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Effects of alkylamine chain length on perovskite nanocrystals after washing and perovskite light-emitting diodes

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Organic–inorganic hybrid lead halide perovskite nanocrystals (PeNCs) have received great attention as a light source for perovskite LEDs (PeLEDs) owing to the superior optical properties. However, PeNCs typically use octylamine (OAm) as capping ligands which have insulating properties. Exploring a desirable short alkylamine instead of OAm is required for the improvement of PeLEDs. Here, as one of the strategies to solve this issue, the effects of alkylamine chain length for optical properties of PeNCs and PeLED characteristics are investigated. Pentylamine is an optimal short alkylamine and precipitate luminescent PeNCs with high PLQY values of 90%. Importantly, pentylamine maintains a relatively high PLQY of 48% after spin-coating, due to the durability pentylamine has to ethyl acetate as a washing solvent. PeNCs capped with pentylamine also demonstrate an external quantum efficiency of over 1% with luminance of over 2000 cd cm^{-2}, indicating that pentylamine has the potential to overcome the insulator properties of PeNC thin film. © 2019 The Japan Society of Applied Physics

1. Introduction

Organic–inorganic hybrid lead halide perovskite $APbX_3$ ($A = CH_3NH_3$, CNH_3CNH_5, $X = Cl$, Br, and I) nanocrystals (PeNCs) have been given great attention as a light source for versatile opto-electrical applications such as photodetectors[1,2] photovoltaics,[3–5] LEDs,[6–14] and wide color gamut displays owing to their high photoluminescence quantum yields (PLQYs, 70%–100%),[15–21] narrow FWHM (19 nm–36 nm),[15–17] color tunability adjusted by halide component in an entire visible range (405 nm–730 nm),[15] as well as their solution processability.[15,18] Highly luminescent methylammonium lead tri-bromide ($MAPbBr_3$) PeNCs are commonly prepared by ligand-assisted reprecipitation (LARP)[15,17] using octylamine (OAm) as a capping ligand to modify the surface defects. LARP is a simple protocol to precipitate stable PeNC colloids through mixing the precursor solutions and poor medium. Surprisingly, the PLQY has reached up to nearly 100%[20,21] using LARP with capping ligand modifications. Additionally, LARP can easily enable highly luminescent PeNCs for the production of perovskite LEDs (PeLEDs) and has demonstrated the electroluminescence with high color purity to be comparable to the colloidal PeNCs.[8–11] Recent optimizations of device formation protocols have led to drastic improvements of green color emissive $MAPbBr_3$ NCs PeLEDs with EQE improvements from 3.8%[11] to 12.9%[9] by suppressing Auger non-radiative recombination. Although octylamine provides sufficient colloidal stability due to its steric hindrance, the PeNC thin films concurrently exhibit insulating properties which hinder ideal carrier injection and transportation. Suppressing insulating properties of octylamine is one of the strategies to improve PeLED performance. Therefore, it is significant for the development in this field to explore desirable and conductive alkylamines that are suitable for preparing stable $MAPbBr_3$ PeNC colloids and PeLEDs instead of long-chain OAm.

As mentioned above, overcoming the trade-off between PeNC colloidal stability and conductive properties of PeNC thin films is required. Notably, conjugated short capping amine ligands are commonly used due to the molecular electric orbital which is attractive for opto-electric applications. For instance, as a conjugated amine, 3-phenyl-2-propen-1-amine (PPAm) was employed as a capping ligand for precipitation instead of OAm and prepared uniform PeNCs 4 nm in diameter and 63% PLQY.[22] PPAm capped PeNCs strikingly improved the carrier mobility of PeNC thin films from 0.19 to 4.3 cm^2 V^{-1} s^{-1} compared with OAm capped PeNCs, which subsequently enhanced device characteristics due to the suppressing insulator characteristics of PeNC thin films. Similarly, conjugated aromatic acids are also effective capping ligands to suppress insulator characteristics. $MAPbBr_3$ PeNCs prepared using benzylamine (BZA) and benzoic acid (BA) improved PLQYs up to 86% while retaining colloidal stability, which led to lower impedance of $MAPbBr_3$ PeNC thin films.[23] Although the strategy using conjugated capping ligands shows improvement in the insulator characteristics of $MAPbBr_3$ PeNC thin films, to shorten the length of alkylamine is one of the simplest approaches. Particularly, exploring the optimal short alkylamine is still a challenging subject considering the trade-off between colloidal instability and electric properties. Moreover, for incorporation of $MAPbBr_3$ NCs into PeLEDs, investigation of durable capping ligands during the washing process is required because washing is essential to form uniform PeNC thin films and develop efficient PeLEDs.

