: Schematic of a portion of a CsBa_2I_5 crystal structure [1] showing coordination of different ions. The monovalent Cs^+ site (on which the Ti^+ , Na^+ or In^+ can be substituted as an activator) is shown in purple and divalent Ba^{2+} sites (on which the Eu^{2+} can be substituted as an activator) are shown in orange. I^- ions are shown in blue.

was performed by powder XRD using a Bruker Nonius FR591 with a rotating anode X-ray generator using $\text{CuK}\alpha$ radiation.

Optical excitation, X-ray emission and pulsed X-ray decay time measurements were performed using the high-throughput facility at LBNL [11, 12]. The measurements were performed on samples of crystalline pieces ranging from 0.5 to 2 mm which were packed in gas tight quartz ampoules with inner diameter of 4 mm and length 15 mm. X-rays generated from the same setup previously described for XRD measurements were used to excite samples and their spectral response was recorded by a SpectraPro-2150i spectrometer (Acton Research Corp., Acton, MA) coupled to a PIXIS:100B charge-coupled detector (Princeton Instruments, Inc., Trenton, NJ). Photoluminescence excitation and emission spectra were recorded using the same spectrometer previously mentioned. A Xenon broad-spectrum lamp was used to excite the samples between 220 and 490 nm. The emission was measured from 250 to 850 nm. The experimental results were compared to $\text{CsBa}_2\text{I}_5(0.5\% \text{ Eu})$ measured in the same conditions [1, 2]. X-ray excited decay curves were measured on a custom made pulsed X-ray system consisting of an ultrafast laser (200 fs pulses at 165 kHz), a Hamamatsu N5084 light-excited X-ray tube, a Hamamatsu R3809U-50 microchannel PMT and an Ortec 9308 picosecond time analyzer. The instrumental response of the system is 100 ps FWHM.

Pulse height gamma-ray spectra were collected using sealed gamma-ray sources: ^{57}Co , ^{133}Ba , ^{109}Cd , ^{152}Eu , ^{241}Am , ^{54}Mn , ^{137}Cs , and ^{22}Na (Eckert & Ziegler Isotopes Products GmbH). Spectra were collected using two photosensors: 1) a Hamamatsu R6231-100 photomultiplier tube (PMT) connected to a Canberra 2005 preamplifier, an Ortec 672 spectroscopic amplifier and an Ortec EASY-MCA-8K multichannel analyzer; 2) a Hamamatsu S8664-1010 avalanche photodiode (APD)

connected to a Canberra 2004 preamplifier, a Canberra 2022 spectroscopic amplifier and an Amptek MCA multichannel analyzer. A satisfactory signal-to-noise ratio was ensured by collecting at least 10,000 counts for each photopeak, which were fit using a superposition of Gaussian functions corresponding to the photopeak and the X-ray escape peak and an exponential background to find the centroid and full-width at half-maximum. The high voltage was fixed at -700V and 430V for the PMT and APD, respectively. A 10 μs and 12 μs shaping times were used for the measurements with PMT and APD, respectively. Samples were optically coupled onto the window of the PMT with Viscasil 600,000 (GE) and covered with layers of reflecting tape (PTFE). All the measurements were done on samples of sizes ranging from 1 to 10 mm^3 . Light output was estimated by comparison of the photopeak position with the response of a 12 x 12 mm^3 commercial NaI:Tl single crystal from Saint Gobain measured under identical conditions and taking into account the PMT quantum efficiency provided by the manufacturer. The energy resolution, defined as the FWHM of the 662 keV full absorption peak, was also calculated.

3. Results and discussion

3.1. Optical characterization

Figure 2 presents the room temperature optically excited emission spectra of $\text{CsBa}_2\text{I}_5:0.08\%\text{Ti}^+$, $\text{CsBa}_2\text{I}_5:1\%\text{Na}^+$ and $\text{CsBa}_2\text{I}_5:0.2\%\text{In}^+$ in parts (a), (b) and (c), respectively. The spectrum of $\text{CsBa}_2\text{I}_5:0.5\%\text{Eu}^{2+}$ is shown in part (d) for comparison. The signature of the 5d-4f Eu^{2+} transition (Region I) is clearly seen in part (d). The maximum of the emission is around 430 nm (20 nm FWHM) and the excitation extends between 220 nm and 425 nm. This emission band is also present in the other three samples in parts (a)-(c), likely due to unavoidable traces of Europium contamination during growth. All samples also exhibit an emission band with maximum around 390 nm and excitation between 220 nm and 250 nm (Region II) which, according to Alekhin et al. [13] is ascribed to the host related luminescence.

