

Scintillation and Luminescence Properties of Undoped and Cerium-doped LiGdCl₄ and NaGdCl₄

Yetta D. Porter-Chapman, Edith D. Bourret-Courchesne, Gregory Bizarri, Marvin J. Weber, and Stephen E. Derenzo, *Fellow, IEEE*

Abstract—We report the scintillation properties of the undoped and cerium-doped variations of LiGdCl₄ and NaGdCl₄. Powder samples of these materials exhibit significant scintillation under X-rays. The samples were synthesized by solid-state methods from a 1:1 molar ratio of lithium or sodium chloride and gadolinium chloride. Cerium trichloride was used as the dopant. The physical, optical, and scintillation properties of these materials were analyzed by powder X-ray diffraction, photoluminescence, X-ray excited luminescence, and pulsed X-ray luminosity measurements. Increases in light yields are observed as the concentration of cerium increases. The highest light yields occurred at 20 % cerium doping for both compounds. At larger concentrations neither compound formed, indicating a breakdown of the lattice with the addition of large amounts of cerium cations. At 20 % cerium, LiGdCl₄ and NaGdCl₄ display scintillation light 3.6 times and 2.2 times the light yield of the reference material, YAlO₃:Ce³⁺, respectively. Both emit in the ranges of 340 – 350 nm and 365 - 370 nm and display multiexponential decays with cerium-like decay components at 33 ns (LiGdCl₄:Ce) and 26 ns (NaGdCl₄:Ce).

Index Terms— cerium, decay time, gadolinium, luminescence, scintillators.

I. INTRODUCTION

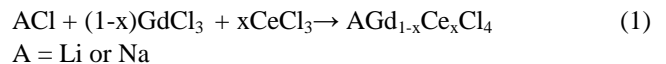
THE need for radiation detecting materials has been at the forefront of materials research in recent years due to applications in national security, medical imaging, X-ray detection, oil well logging, and high-energy physics. For gamma-ray detection, several known inorganic scintillators, such as bismuth germanium oxide (BGO) and lutetium oxyorthosilicate (LSO), are currently being utilized in various radiation detection devices. Although these scintillators have good luminosity, their scintillation properties (decay times, linearity, etc.) are lacking in one or more areas. Within the last

decade, researchers have reported significant scintillation in the cerium-doped binary lanthanide halides [1]-[9]. Additionally, Ce³⁺-doped ternary lanthanide halides, particularly those containing alkali metals, exhibit high luminosities (>20,000 ph/MeV) and fast decay times (< 1 μs) [10]-[15]. This report discusses the investigation of new scintillators in the alkali metal gadolinium halide (AGdCl₄, A= Li, Na) family. Five known compounds follow this general formula: LiGdF₄, NaGdF₄, KGdF₄, LiGdCl₄, and NaGdCl₄. Of these compounds, LiGdCl₄ and NaGdCl₄ exhibit efficient activation by Ce³⁺ cations. Each compound was synthesized using solid-state chemistry techniques and was characterized by powder X-ray diffraction (PXR), photoluminescence (PL), X-ray excited luminescence (XRL), and pulsed X-ray luminosity measurements (PXRL).

II. EXPERIMENTAL METHOD

A. Synthesis

LiGdCl₄ and NaGdCl₄ were synthesized using the solid-state reaction,



Due to the hygroscopic nature of the halides, all handling was performed in an argon-filled glove box under dry conditions. The LiGdCl₄ samples were heated at 700 °C for 1 h, whereas those of NaGdCl₄ were heated to 550 °C for 10 h, all in sealed evacuated quartz tubes. Both compounds were doped with the following molar percentages of cerium trichloride: 0, 0.5, 1, 2, 5, 10, and 20 %. Colorless hygroscopic powders were obtained for each sample.

B. Characterization

The crystallinity and structure identification of each sample was determined by PXR. An in-house diffraction setup comprised of a Bruker Nonius FR591 rotating anode X-ray generator equipped with a copper target and a 50 kV and 60 mA electron beam was utilized. A more detailed description of this setup is supplied in [16]. The resultant diffraction rings were converted into a 1D standard diffraction pattern with Fit2D freeware [17]. This pattern was then used to phase match with known diffraction patterns in the Powder

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Correspondence should be directed to S. E. Derenzo who is with the Lawrence Berkeley National Laboratory, Berkeley, CA 94720 USA (telephone: 510-486-4097, e-mail: sederenzo@lbl.gov).

