Synthesis of Asymmetric Anthracene Derivatives and Their Application for Blue Organic Light-emitting Diodes

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Aryl-substituted asymmetric anthracene blue host materials, 10-naphthalene-2-yl-9-phenyl-2triphenylsilylanthracene (6) and 9-(4-(*tert*-butyl)phenyl)-10-(2-methyl-5-(naphthalen-1-yl)phenyl)anthracene (7) were synthesized and characterized. Asymmetric anthracene derivatives 6 and 7 possessed high thermal stabilities that are suitable as emitting layer materials for blue organic light-emitting device. The electroluminescence emission maximum of BD-142-doped (7 wt %) two devices exhibited at 464 nm (for 6) and 472 nm (for 7), respectively. All devices showed good performances as blue host materials. The 7-based device especially displayed higher device performances in terms of brightness ($L_{max} = 37\ 090\ cd/m^2$), luminous current density (7.26 cd/A), and external quantum efficiencies (5.80%) than those of 6-based device.

Keywords: Anthracene, OLED, Blue-emitter, Host material, Electroluminescence

Introduction

Organic light-emitting diodes (OLEDs) have a number of attentions because of their high luminance, fast response time, broad viewing angle, and full-color realization.^{1,2} In order to achieve full-color images, OLEDs require highly stable, efficient, and extremely pure red, green, and blue emission. OLED materials with high performances for red and green colors developed to date,^{3,4} further enhancement for blueemitting materials is still required.⁵ The development of novel blue host materials with outstanding electroluminescence (EL) properties is very difficult owing to their inherent large band gap. A variety of blue host materials such as distyrylarylene⁶ and anthracene derivatives⁷⁻¹⁴ have been recently developed. Among them, anthracene-based materials are shown to be one of the most effective blue host materials because of their remarkable photophysical and electroluminescent properties.^{15–19} Despite their excellent electronic and optical properties, these organic fluorescent π -conjugated materials have some problems about device failure and thermal stability. To prevent such problems and improve thermal stabilities, silicon-cored (triphenylsilane)²⁰ or asymmetric anthracene derivatives^{21,22} containing bulky aryl groups were recently reported. For example, nonplanar anthracene compounds with bulky aryl substituent at the 10-position exhibited high thermal stability and outstanding EL property as blue host materials.²² Accordingly, the anthracene derivatives with the bulky substituent at the 9,10-position can lead to hindering close packing of the molecular structure and achieving highly efficient EL properties. In this regard, we prepared new blueemitting asymmetric anthracene derivatives such as 10naphthalene-2-yl-9-phenyl-2-triphenylsilylanthracene (6) and 9-(4-(*tert*-butyl)phenyl)-10-(2-methyl-5-(naphthalen-1yl)phenyl)anthracene (7) to provide improved thermal stability and excellent electronic/optical properties. Additionally, we aimed to introduce bulky aryl groups into the anthracene moieties to effectively reduce intramolecular π - π stacking as well as minimize concentration quenching, which can result in bright blue EL emission. Details of synthesis, characterization of asymmetric anthracene compounds (6 and 7), and their luminescence properties as blue host materials for OLEDs are described.

Experimental Section

General Considerations. All chemicals were purchased from Aldrich (St. Louis, MO, USA) and were used without any further purification. All solvents such as toluene, diethyl ether, and *n*-hexane were dried by distillation from sodium diphenylketyl under dinitrogen and were stored over 3 Å activated molecular sieves.²³ Spectrophotometric grade CH₂Cl₂ was used as received. 3-(4-Bromophenyl)phthalide,²⁴ 2-(10-(4-(tert-butyl)phenyl)anthracen-9-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane,²⁰ and 1-(3-iodo-4-methylphenyl)naphthalene²² were analogously prepared according to the reported procedures. All reactions were carried out under a nitrogen atmosphere. Deuterated solvents from Cambridge Isotope Laboratories were used after drying over activated molecular sieves (5 Å). NMR spectra were recorded at ambient temperature on Bruker Avance 400 spectrometer (400.13 MHz for ¹H and 100.62 MHz for ¹³C; Billerica, MA, USA) or Bruker DPX-300 NMR spectrometer (300.13 MHz for ¹H and 75.47 MHz for ¹³C) using standard parameters. Chemical shifts are given in ppm, and are referenced against external Me₄Si (¹H and ¹³C). The thermal properties of compound were investigated by TA Instrument TGA2940 system and TA Instrument DSC2910 system (New Castle. Delaware, USA) under a nitrogen atmosphere at a heating rate of 10 °C/ min. UV/Vis absorption and PL spectra were recorded on a Beckman PU 650 (San Francisco, CA, USA) and a Jasco FP-750 (Todyo, Japan), respectively, in CHCl₃ solvent with a 1-cm quartz cuvette at ambient temperature. Cyclic voltammetry (CV) measurements were performed using an AUTO-LAB/PGSTAT12 system (Artisan Technology Group, Champaign, IL, USA). Electroluminescent spectra were measured at Dongjin Semichem Co. Ltd. (Seoul, Republic of Korea).