While the Eu doped sample only exhibits these two emission bands, the Tl, Na and In samples all exhibit a third emission band which is different in each sample (Region III). While there is no data published about CsBa_2I_5 with these activators, a natural comparison can be made with CsI doped with Tl, Na or In which has been extensively studied. The $\text{CsBa}_2\text{I}_5:\text{Ti}^+$ sample shows a characteristic $6s^2 \rightarrow 6s^1 6p^1$ broad band emission with a maximum around 500 nm (160 nm FWHM) (Region III) and characteristic excitation between 250 nm and 375 nm. A similar band at about 560 nm was observed in CsI(Tl), which was attributed to thallium luminescence centers [14, 15, 16]. A slight shift in the emission of this band can be expected due to the differences in the host. The Na doped sample is the least intense of the three and it has an emission band around 400 nm (Region II) and an emission band around 700 nm (Region III) with excitations at around 250 nm and 275 nm, respectively.

Figure 2: Emission spectrum under optical excitation of CsBa_2I_5 doped with Tl, Na, In and Eu in parts (a), (b), (c) and (d), respectively.

According to Yakovlev et al. [9] and reference therein, $\text{CsI}(\text{Na})$ at 80K is characterized by two bands at 410 nm and 720 nm, which are caused by Vk and Na^0 color centers, respectively. These bands coincide with Regions II and III in our Na doped sample and therefore we can ascribe them to the same origins. In the case of the In doped sample we observe a broad and intense emission band with a maximum at 550 nm (120 nm FWHM) (Region III). According to Grindin et al. [17], $\text{CsI}(\text{In})$ and $\text{CsI}(\text{Tl})$ have similar bands but the ones for Indium are slightly shifted to lower energies. They observed an intense band at 545 nm similar to Region III, which was ascribed to the emission of the Indium luminescence center.

Figure 3: Normalized emission spectrum under optical excitation of CsBa_2I_5 doped with Tl^+ , Na^+ , In^+ and Eu^{2+} .

Figure 3 presents the normalized optical excited emission and excitation bands of CsBa_2I_5 doped with Tl, Na, In, and Eu at the maximum emission wavelengths of each compound. These spectra were measured under 2.48 eV (530 nm), 2.88 eV (430 nm), 2.30 eV (530 nm) and 2.88 eV (430 nm) for $\text{CsBa}_2\text{I}_5:0.08\%\text{Tl}^+$, $\text{CsBa}_2\text{I}_5:1\%\text{Na}^+$, $\text{CsBa}_2\text{I}_5:0.2\%\text{In}^+$, and $\text{CsBa}_2\text{I}_5:0.5\%\text{Eu}^{2+}$, respectively. The overlap between the emission and excitation bands is quite small in the Tl, Na and In doped samples, as opposed to the Eu doped samples where there is significant overlap. A rough estimate of the Stokes shift in Figure 3 can be done by comparing energy different between the maximum (lowest) excitation band and that of the emission band [18]. This estimate shows a 1.7 eV, 2.1 eV, 2.5 eV for the Tl, Na, In doped samples, respectively while it drops to only 0.8 eV in the case of the Eu doped sample. We thus expect much less self absorption in the Tl, Na and In doped samples.