Here, we report the effects of alkylamine chain length varying from ethylamine to heptylamine on optical properties of $MAPbBr_3$ PeNCs after washing and the PeLED performance. This investigation shows that PeNCs optical properties depend on the capping ligand when using ethyl acetate as the washing solvent since the length of the alkyl chain affects the PLQY after the washing process. Considerably short-amines such as ethylamine and propylamine deteriorate the PLQYs to approximately 18% and 26% after washing due to insufficient steric hindrance to stabilize colloids, while butylamine retains PLQY at 50% after spin-coating. Moreover, the PeLEDs using pentylamine exhibits up to 1% EQE with 2000 cd cm^{-2}.

2. Experimental methods

2.1. Materials

Lead (II) bromide powder ($PbBr_2$, 99.999%) was purchased from Aldrich. methylammonium (MABr, 98.0%) was synthesized as follows. MABr was synthesized by reacting 7 ml of HBr (48% in methanol, Wako Pure Chemical Co. Ltd.) with 30 ml of methylamine (40% in methanol, Wako Pure Chemical Co. Ltd.) in a 250 ml round-bottom flask. After evaporating the solvents, we collected the white precipitates. Then, we purified and recrystallized the products by using ethanol and diethyl ether. 1-Methyl-2-pyrrolidone (NMP), Chlorobenzene, Oleic acid and alkylamine (Ethylamine to Heptylamine) were purchased from Wako Pure Chemical Co. Ltd. Poly(3,4-ethylenedioxythiophene):poly-(styrenesulfonate) (PEDOT:PSS, AI4083) and poly(4-butylphenyl-diphenyl-amine) (poly-TPD) and Tris-[1-phenyl-1H-benzimidazole] (TPBi) were purchased from Clevious and American Dye Source and e-Ray Optoelectronics Technology Co., Ltd. All the chemicals were used without purification.

2.2. Preparation of $MAPbBr_3$ NCs

$MAPbBr_3$ NCs were fabricated following the ligand-assisted reprecipitation with modification. 3.6 mg MABr and 14.7 mg $PbBr_2$ was dissolved in 1 ml of NMP as a good solvent contained 4 μl of alkylamines (ethyl, propyl, butyl, pentyl, hexyl, heptyl) and 5 μl of oleic acid. 1 ml of precursors solution was dropped into 25 ml of vigorously stirring chlorobenzene as a poor solvent. Obtained dispersions were centrifuged at 6000 rpm for 10 min.

2.3. Washing of $MAPbBr_3$ NCs

Ethyl acetate was added to $MAPbBr_3$ NCs dispersion in a volume ratio of 2:1, and were centrifuged at 12 000 rpm at 10 min. The precipitate washed with ethyl acetate was collected and dispersed in toluene with concentration of 10 mg ml^{-1}.

2.4. Fabrication of perovskite LEDs

PEDOT:PSS (55 wt% containing in AI4083) was spin-coated onto cleaned ITO-coated substrate and annealed at 150 °C for 10 min resulting in a 40 nm thick layer. For the film formation of hole injection layer, Poly-TPD was dissolved in chlorobenzene with a concentration of 4 mg ml^{-1}. This solution was spin-coated onto PEDOT:PSS layer and annealed at 100 °C for 10 min resulting in a 20 nm thick layer. Colloidal $MAPbBr_3$ PeNCs (5 nm) was spin-coated onto poly-TPD at 2000 rpm for 30 s in N_2- filled glovebox. TPBi (50 nm), Liq (1 nm), and Al (100 nm) anode were deposited by thermal evaporated under high vacuum ($\sim 1 \times 10^{-5}$ Pa). Active area of the device was 2 mm^2.