Synthesis of 2-(3-bromobenzyl)benzoic acid (1). 3-(4-Bromophenyl)phthalide (10.5 g, 36.5 mmol), iodine (6.3 g, 24.8 mmol) and red phosphorus (5.7 g, 184.0 mmol) were dissolved in propionic acid (60 mL) and a distilled water (1.5 mL) was added. After stirring for 20 min at 100 °C, the solution was cooled to room temperature. The resulting solution was extracted with CHCl₃. The combined organic portions were dried over MgSO₄ and the solvent was removed under reduced pressure. A yellow solid **1** was obtained (8.8 g, 83.2%) without further purification. ¹H NMR (CDCl₃, 300.13 MHz): δ 8.10 (dd, *J* = 7.8 Hz, 1.2 Hz, 1H), 7.51 (td, *J* = 7.6 Hz, 1.4 Hz, 1H), 7.37 (d, *J* = 7.7 Hz, 1H), 7.33 (d, *J* = 1.4 Hz, 1H), 7.31 (s, 1H), 7.23 (d, *J* = 7.7 Hz, 1H), 7.16–7.09 (m, 2H), 4.42 (s, 2H).

Synthesis of 3-bromo-9,10-dihydro-9-oxoanthracene (2). 1 (8.8 g, 30.3 mmol) was slowly added to concentrated H_2SO_4 at 0 °C. After stirring for 1 h, the reaction was slowly allowed to warm to room temperature and stirred for 1 h. After quenching with water, the resulting solid was collected by filtration and successively washed with hot ethanol. A light gray solid 2 was obtained (6.9 g, 82.9%). ¹H NMR (CDCl₃, 300.13 MHz): δ 8.34 (d, J = 7.9 Hz, 1H), 8.21 (d, J = 8.4 Hz, 1H), 7.63 (s, 1H), 7.61–7.57 (m, 2H), 7.48 (d, J = 8.0 Hz, 1H), 7.46 (d, J = 7.8 Hz, 1H), 4.32 (s, 2H). ¹³C NMR (CDCl₃, 75.47 MHz): δ 183.43, 142.03, 139.76, 133.02, 131.66, 131.28, 130.84, 130.55, 129.34, 128.42, 127.97, 127.60, 127.25, 31.94.

Synthesis of 2-bromo-10-(naphthalene-2-yl)anthracene (3). A hexane solution of *n*-BuLi (34.9 mL, 55.8 mmol, 1.6 M solution in hexane) was slowly added to a solution of 2-bromonaphthalene (11.6 g, 55.8 mmol) in THF (390 mL) at -78 °C. After stirring for 1 h, a solution of 2 (12.6 g, 46.5 mmol) in THF (230 mL) was added to the reaction mixture and stirred for 30 min. The solution was slowly allowed to warm to room temperature. The completeness of the reaction was checked by TLC. After the reaction was quenched with 6 M aqueous solution of HCl (90 mL), the organic layer was separated, and the aqueous layer was extracted with ethyl acetate (150 mL). The combined organic portions were dried over MgSO₄ and concentrated under reduced pressure. Purification by column chromatography (eluent: CH₂Cl₂/*n*-hexane,