3.2. X-ray luminescence and time response

Figure 4: X-Ray Luminescence of $\text{CsBa}_2\text{I}_5(\text{Tl})$, $\text{CsBa}_2\text{I}_5(\text{Na})$, $\text{CsBa}_2\text{I}_5(\text{In})$ and $\text{CsBa}_2\text{I}_5(\text{Eu})$

Figure 4 shows the normalized X-ray excited emission spectra of CsBa_2I_5 doped by the three activators studied in this work. The Eu doped sample, shown for comparison, exhibits the previously mentioned Eu^{2+} 5d-4f emission band at 430 nm, which is very sharp (24 nm FWHM). These spectra are similar to the emission bands observed under optical excitation in Figure 3, except in the case of the Na doped sample. In this case, the Eu impurities show a much clearer signal under X-ray excitation than under optical excitation. As we can see the emission of the Na doped sample is very broad while in the In and Tl doped sample are less broad. The In doped sample is red shifted relative to the Tl doped sample.

Figure 5: Pulsed X-Ray decay time of $\text{CsBa}_2\text{I}_5(\text{Tl})$, $\text{CsBa}_2\text{I}_5(\text{Na})$, $\text{CsBa}_2\text{I}_5(\text{In})$ and $\text{CsBa}_2\text{I}_5(\text{Eu})$

Figure 5 shows the X-ray excited decay curves for the three activators and the Eu doped sample for comparison. In this plot, we observe that the samples doped with Tl, In and Na are significantly faster than the Eu doped sample. The longer lifetime of the Eu doped sample decay is explained by the presence of self-absorption and delayed energy transfer mechanisms from the host to the Eu ions [2]. The decay curves of the Tl, Na and In doped samples were fit to a superposition of three single exponential components; the decay of the Eu doped sample was well fit by a superposition of only two components. The results of the fits are presented in Table 1. Relative fractions are included in parentheses next to decay times. Constant fraction means components significantly longer than 15 μs .

3.3. Pulse height measurements and non-proportionality

In order to define the ideal photodetector to be used, several factors have to be taken into account: the type of photosensor, the matching of the scintillator emission spectrum with the photosensor sensitivity spectrum, the quantum efficiency of the photosensor and the reflectivity of its entrance window, and finally the photoelectron or electron-hole pair collection efficiency [23]. Photomultiplier tubes (PMTs) and photodiodes such as PIN diodes (PDs) and Avalanche photodiodes (APDs) are the most widely used devices for

Figure 6: Pulse Height measurements obtained with a Hamamatsu R6231 PMT (a) and a S8664-1010 Hamamatsu APD (b) for $\text{CsBa}_2\text{I}_5(\text{Tl})$, $\text{CsBa}_2\text{I}_5(\text{Na})$, and $\text{CsBa}_2\text{I}_5(\text{In})$.

detecting scintillation light. The spectral response of the silicon detectors extends much farther into the long wavelength region than that of typical photocathodes. This extended spectral response is particularly well suited for scintillators with emission spectra that have a significant yield at longer wavelengths [19]. PDs have no internal gain which restricts their use to the detection of optical photons. Avalanche photodiodes (APD), like photomultiplier tubes (PMT), have an internal gain which enables the discrimination of weaker signals from the noise. We present a comparison of APDs and PMTs as detectors, and demonstrate improved sensitivity and energy resolution in the red spectral regions using the APD.

Figure 6 shows the pulse height spectra obtained using the PMT and APD in parts (a) and (b), respectively. When measured with a PMT the Tl doped sample shows the highest photopeak channel and best energy resolution of the three samples. However, when measured using the APD the In doped sample performs the best due to a better match of the optical emission of this crystal with the APD quantum efficiency (60% between 390 and 930 nm) [20]. The light output was estimated taking into account the photopeak position relative to a calibrated $\text{NaI}(\text{Tl})$ sample and by correcting for the quantum efficiency of the PMT. The results are presented in Table 2.

The light output of CsBa_2I_5 doped with Eu is much larger than the light output of Tl, Na and In doped CsBa_2I_5 sam-

Compound(dopant)	Decay time components (relative intensities)			
	1st	2nd	3rd	Constant fraction
CsBa ₂ I ₅ (Tl)	28 ns (1%)	414 ns (42%)	1241 ns (24%)	34%
CsBa ₂ I ₅ (Na)	39 ns (7%)	183 ns (36%)	995 ns (37%)	20%
CsBa ₂ I ₅ (In)	32 ns (2%)	162 ns (8%)	1980 ns (43%)	47%
CsBa ₂ I ₅ (Eu)	-	407 ns (47%)	1787 ns (27%)	26%

Table 1: Summary of pulsed X-rays decay time main components of the tested crystals.