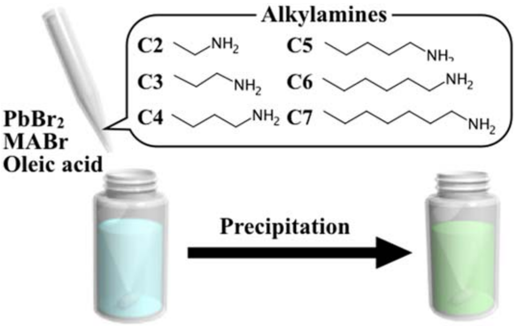


Fig. 1. (Color online) Schematic illustration of the colloidal $MAPbBr_3$ PeNCs prepared by LARP varying alkylamines from ethylamine to heptylamine.

2.5. Characterization

X-ray diffraction (XRD) patterns of the samples were obtained from in-plane diffraction using membrane filters and were measured on a Rigaku Smart Lab (using Cu Kα radiation at 45 kV and 200 mA). The samples were observed by a JEOL JSM-6700F scanning electron microscope (SEM) (accelerating voltage of 10 kV) and a JEOL JEM-2100F transmission electron microscope (TEM) (accelerating voltage of 200 kV). Visible absorption spectra of the samples were obtained on a JASCO V-670 spectrophotometer (detecting wavelength range of 400 to 600 nm). Photoluminescence (PL) spectra of samples were obtained with HORIBA FluoroMax-2 luminescence spectrometer (exciting wavelength of 370 nm and detecting wavelength range of 400 to 600 nm). Photoluminescence quantum yield (PLQY) was measured using a Hamamatsu C9920−01 integral sphere system. Electroluminescence spectra were recorded using Hamamatsu PMA-11 photonic multichannel analyzer. The current-density−voltage and luminance−voltage characteristics were measured using a Keithley source measure unit 2400 and a Minolta CS200 luminance meter, respectively.

3. Results and discussion

Colloidal $MAPbBr_3$ PeNCs capped with alkylamines varying from ethylamine to heptylamine have been successfully synthesized using the LARP with some modifications, which prepare superior luminescent the PeNCs through mixing a precursor solution containing alkylamines and oleic acid with toluene as a poor medium (Fig. 1). Even though using the alkylamines shorter than octylamine, bright luminescent green color [Fig. 2(a)] was clearly observed from the PeNCs under UV irradiation at 365 nm. The PeNCs show a single PL peak at 514–522 nm [Fig. 2(b)] which is a similar emission energy to that of bulk $MAPbBr_3$.[24–26] Except for ethylamine and propylamine, the PeNCs show high PLQYs (83%–90%) with narrow FWHM close to 20 nm (Table I), which is comparable to the optical properties from the PeNCs using OAm.[7,8] Although ethylamine and propylamine respectively have relatively low PLQY values of 67% and 83%, respectively (Table I) compared to the longer chain ligands, they precipitate stable colloidal PeNCs. This implies that ethylamine and propylamine are an insufficient steric hindrance to stabilize the colloidal PeNCs, which causes to inferior PLQYs. Additionally, in order to evaluate the effects of alkylamine chain length on the crystal structure of precipitated PeNCs, XRD measurements were conducted.