1:7 v/v) afforded **3** as a yellowish green solid (6.1 g, 33.8%). ¹H NMR (CDCl₃, 300.13 MHz): δ 8.41 (s, 1H), 8.22 (d, *J* = 1.8 Hz, 1H), 8.06 (s, 1H), 8.03 (s, 1H), 8.02–7.99 (m, 1H), 7.90–7.88 (m, 2H), 7.66 (d, *J* = 8.8 Hz, 1H), 7.61–7.57 (m, 2H), 7.54–7.46 (m, 3H), 7.37–7.35 (m, 1H), 7.35–7.31 (m, 1H). ¹³C NMR (CDCl₃, 75.47 MHz): δ 137.41, 135.63, 133.30, 132.78, 132.04, 131.91, 130.49, 130.09, 129.88, 129.20, 128.80, 128.59, 128.30, 128.05, 127.88, 126.96, 126.54, 126.36, 125.79, 125.72, 119.47.

Synthesis of 10-naphthalene-2-yl-2-triphenylsilylanthracene (4). A hexane solution of n-BuLi (10.8 mL, 17.3 mmol, 1.6 M solution in hexane) was slowly added to a solution of 3 (5.5 g, 14.5 mmol) in THF (75 mL) at -78 °C. After stirring for 30 min, a solution of triphenylsilyl chloride (4.3 g, 14.5 mmol) in THF (15 mL) was added to the reaction mixture and stirred for 30 min. The solution was slowly allowed to warm to room temperature. The completeness of the reaction was checked by TLC. After the reaction was quenched with water, the aqueous layer was extracted with ethyl acetate (30 mL). The combined organic portions were dried over MgSO4 and concentrated under reduced pressure. Purification by column chromatography (eluent: CH_2Cl_2/n -hexane, 1:3 v/v) afforded 4 as a yellowish green solid (6.7 g, 69.9%). ¹H NMR (CDCl₃, 300.13 MHz): δ 8.49 (d, J = 17.6 Hz, 1H), 8.33-8.28 (m, 1H), 8.04-7.87 (m, 5H), 7.69-7.62 (m, 8H), 7.58-7.55 (m, 4H), 7.50-7.33 (m, 11H). ¹³C NMR (CDCl₃, 75.47 MHz): δ 138.90, 136.44, 135.40, 134.20, 134.05, 133.35, 132.72, 131.43, 131.13, 130.94, 130.79, 130.50, 130.24, 130.12, 129.67, 129.44, 128.56, 128.03, 127.94, 127.92, 127.84, 127.40, 126.82, 126.64, 126.37, 126.16, 125.74, 125.14.

Synthesis of 9-bromo-10-naphthalene-2-yl-2-triphenylsilylanthracene (5). A solution of 4 (6.2 g, 11.0 mmol) in N,N-dimethylformamide (DMF; 620 mL) was slowly added to a solution of N-bromosuccinimide (NBS; 2.1 g, 12.1 mmol) in DMF (70 mL). The completeness of the reaction was checked by TLC. After the reaction was quenched with water, the resulting solid was collected by filtration and successively dissolved in CH2Cl2. The combined organic portions were dried over MgSO4 and concentrated under reduced pressure. Purification by column chromatography (eluent: CH_2Cl_2/n -hexane, 1:3 v/v) afforded 5 as a yellow solid (4.7 g, 67.3%). ¹H NMR (CDCl₃, 300.13 MHz): δ 8.91 (s, 1H), 8.61 (d, J = 8.9 Hz, 1H), 8.04–7.97 (m, 3H), 7.90-7.86 (m, 2H), 7.67-7.64 (m, 7H), 7.59-7.55 (m, 4H), 7.46-7.37 (m, 11H). ¹³C NMR (CDCl₃, 75.47 MHz): δ 138.14, 137.37, 136.45, 135.69, 133.78, 133.64, 133.25, 132.79, 133.78, 133.64, 133.25, 132.79, 131.70, 131.41, 130.97, 130.36, 130.11, 129.77, 129.43, 129.17, 127.98, 127.39, 126.92, 126.55, 126.36, 126.19, 125.91, 123.73.