Y. D. Porter-Chapman, E. D. Bourret-Courchesne, Gregory Bizarri, M. J. Weber, and S. E. Derenzo are with the Lawrence Berkeley National Laboratory, Berkeley, CA 94720 USA.

Diffraction File (PDF-4+) database [18] using the Match! Software (Crystal Impact GbR) [19].

All luminescence measurements were conducted at room temperature on samples consisting of micron-size particles contained in quartz cuvettes. Photoluminescence excitation and emission spectra were measured utilizing a Horiba Fluorolog 3 fluorescence spectrometer within the spectral range of 250 nm – 800 nm using spectral resolutions of 3 nm for excitation and 2 nm for emission. X-ray excited emission spectra were also measured for each sample using a second port of the Bruker Nonius x-ray generator and their spectral response was recorded by a SpectraPro-2150i spectrometer (Acton Research Corp., Acton, MA) coupled to a PIXIS:100B charge-coupled detector (CCD) (Princeton Instruments, Inc., Trenton, NJ) [16].

Scintillation decay measurements were performed on each sample and a sample of $\text{YAlO}_3:\text{Ce}^{3+}$ (YAP:Ce Phosphor Technology Ltd., PTL Grade – QM58/N-S1). An in-house pulsed X-ray system that produces 80 ps (fwhm) pulses of X-rays having a mean energy of 18 keV was utilized in these measurements. Fluorescent photons from each sample were detected by a microchannel phototube with 35 ps (fwhm) response [20]. The luminescence decay times were determined by fitting the data to a sum of exponential decay curves. In summary, the intensity is described by a sum of exponential components:

$$I(t) = f_0 + \sum_{i=1} [f_i/\tau_i] \exp[-(t-t_0)/\tau_i] \quad (2)$$

where the fractions sum to unity

$$\int I(t) dt = \sum_{i=0} f_i = 1 \quad (3)$$

The constant fraction f_0 and the fractions f_i and exponential decay times τ_i are varied to minimize chi-squared between $I(t)$ and the data. The pulsed X-ray source, microchannel phototube, and fitting procedures are described in [20]. The reported estimated luminosities are determined by comparison of the sample luminosities to the luminosity measured for the YAP:Ce powder standard. In addition, for normalization, we used an average luminosity of 18,000 ph/MeV for YAP:Ce in crystal form [21]-[25]. This gives a ratio of YAP(powder) to YAP (crystal) of 1.8 for our measurements. While we understand that this ratio might not be appropriate for all compounds and crystal lattices, it is used as a convenient method to estimate the luminosities of our samples.

III. RESULTS

LiGdCl_4 forms a tetragonal structure with a density = 3.70 g/cm^3 , and NaGdCl_4 forms a triclinic structure with a density = 3.44 g/cm^3 . In LiGdCl_4 , the dopant, Ce^{3+} , is assumed to occupy the gadolinium sites because it has a better fit in terms of ionic radii ($r(\text{Li}) = 0.59 \text{ \AA}$, $r(\text{Gd}) = 1.05 \text{ \AA}$ and $r(\text{Ce}) = 1.14 \text{ \AA}$) [26] and charge. In the case of NaGdCl_4 , the Na^+ and Gd^{3+} have similar bonding (both CN = 7) and ionic radii ($r(\text{Na}) = 1.12 \text{ \AA}$, $r(\text{Gd}) = 1.00 \text{ \AA}$ compared to $r(\text{Ce}) = 1.07 \text{ \AA}$) however, it is more probable that the cerium substitutes into the trivalent gadolinium site than the monovalent site of sodium. Both

compounds show high scintillation light yield up to 20 % cerium doping level. Doping beyond this concentration causes a breakdown of the lattice where neither compound could be formed. The crystalline structure of the LiGdCl_4 samples was confirmed by PXRD. Only low crystallinity was obtained for the NaGdCl_4 samples.