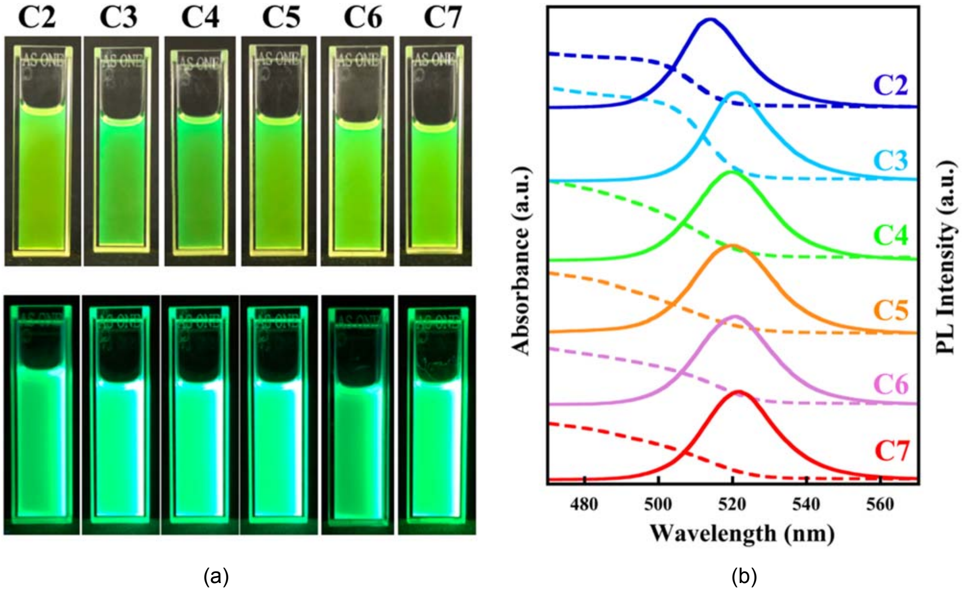


Fig. 2. (Color online) (a) Photographs and (b) Absorption and PL spectra of the colloidal $MAPbBr_3$ PeNCs prepared by using alkylamies from ethylamine (C2) to heptylamine(C7).

Table I. PL peaks, FWHM, and PLQYs of $MAPbBr_3$ PeNCs prepared by ethylamine(C2) to heptylamine(C7).

Carbon number	λ_{PL} nm^{-1}	FWHM nm^{-1}	PLQY/%
C2	514	20	67.3
C3	521	20	83.0
C4	520	22	89.4
C5	520	22	89.5
C6	522	22	89.0
C7	521	23	90.8

From XRD spectra, all samples obviously show strong peaks corresponding to (100), (110), and (200) diffraction planes (Fig. 3), which indicates a cubic perovskite crystal structure.[24,27,28] Thus, the decrease in the PLQY values of $MAPbBr_3$ PeNCs capped with ethylamine and propylamine cannot be attributed to the crystal structures.

The effect of decreasing PLQYs with the alkylamines chain length is more obvious after the washing. The PLQY measurements of the PeNCs after washing show that from pentylamine to heptylamine a high PLQY of over 65% is maintained after washing three times (Fig. 4) with ethyl acetate. In contrast, propylamine and butylamine decreased PLQYs from 89.4% to 30.7% and 83.0% to 26.3%, respectively. Moreover, ethylamine drastically decreased PLQYs to 18.3%, which is severely worse than before washing. These results show that the durability of alkylamines during washing clearly depends on the chain length. Ligands longer than butylamine provide sufficient steric hindrance to have a durability to ethyl acetate used as a washing solvent and are comparable to octylamine. In particular, the washing is a significant step to adjust the concentration of the PeNCs for preparing uniform films and to remove impurities such as excess capping ligands and polar solvent from the as-prepared colloidal PeNCs. Considering the durability of the alkylamines, pentylamine is a candidate for a short alkylamine because it is maintaining high PLQYs after washing and suppressing the insulator properties of PeNC thin films.

Evaluation of the PLQYs from the PeNC films is also critical, the PeNC films were simply formed by spin-coating using the washed colloidal PeNCs. Importantly, the PLQYs of the films show a trend similar to the durability of

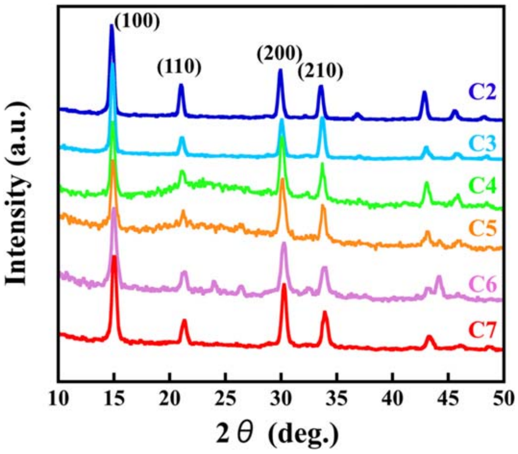


Fig. 3. (Color online) XRD patterns of $MAPbBr_3$ PeNCs prepared by using from ethylamine(C2) to heptylamine(C7) as a capping ligand.