Synthesis of 10-naphthalene-2-yl-9-phenyl-2-triphenylsilylanthracene (6). Phenylboronic acid (0.90 g, 8.03 mmol), 5 (4.69 g, 7.30 mmol), $Pd(PPh_3)_4$ (0.17 g, 0.15 mmol), and K_2CO_3 (6.05 g, 43.80 mmol) were dissolved in toluene (80 mL) and a distilled water (20 mL) was added. After the solution was heated at reflux overnight,

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it was cooled to ambient temperature. After the reaction was quenched with water, the aqueous layer was extracted with CH₂Cl₂ (30 mL). The combined organic portions were dried over MgSO4 and concentrated under reduced pressure. Purification by column chromatography (eluent: CH₂Cl₂/n-hexane, 1:9 v/v) afforded **6** as a yellow solid (2.94 g, 63.1%). ¹H NMR $(CDCl_3, 300.13 \text{ MHz}): \delta 8.05 \text{ (d}, J = 8.4 \text{ Hz}, 1\text{H}), 8.02-7.98$ (m, 3H), 7.92–7.89 (m, 1H), 7.77–7.70 (m, 3H), 7.63–7.56 (m, 3H), 7.51-7.47 (m, 7H), 7.42-7.39 (m, 3H), 7.38-7.36 (m, 2H), 7.35-7.34 (m, 2H), 7.34-7.33 (m, 2H), 7.31-7.28 (m, 6H), 7.26 (s, 1H). ¹³C NMR (CDCl₃, 75.47 MHz): δ 138.33, 137.98, 137.87, 136.59, 136.42, 136.23, 133.97, 133.38, 132.74, 131.05, 130.97, 130.64, 130.36, 130.22, 130.14, 129.89, 129.53, 129.45, 129.21, 128.17, 128.05, 127.93, 127.87, 127.79, 127.26, 127.21, 126.96, 126.39, 126.17, 125.72, 125.38, 124.97.

Synthesis of 9-(4-(*tert*-butyl)phenyl)-10-(2-methyl-5-(naphthalen-1-yl)phenyl)anthracene (7). 2-(10-(4-(*tert*-Butyl)phenyl)anthracen-9-yl)-4,4,5,5-tetramethyl-1,3,2-

dioxaborolane (2.50 g, 5.72 mmol), 1-(3-iodo-4-methylphenyl)naphthalene (1.97 g, 5.72 mmol), Pd(PPh₃)₄ (0.33 g, 0.29 mmol), and K₂CO₃ (3.16 g, 22.90 mmol) were dissolved in toluene (80 mL) and a distilled water (30 mL) was added. After the solution was heated at reflux overnight, it was cooled to ambient temperature. After the reaction was quenched with water, the aqueous layer was extracted with CH₂Cl₂ (80 mL). The combined organic portions were dried over MgSO₄ and concentrated under reduced pressure. Purification by column chromatography (eluent: CH₂Cl₂/n-hexane, 1:10 v/v) afforded 7 as a white solid (1.70 g, 56%). ¹H NMR $(CDCl_3, 400.13 \text{ MHz})$: $\delta 8.17 (d, J = 3.75 \text{ Hz}, 1\text{H}), 7.90 (d, J = 3.75 \text{ Hz})$ J = 3.75 Hz, 1H), 7.84 (d, J = 4.0 Hz, 2H), 7.77 (d, J = 4.0 Hz, 2H), 7.58–7.64 (m, 4H), 7.52 (d, J = 3.5 Hz, 1H), 7.49-7.52 (m, 2H), 7.43-7.48 (m, 3H), 7.33-7.40 (m, 5H), 2.05 (s, 3H), 1.48 (s, 9H). ¹³C NMR (CDCl₃, 100.62 MHz): 8 150.24, 139.92, 138.54, 138.30, 137.31, 137.05, 135.93, 135.83, 133.88, 133.02, 131.65, 131.02, 130.94, 130.13, 129.97, 129.70, 129.52, 128.30, 127.54, 127.38, 127.12, 126.56, 126.05, 125.67, 125.38, 125.22, 125.17, 124.88, 34.75, 31.55, 29.70, 19.67.

Cyclic Voltammetry. CV measurements were carried out with a three-electrode cell configuration consisting of platinum working and counter electrodes and an Ag/AgNO₃ (0.1 M in MeCN) reference electrode at room temperature. The solvent was MeCN and tetra-*n*-butylammonium hexa-fluorophosphate (0.1 M) was used as the supporting electrolyte. The oxidation potentials were recorded at a scan rate of 50 mV/s and reported with reference to the ferrocene/ferrocenium (Fc/Fc⁺) redox couple.