A. Emission/Excitation Spectra

LiGdCl_4 and NaGdCl_4 excitation and emission spectra of the undoped and doped materials were obtained from room temperature photoluminescence measurements (Fig. 1 (a) and (b)). Both doped samples exhibit a double emission band between 310 and 410 nm typical of Ce^{3+} 5d-4f emission. The maxima of the doublet emission are found at approximately 345 nm and 365 nm for LiGdCl_4 and 350 nm and 370 nm for NaGdCl_4 .

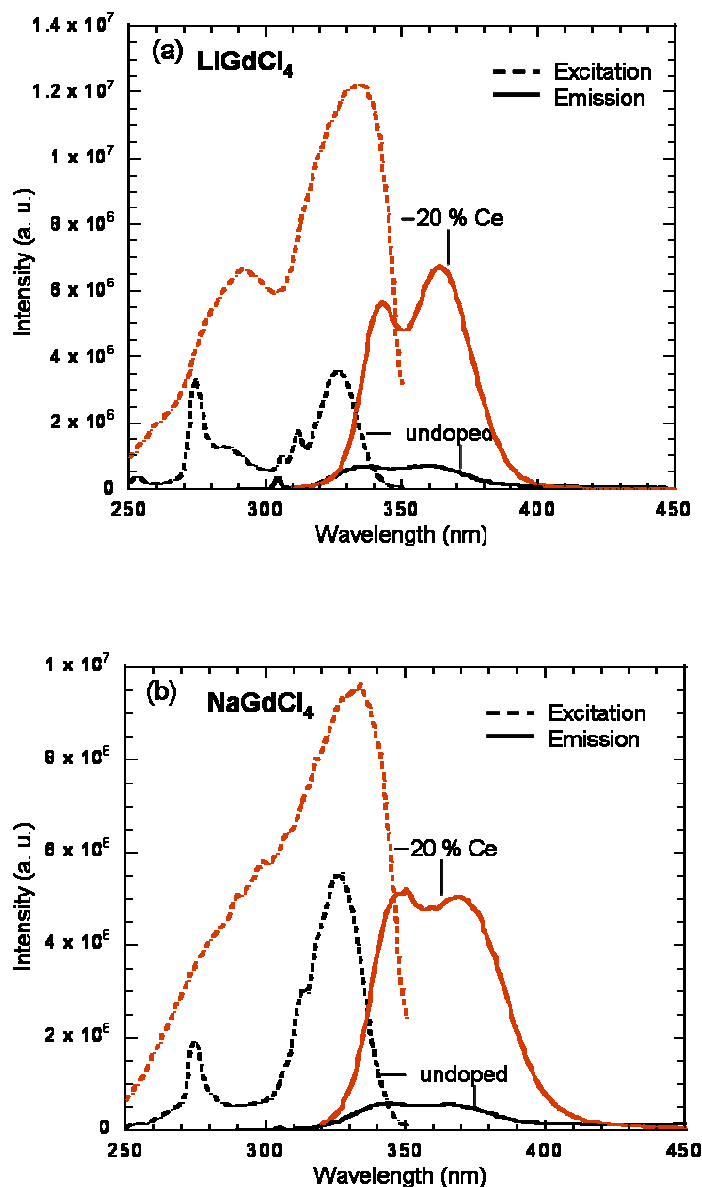


Fig. 1. Excitation and emission spectra of undoped and 20 % Ce^{3+} -doped (a) LiGdCl_4 and (b) NaGdCl_4 . Emission at 370 nm was used to generate the excitation spectra for all samples. An excitation wavelength of 270 nm was used to generate the emission spectra for the undoped materials. An excitation wavelength of 290 nm was used to generate the emission spectra for the doped LiGdCl_4 , whereas an excitation wavelength of 300 nm was used for the emission spectra of doped NaGdCl_4 . All spectra were recorded at room temperature.

The excitation spectra are characteristic of the 5d excitation bands of Ce^{3+} emission at room temperature with two broad bands between 250 and 350 nm. The maximum excitation is found at 335 and 330 nm for LiGdCl_4 and NaGdCl_4 , respectively. We note a significant overlap of the cerium excitation bands and the high-energy cerium emission band, indicating a possible self-absorption mechanism. In the undoped materials, the doublet cerium-related emission is weak but still present, indicating that some cerium was present, most likely in the GdCl_3 used for the synthesis of the samples. Additional bands are observed for both undoped samples around 310 nm in emission and 275 nm in excitation. These bands correspond to the optical signature of Gd^{3+} ions.