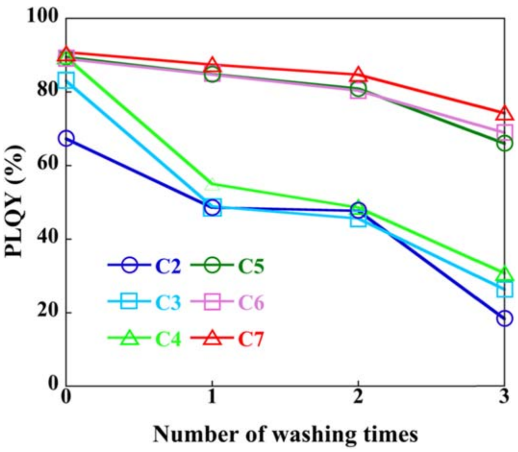


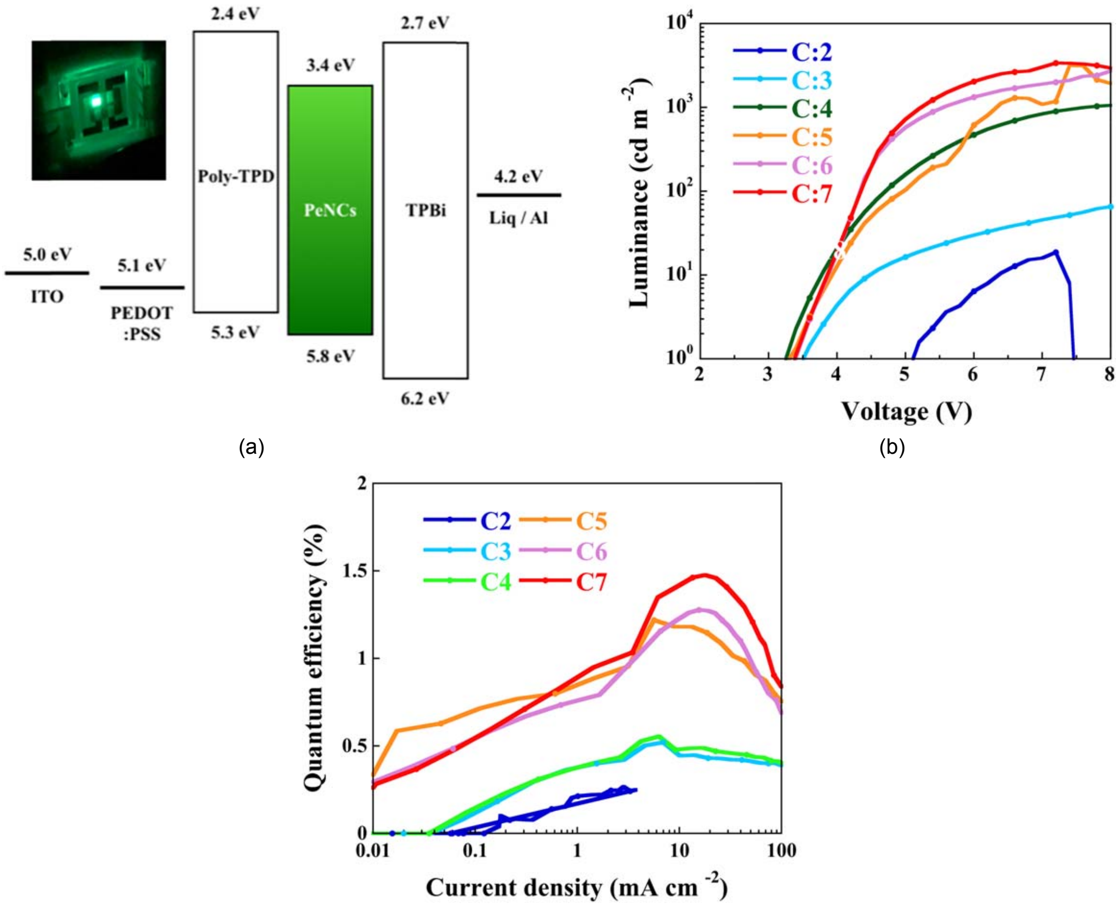
Fig. 4. (Color online) PLQYs of the colloidal $MAPbBr_3$ PeNCs as a function of washing cycles.

Table II. PL peaks, FWHM, PLQYs of the $MAPbBr_3$ PeNC films.