Results and Discussion

Synthesis and Characterization. The synthetic procedures toward anthracene derivatives **6** and **7** are shown in Scheme 1. A new compound, 3-bromo-9,10-dihydro-9-oxoanthracene

(2), was prepared from ring opening reaction of 3-(4-bromophenyl)phthalide in the presence of red phosphorus and iodine, followed by ring cyclization in high yield (83%). 2-Bromo-10-(naphthalene-2-yl)anthracene (3) was produced by the reaction of lithium salts of 2-bromonaphthalene, followed by acidification in good yield. The compound 3 can be readily converted into the corresponding lithium salt in THF, which subsequently reacted with triphenylsilyl chloride to afford 4 in 70% yields. After the compound 5 was synthesized by bromination of 4 using NBS in DMF, the final compound 6 was obtained by the Pd-catalyzed Suzuki coupling reaction of 5 with phenylboronic acid in the presence of a catalytic amount of $Pd(PPh_3)_4$ and excess K_2CO_3 as a yellow solid (63%). The novel compound 7 was also prepared from Pd-catalyzed Suzuki coupling reaction of 2-(10-(4-(tert-butyl)phenyl)anthracen-9-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane with 1-(3-iodo-4-methylphenyl) naphthalene according to a published procedure.²² The formation of all compounds 1-7 was confirmed by ¹H and ¹³C NMR spectroscopy (Figure S1–S13, Supporting information).

Thermal Properties. The thermal properties of 6 and 7 were investigated by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) under N2 atmosphere. Thermal stability was compared on the basis of T_{d5} , which represents 5% weight loss of compounds with increasing temperature. The compound 6 and 7 exhibited T_{d5} values of 350 and 320 °C, respectively, indicating excellent thermal stability. Additionally, the morphological stability of 6 and 7 was monitored by DSC, which was performed from 0 to 450 °C at a heating rate of 10 °C/min. The glass transition temperature $(T_{\rm g})$ of **6** and **7** was obtained at 253 and 221 °C, respectively. Also, the melting temperature of 6 and 7 occurred at 331 and 299 °C, respectively (Figure S14). The results of the second scan were recorded to eliminate differences from the sample history. These results clearly show that the bulky triphenylsilyl group on the C2 position of anthracene moiety of the compound 6 leads to enhanced thermal stability compared with 7. Additionally, high $T_{\rm g}$ of 6 and 7 can enhance operational device lifetime for OLEDs using two asymmetric anthracene compounds.25,26

Photophysical and Electrochemical Properties. The photophysical properties of anthracene-based compounds **6** and **7** were investigated by UV–Vis absorption and photoluminescence (PL) spectroscopy (Figure 1 and Table 1). The absorption spectra of **6** and **7** in dilute CH₂Cl₂ exhibited the characteristic π – π * vibronic transition patterns ($\lambda_{abs} = 364$, 383, and 404 nm for **6** and $\lambda_{abs} = 356$, 375, and 396 nm for **7**) of the isolated anthracene group. The absorption maximum wavelengths (λ_{abs}) of **6** are slightly red-shifted by 8 nm in comparison with those of compound **7**. The emission maximum (λ_{em}) of **6** and **7** was 431 and 434 nm, respectively. The PL spectra of two compounds were similar to previously reported asymmetric anthracene derivatives,^{20,22} assignable to the lowest π – π * transition of anthracene moieties.





Scheme 1. Synthetic routes for asymmetric anthracene derivatives 6 and 7.



Figure 1. UV–Vis absorption and PL spectra of 6 (a) and 7 (b) in CH₂Cl₂ solution.

The electrochemical properties of **6** and **7** were examined by CV measurements. From the first oxidation onset potential, the HOMO energy levels of **6** and **7** were estimated to be -5.56 and -5.51 eV, respectively. The LUMO energy levels of **6** and **7** were also calculated as -2.64 and -2.49 eV,

respectively. The anthracene-based compounds **6** and **7** have wide optical band gaps (E_g) of *ca.* 2.9–3.1 eV.

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Electroluminescent Properties. The anthracene-based compounds 6 and 7 were investigated as blue host materials in blue-emitting OLEDs with BD-142 fluorescent dopant. Because of the similar HOMO energy levels of **6**, **7**, and 4,4'bis[N-(1-naphthyl)-N-phenylamino]biphenyl (NPB) (*ca.* 5.4 eV), NPB was used as the hole-transporting layer (HTL) material.