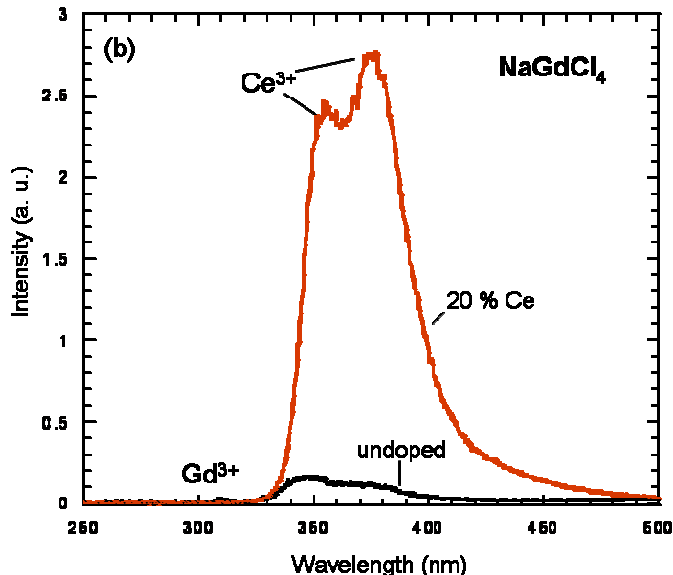
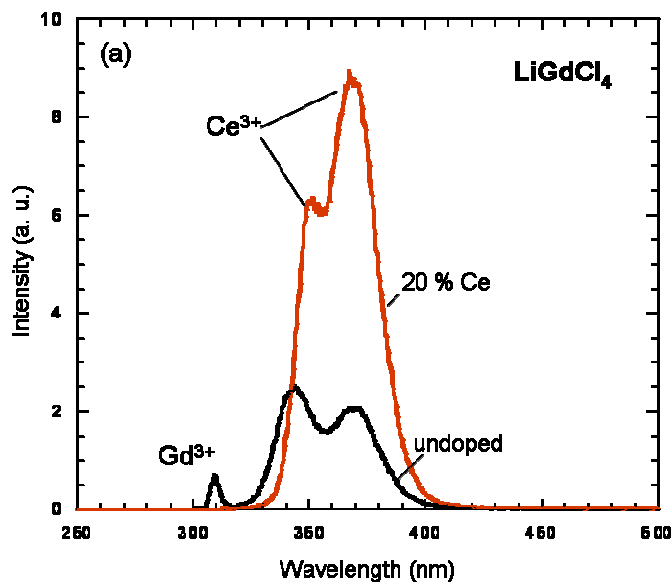


Fig. 2. Room temperature X-ray excited luminescence spectra of (a) LiGdCl_4 and (b) NaGdCl_4 . Gadolinium emission peaks at ~ 310 nm are evident in the undoped samples in both materials.

Fig. 2 (a) and (b) present the X-ray excited emission spectra at room temperature of the undoped and 20 % doped LiGdCl_4 and NaGdCl_4 , respectively. These spectra show similar characteristics as those found under optical excitation: Ce^{3+} 5d-4f emission and residual emission from Gd^{3+} ions.

In addition, the X-ray excited emission spectra highlights two new features. For the $\text{LiGdCl}_4:\text{Ce}$, the self-absorption mechanism can be observed in the reduction of the high energy part of the doublet from low to high cerium concentrations and in the relative intensity of the two peaks.

In the case of $\text{NaGdCl}_4:\text{Ce}$ there is a new broad-band emission at low energy. The band centered around 425 nm overlaps with the cerium emission. Very similar features were observed for $\text{LaCl}_3:\text{Ce}^{3+}$ and attributed to self-trapped exciton emission (STE) [27]-[28]. In LaCl_3 , the presence of STEs was established by X-ray excited electron-paramagnetic-resonance measurements and accredited to an *out-of-plane* self-trapped exciton formed by two nearest Cl^- neighbors [29]. Since we are investigating chloride compounds as well, a similar configuration could exist.