Carbon number	λ_{PL} nm^{-1}	FWHM nm^{-1}	PLQY/%
C2	518	21	10.5
C3	518	25	42.8
C4	523	26	48.9
C5	523	24	47.8
C6	520	29	53.4
C7	524	23	65.2

alkylamine to ethyl acetate (Fig. 4). Due to the aggregation of the PeNCs after film formation, it causes quenching by interaction, resulted in PLQYs drastically decreased than the colloidal states of PeNCs.[29,30] Ethylamine in particular has a much worse PLQY that drops from 49% to 10% after film formation. This implies that there are more surface defects caused by the desorption of ethylamine after washing. The exposure of surface defects causes the quenching attributed to strong interactions of PeNCs with each other on the film and results in a PLQY drop to 10% (ΔPLQY = 40%) (Table II). Therefore, undesirable steric hindrance leads to decreases in the performance of PeLEDs. In contrast, alkylamines longer than ethylamine exhibit 43%–65% PLQYs in the PeNC film state, which are considerably higher values than ethylamine (Table II). They are useful capping ligands for maintaining the PLQYs after film formation and for applying to PeLEDs.

In order to discuss the relationship between the chain length of alkylamine and the PeLEDs characteristics, PeLEDs were fabricated using PeNCs capped with ligands ranging from ethylamine to heptylamine in the following structure: indium tin oxide (ITO, 130 nm)/poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS, 40 nm)/poly(4-butylphenyl-diphenyl-amine) (poly-TPD, 20 nm)/$MAPbBr_3$ PeNCs (5 nm)/tris-(1-phenyl-1H-benzimida-zole) (TPBi, 50 nm)/lithium 8-quinolate (Liq, 1 nm)/Al (100 nm), [Fig. 5(a)]. Figures 5(b) and 5(c) show luminance–voltage characteristic and EQE–current-density of PeLEDs of varying alkylamines and the values of device characteristics which are summarized in Table III. Regarding PeLEDs using ethylamine and propylamine, appropriate device characteristics were not observed [Fig. 5(b)] due to severely low PLQY values of the films (Table II) and worse morphologies of the films. On the other hand, from butylamine to heptylamine, the PeLEDs exhibited favorable luminance–voltage characteristics with low turn-on voltages (3.4–3.5 V) [Fig. 5(b), Table II]. Heptylamine showed the highest EQE value of 1.5% (Table III) owing to high PLQY values in PeNC thin film state, while pentylamine and hexylamine also exhibited EQEs over 1% with luminance over 2000 cd cm^{-2}. This EQE values for all devices are relatively lower compared with the previous reports,[9,12] this reducing the PeLEDs performances might be because of the insulating properties of OAc in perovskite films. Further improvement is required however this implies that pentylamine which is relatively shorter than octylamine as a typical capping ligand has a potential for sufficient steric hindrance to demonstrate efficient PeLEDs.



(a)

(b)

(c)

Fig. 5. (Color online) $MAPbBr_3$ PeLEDs performance. (a) energy diagram and Photograph of the PeLED, (b) luminance−voltage characteristics and (c) external quantum efficiency–current-density characteristics.

Table III. Summary of the $MAPbBr_3$ PeLEDs performances with different chain length alkylamines.

Carbon number	EQE/%	V_{on} V^{-1}	$L_{max}/cd \cdot cm^{-2}$	λ_{EL} nm^{-1}	λ_{FWHM} nm^{-1}
C2	0.104	5.2	18.7	—	—
C3	0.190	3.5	58.2	—	—
C4	0.489	3.3	1090	—	—
C5	1.08	3.3	2080	529	27
C6	1.26	3.4	2770	526	24
C7	1.47	3.4	2790	527	24

4. Conclusions

We have investigated the effects of alkylamine chain length by varying from ethylamine to heptylamine for optical properties of the as-prepared PeNCs, after washing and the PeLED characteristics. We have successfully prepared the luminescent colloidal PeNCs with high PLQY values (83%–90%) with narrow FWHM close to 20 nm ranging from butylamine to heptylamine, which are shorter than octylamine as a typical capping ligand. Notably, from pentylamine to heptylamine, we maintained high PLQYs over 65% after washing three times and relatively high PLQYs over 47% after film formation because the alkylamines show durability to ethyl acetate as a washing solvent. Moreover, the durable alkylamines also exhibited EQEs of over 1% with luminance of over 2000 cd cm^{-2} from a simple device structure. This report shows the possibility of short alkylamine as a capping ligand for desirable electronic injection and transportation of PeNC thin films.

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