As shown in Figure 2, the OLEDs were constructed with the following configuration: ITO/HI-406 (65 nm)/NPB (20 nm)/ **6** or **7** (35 nm, BD-142 (7 wt %))/Bebq₂ (20 nm)/LiF (1 nm)/ Al device, where ITO is the anode; HI-406 (purchased from Idemitsu Kosan Co. Tokyo, Japan) is served as the holeinjection layer (HIL); NPB is the HTL; the newly purified anthracene derivatives **6** or **7** (host) and BD-142 (purchased from Idemitsu Kosan Co.) (dopant) are served as the EML; bis(10-hydroxy-benzo[h]-quinolinato)beryllium (Bebq₂) is the electron-transporting layer (ETL); a thin LiF serves as an electron-injection layer at the Al cathode interface. As shown in Figure 3, two devices emitted the expected blue light. Figure 3 shows the EL spectra of **6** or **7** based devices with a 7 wt % ratio of BD-142 at 20 mA/cm². The EL emission maximum of two devices exhibits one peak at 464 (for **6**) and 472 (for **7**) nm, respectively.

The CIE color coordinates appeared at (0.143, 0.167) for **6** and (0.135, 0.175) for **7**. The detailed EL characteristics of the fabricated devices were summarized in Table 2. According to the current density–voltage–luminance (I-V-L) curves (Figure 4), two devices showed good performance as blue host materials. In particular, **7**-based device exhibited a turn-on voltage of 3.0 V, a maximum brightness (L_{max}) of 37 090 cd/m² at 11.0 V, and a maximum external efficiency (η_{ext}) of 5.80% (7.26 cd/A). These results indicate that **6** and **7** properly function as a blue emitting layer.

Table 1. Photophysical and electrochemical properties of 6 and 7.

Compound	$\lambda_{abs} (nm)^a$	$\lambda_{\rm em} \left({\rm nm} \right)^a$	$E_{\rm g} \left({\rm eV} \right)^b$	$E_{\rm ox} \left({\rm V} \right)^c$	HOMO $(eV)^d$	LUMO $(eV)^e$
6	364, 383, 404	431	2.92	0.76	-5.56	-2.64
7	356, 375, 396	434	3.02	0.71	-5.51	-2.49

^{*a*} In CH₂Cl₂.

^b Estimated from the absorption edge.

^c The oxidation onset potential vs. an Fc/Fc⁺ couple.

^{*d*} Calculated from the E_{ox} .

^{*e*} Estimated from the HOMO and band-gap (E_g) energies.





Table 2. EL performances of BD-142 (7 wt %)-doped blue OLEDs.



Figure 3. EL spectra of the devices using 6 and 7 with BD-142 (7 wt %) at 20 mA/cm².

Compound	$V_{\rm on}\left({\rm V}\right)^a$	$V(\mathbf{V})^{b}$	$\eta_{ext} \left(\%\right)^c$	$\eta_{lum} \left(cd/A \right)^d$	$\eta_{pw} (lm/W)^e$	$L_{\max} (cd/m^2)^f$	CIE (x, y)
6	3.00	4.56	4.83	6.15	4.23	19 680	(0.143, 0.167)
7	3.00	5.20	5.80	7.26	4.38	37 090	(0.135, 0.175)

^a Turn-on voltage.

^{*b*} Recorded at 20 mA/cm^2 .

^{*c*} Maximum external quantum efficiency.

^{*d*} Maximum luminous current density.

^e Maximum power efficiency.

^f Maximum luminance at 11 V.



Figure 4. The *I*–*V*–*L* curves of the (a) 6- and (b) 7-based device.

Conclusion

In summary, anthracene-based new blue host materials **6** and **7** were synthesized and characterized. The anthracene derivatives possessed high thermal stabilities that are appropriate as novel host materials for blue OLEDs. All devices showed good performances as blue host materials. The **7**-based device especially displays higher device performances with respect to brightness, luminous current density, and external quantum efficiencies than those of **6**-based device. Relevant further studies using various anthracene derivatives to enhance the performance of devices are in progress.

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Supporting Information. Additional supporting information is available in the online version of this article.

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