B. Estimated Luminosities and Decays

Estimated luminosities of $\text{LiGdCl}_4:\text{Ce}$ and $\text{NaGdCl}_4:\text{Ce}$ were obtained from pulsed x-ray (PXRL) measurements by normalizing their measured values to that of polycrystalline $\text{YAP}:\text{Ce}$. The total light yields increase in both compounds as the Ce^{3+} concentrations increase from 0 to 20 % (Fig. 3).

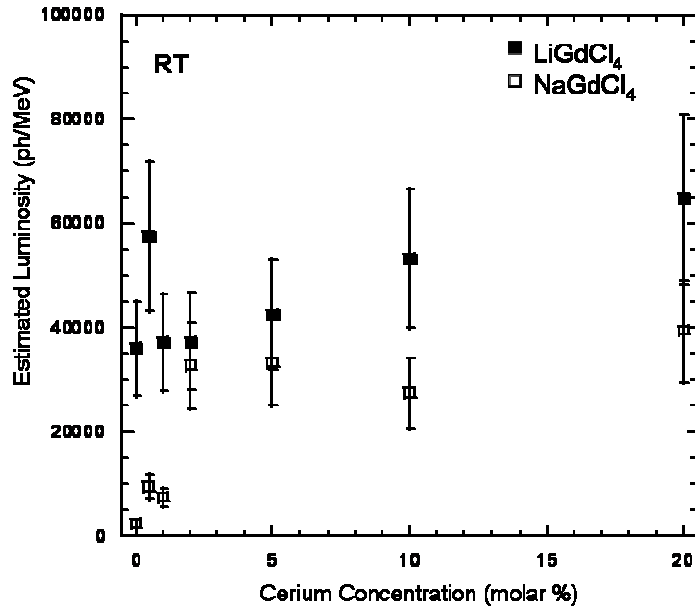


Fig. 3. Estimated luminosity of LiGdCl₄ and NaGdCl₄ at room temperature as a function of cerium concentration.

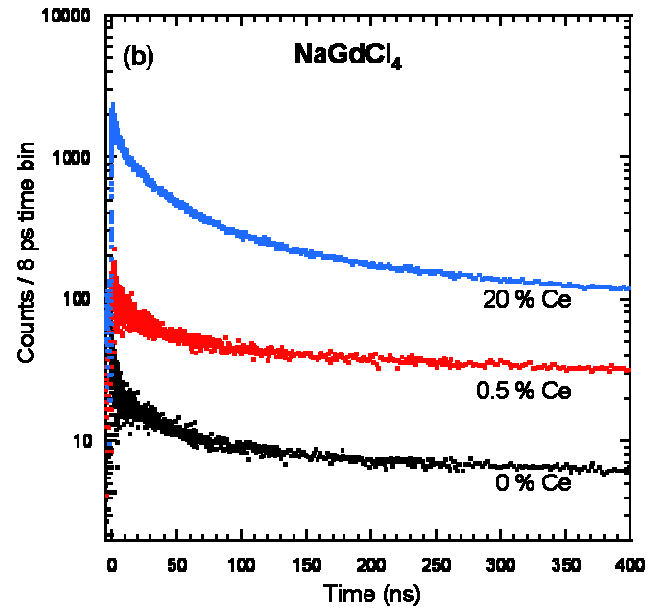
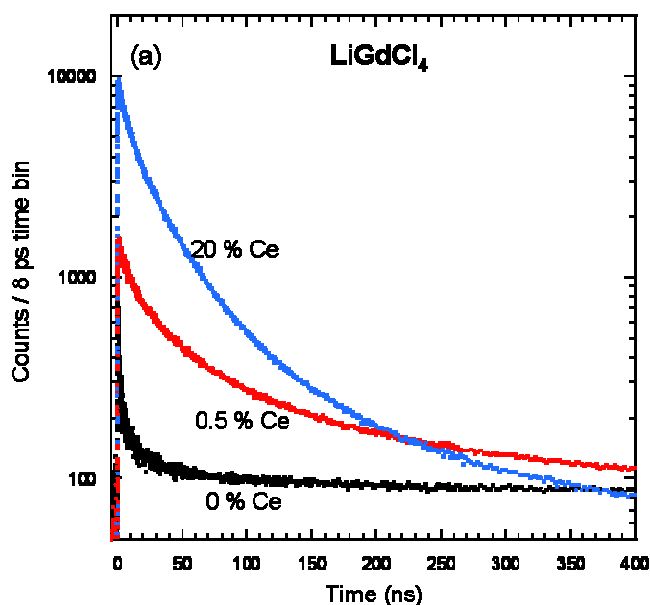