Dopant (concentration)	Corrected Light Yield (^a)	Emission (λ_M) (^b)	APD QE(λ_M) (^c)	E.R. (^d) - PMT	E.R. (^d) - APD
Tl (0.08%)	40,000 $\pm$ 3,500	500 nm	81%	7.5 $\pm$ 0.7	7.1 $\pm$ 0.8
Na (1.0%)	33,500 $\pm$ 3,000	430 nm	71%	10.8 $\pm$ 0.8	7.9 $\pm$ 1.1
In (0.2%)	35,000 $\pm$ 3,000	540 nm	84%	8.1 $\pm$ 0.7	7.1 $\pm$ 0.8

^a Corrected Light Output in photons per MeV. See explanation in text

^b Maximum of the X-ray luminescence emission spectrum (Figure 4).

^c APD quantum efficiency at maximum emission wavelengths.

^d Energy resolution at 662 keV in %.

Table 2: Summary of the main properties of the tested crystals.

ples. However, when this comparison is done with CsI doped with the same activators different conclusions can be extracted: CsBa₂I₅(Tl) showed smaller values for light output and worse energy resolution than reported values for CsI(Tl), which are in the range of 56,000 to 61,000 ph/MeV [21, 22, 23, 24] and 4.5 to 5.7% [20, 21], respectively. In the case of CsI(Na), the reported values of light output vary between 38,000 to 49,000 ph/MeV [22, 24] and a 7.4% energy resolution [21], which are slightly better than the performance of CsBa₂I₅(Na). For CsI(In), [17] reported 27,000 ph/MeV and 9.5% energy resolution, values that are below the good performance of CsBa₂I₅(In).

Figure 7 shows the proportionality response to X- and γ -rays (part a) and energy resolution (part b) acquired with a PMT for energies between 22 keV and 835 keV using a standard set of radioactive sources. The three studied samples showed an excellent non-proportionality in this range, especially as compared to the response of CsI(Tl) [25]. The measurements for the samples with the three activators roughly coincide within the margin of error, implying that this property is likely governed by the host rather than the dopant used. The energy resolution of the scintillator can be defined as a quadratic sum of three contributions: the photodetector contribution, the crystal inhomogeneities contribution, and the non-proportional response of the crystal [26]. Considering the small size of this third term in these systems there is clear room for improvement in the energy resolution based on better crystal growths. Moreover, as shown by M. Moszynski et al. [27] the integration of the slow component may significantly improve energy resolution, especially for CBI(In) which shows the largest constant fraction.

Figure 7: Light yield non-proportionality (a) and Energy resolution (b) as a function of energy obtained with a PMT for CsBa₂I₅(Tl), CsBa₂I₅(Na), and CsBa₂I₅(In), respectively. CsI(Tl) was added for comparison. The dashed line is a guide to the eye. The solid lines are fits using a function $ER = C/\sqrt{E}$.

4. Summary and conclusions

We present here the first crystal growths and scintillation properties of single CsBa₂I₅ crystals doped with Tl, Na and In. These crystals were grown by the Bridgman technique.

The results show that these three monovalent ions are new activators in CsBa₂I₅. The light output under ¹³⁷Cs excitation is estimated to be 40,000, 33,500 and 35,000 ph/MeV for Tl, Na and In, respectively. As opposed to Eu doped samples, samples doped with these activators exhibit very small overlap between emission and excitation bands, minimizing self absorption. The emission bands of the samples all fall in the range between 350-700 nm which makes them suitable for pulse height measurements with photomultipliers and especially with avalanche photodiodes which have a high quantum efficiency of 60% between 390 nm and 930 nm. While the scintillation light is slower than that produced from Ce³⁺ activation, most of the light is emitted within the sub- μ s range, which still is suitable for most gamma-ray spectroscopy applications. The three samples exhibited an excellent non-proportionality as compared to CsI(Tl) and therefore a better energy resolution is expected from improved growths. These initial results are very promising considering that the crystal quality can still be improved and optimized.

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