Fig. 4. Room temperature pulsed X-ray decay curves of (a) LiGdCl₄:Ce and (b) NaGdCl₄:Ce, with [Ce] = 0, 0.5 and 20 %.

Decay curves were also obtained from PXRL measurements of the undoped and cerium-doped variations of LiGdCl₄ and NaGdCl₄. The curves were fitted to determine the decay components of the light yield (Fig. 4).

The decay curves of each sample of these two compounds are multiexponential. We divide the decay curve into three time bands: $\tau \leq 50$ ns, τ in the range of 50 - 1000 ns, and $\tau > 1000$ ns. Fig. 5 indicates that as the cerium concentration increases the fraction of light decaying within 50 ns, while the 50 - 1000 ns component stays relatively constant for 2 % < [Ce] < 20 %, for both compounds.



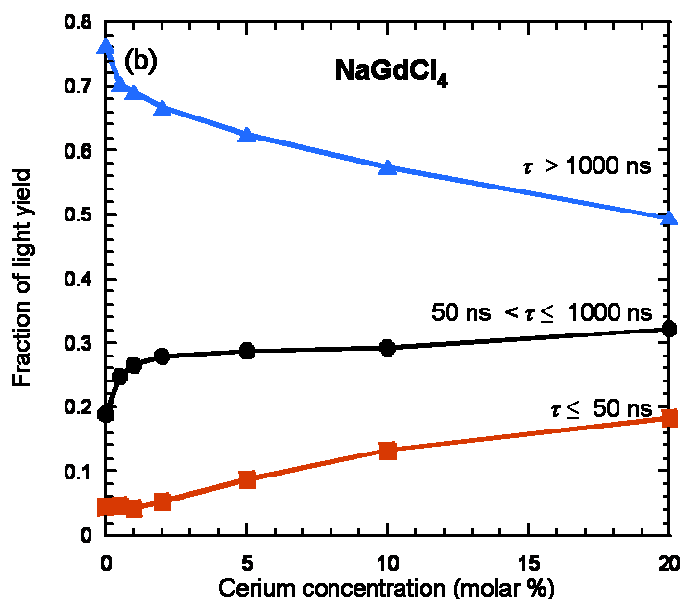
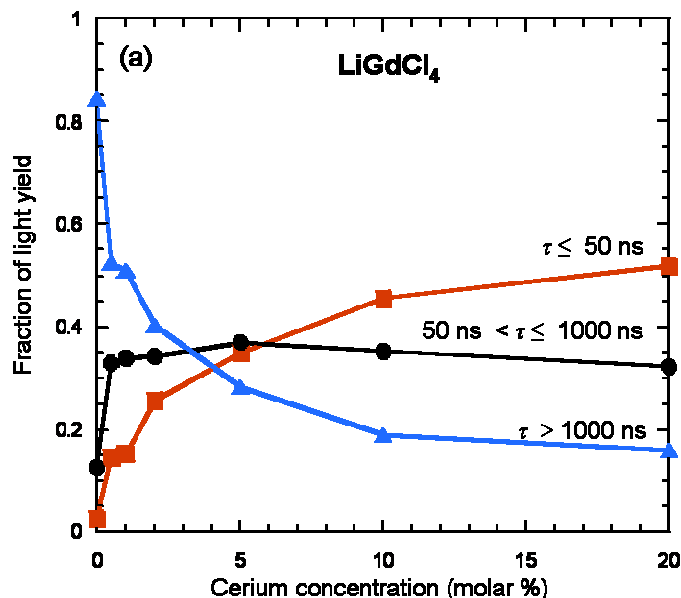


Fig. 5. Room temperature decay component trends of the (a) LiGdCl₄:Ce and (b) NaGdCl₄:Ce scintillation light as a function of cerium concentration.

C. Hygroscopic Nature

The hygroscopicity of powder samples of undoped GdCl₃, LiGdCl₄, and NaGdCl₄ were analyzed (Fig. 6). The presence of lithium cations caused the LiGdCl₄ to absorb more moisture from the air than GdCl₃ and NaGdCl₄. Powder X-ray diffraction analysis of the resultant products after 140 hrs of exposure to air indicated the presence of GdCl₃·6H₂O in all three materials. NaGdCl₄ is the least hygroscopic of the three.

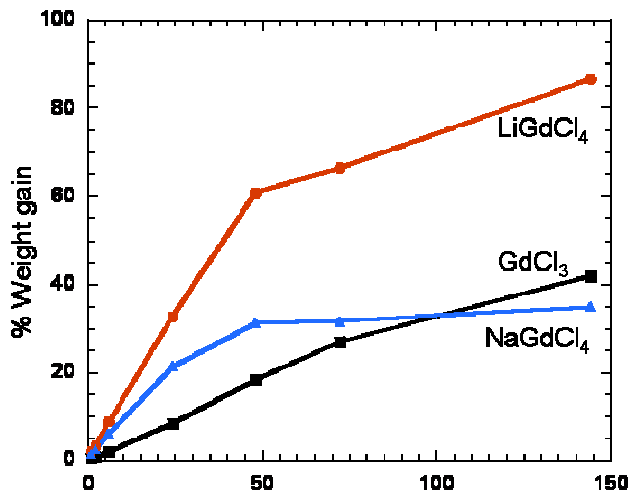


Fig. 6. Hygroscopic nature of GdCl₃, LiGdCl₄, and NaGdCl₄ upon exposure to air over time measured by weight gains.

IV. DISCUSSION

From the experimental results discussed in the previous section, two observations can be made of the scintillation properties of LiGdCl₄:Ce and NaGdCl₄:Ce:

- 1) The scintillation emission spectra are cerium-like and the luminosities increase gradually as the cerium concentration increases in LiGdCl₄ and increases sharply as cerium concentration is increased to 2% doping then levels off for NaGdCl₄ (Fig. 3). The highest light output was found at 20% doping for both compounds. At this concentration, the luminosity is approximately 3.6 and 2.2 times that of YAP:Ce for LiGdCl₄:Ce and NaGdCl₄:Ce, respectively.
- 2) The scintillation time responses for these materials display several decay components (Fig. 4). The contribution to these components are cerium concentration dependent (Fig. 5 and Table I). For the LiGdCl₄:Ce the percentage of fast component (≤50 ns) increases from 2 to 52% with the cerium concentrations increasing from 0 to 20%. For NaGdCl₄:Ce, the fast components increase from 4 to 18%, whereas the slower components increase for cerium concentrations less than 2% and then remain relatively constant at concentrations greater than 2%.

These results cannot be explained by a simple scintillation mechanism involving only the sequential capture of electron/hole pairs at a cerium ion. Based on the evidence of gadolinium emission in our materials, we propose that mechanisms represented schematically in Fig. 7 could be occurring in our cerium doped materials.

Path A represents the direct ionization of cerium by X-rays or sequential capture of electron/hole pairs at a cerium ion that leads to prompt cerium excitation and emission. This is what is observed in the fast components of the scintillation decays.

TABLE I
PHYSICAL AND LUMINESCENCE PROPERTIES OF AGdCl₄ SCINTILLATORS
(A= Li, Na)

Candidate	Density (g/cm ³)	Ce ³⁺ conc. (%)	Emission wavelength (nm)	Relative luminosity (x YAP : Ce)	Estimated luminosity (ph/MeV) ^b	Decay fractions $\tau \leq 50$ ns	Decay fractions $50 \text{ ns} < \tau \leq 1000$ ns	Decay fractions $\tau > 1000$ ns
LiGdCl ₄	3.70	0 ^a	310(Gd ³⁺), 340, 360	2.0	36,000	0.02	0.13	0.85
		0.5	340, 365	3.2	57,500	0.15	0.33	0.52
		1	340, 365	2.1	37,100	0.15	0.34	0.51
		2	345, 365	2.1	37,300	0.26	0.34	0.40
		5	345, 365	2.4	42,500	0.35	0.37	0.28
		10	345, 365	2.9	53,300	0.46	0.35	0.19
		20	345, 365	3.6	64,600	0.52	0.32	0.16
NaGdCl ₄	3.44	0 ^a	305(Gd ³⁺), 345, 365	0.1	2,400	0.05	0.19	0.76
		0.5	350, 370	0.5	9,600	0.05	0.25	0.70
		1	350, 370	0.4	7,400	0.04	0.27	0.33
		2	350, 370	1.8	32,700	0.05	0.28	0.67
		5	350, 370	1.9	33,400	0.09	0.29	0.62
		10	350, 370	1.5	27,400	0.13	0.29	0.58
		20	350, 370	2.2	39,400	0.18	0.32	0.50

^aPossible cerium impurity from starting reagents. ^bBased on YAP : Ce luminosity of 18,000 ph/MeV (average of luminosities in [21]-[25]); Luminosities have a possible error of 25% owing to counting statistics

Gd₂SiO₅:Ce [30]. In this material, these scintillation properties are attributed to non-radiative transfers from Gd³⁺ to Ce³⁺.

Although path D is possible, the only evidence of excitonic emission at room temperature is the broad shoulder at 425 nm in the X-ray excited emission spectrum of NaGdCl₄:20 % Ce (Fig. 2 (b)). Therefore, path D is displayed as only a small component of the emission in our diagram.

In all cases, the theoretically allowable scintillation light yield is not fully realized because of nonradiative loss processes (indicated by path C).

V. CONCLUSION

Two new inorganic alkali gadolinium halide based scintillators were found. LiGdCl₄:20 % Ce³⁺ exhibits scintillation light yield that is 3.6 times that of YAP:Ce polycrystalline powder, with a cerium-like component decay time of 33 ns. NaGdCl₄:20 % Ce³⁺ exhibits scintillation light yield of 2.2 times that of YAP:Ce, with a cerium-like component decay time of 26 ns. Slower decay components are present in the cerium emissions, which are tentatively attributed to a delayed energy transfer mechanism such as Gd³⁺ to Ce³⁺ or STE to Ce³⁺. In addition to their potential use as gamma-ray detectors these materials could have applications in neutron detection devices due to the presence of lithium and/or gadolinium in their lattice.

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Scintillation Mechanisms

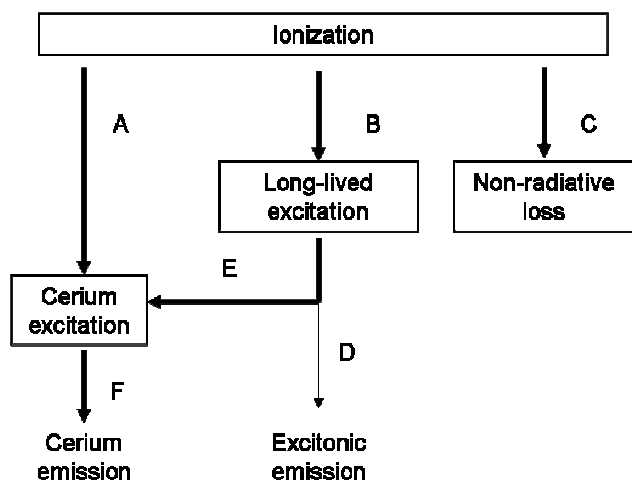


Fig. 7. Diagram detailing proposed scintillation mechanisms to account for the luminescence properties exhibited by cerium-doped LiGdCl₄ and NaGdCl₄. The slow cerium emission originates from path E, where slow emission feeds into cerium excitation.

Path B illustrates the ionization of long-lived luminescent centers, such as Gd³⁺ or self-trapped excitons. From path B, either path D or E can occur in principle. To account for the slow Ce³⁺ emission seen in our cerium doped materials, we propose path E, where the long-lived emission occurring from path B feeds into the cerium excitation and results in slower cerium emission. This mechanism is concentration dependent. A similar scintillation model has been used to explain the slow decay components and cerium concentration dependence of

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