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# Efficient Deep-Blue Organic Light-Emitting Diodes: Arylamine-Substituted Oligofluorenes\*\*

By Zhi Qiang Gao, Zhong Hui Li, Ping Fan Xia, Man Shing Wong,\* Kok Wai Cheah, and Chin H. Chen

Novel deep-blue-light-emitting diphenylamino and triphenylamino end-capped oligofluorenes were synthesized by double palladium-catalyzed Suzuki cross-coupling of dibromo-oligofluorene with the corresponding boronic acid as a key step. These oligofluorenes exhibit deep-blue emission ( $\lambda^{\rm em}_{\rm max}$  = 429–432 nm), low and reversible electrochemical oxidation (highest occupied molecular orbital = 5.15–5.20 eV), high fluorescence quantum yield ( $\Phi_{\rm FL}$  = 0.61–0.93), and good thermal properties (glass-transition temperature,  $T_{\rm g}$  = 99–195 °C and decomposition temperature,  $T_{\rm dec}$  > 450 °C). Remarkably, saturated deep-blue organic light-emitting diodes, made from these oligofluorenes as dopant emitters, have been achieved with excellent performance and maximum efficiencies up to 2.9 cd A<sup>-1</sup> at 2 mA cm<sup>-2</sup> (external quantum efficiency of 4.1 %) and with Commission Internationale de l'Éclairage (x,y) coordinates of (0.152,0.08), which is very close to the National Television System Committee standard blue

### 1. Introduction

Among the three principal colors necessary for full-color organic light-emitting diode (OLED) display applications, blue-light-emitting materials and devices still need to be improved in terms of efficiency and color purity in order to achieve white balance, owing to the far superior stabilities and device performances of green and red emitters. In recent years, development of a deep-blue emitter, which has blue emission with a Commission Internationale de l'Éclairage (CIE) coordinate of y < 0.15, has drawn considerable attention<sup>[1]</sup> as such an emitter can not only effectively reduce the power consumption of a full-color OLED panel but also be utilized to generate emission of other colors by energy transfer to a matching emissive dopant. <sup>[2,3]</sup>

[\*] Prof. M. S. Wong, Dr. Z. Q. Gao, Prof. K. W. Cheah, Prof. C. H. Chen Centre for Advanced Luminescence Materials Hong Kong Baptist University Hong Kong, S.A.R. (China) E-mail: mswong@hkbu.edu.hk Prof. M. S. Wong, Dr Z. H. Li, Prof. P. F. Xia Department of Chemistry, Hong Kong Baptist University Hong Kong, S.A.R. (China) Prof. K. W. Cheah Department of Physics, Hong Kong Baptist University Hong Kong, S.A.R. (China) Prof. C. H. Chen. Display Institute, Microelectronics and Information Systems Research Center, National Chiao Tung University Hsinchu, Taiwan 300 (Taiwan)

In fact several materials have been demonstrated for the use of highly efficient deep-blue OLEDs with a CIE coordinate of y < 0.15. [3,4] However, the highly efficient deep-blue OLEDs with a CIE coordinate of y < 0.10, which matches the National Television System Committee (NTSC) standard blue (CIE (x,y) coordinates of (0.14,0.08)), are rare.<sup>[5]</sup> It is well known in the display community that the power consumption of a fullcolor OLED is highly dependent upon the color of blue emission. [6] The deeper the blue color (smaller CIE y-value), the lower the power consumption (in lm W<sup>-1</sup>) of the device. The main obstacle for the realization of high-efficiency deep-blue OLEDs is the stringent requirements of the device application, which include a high fluorescence quantum yield ( $\Phi_{FL}$ ), a wide-energy bandgap, high thermal stability and good thin-film morphology, and only few materials can meet these requirements so far. Recently, fluorene-based molecules, oligomers, and polymers, which show great promise as highly stable and efficient deep-blue or UV emissive materials, have been extensively investigated. [5,7-9] Triarylamines or their moieties have been employed to incorporate with fluorene-based polymers to improve the hole injection and transport properties with which the OLEDs show enhanced device efficiency and stability.<sup>[10]</sup> In previous studies, we have demonstrated that oligofluorenes end-capped with diphenylamino substituents exhibit desirable functional properties that are useful as deep-blue emitters for OLED devices.<sup>[11]</sup> For example, the diphenylamino end-capped bifluorene, OF(2)-NPh utilized as a nondoped emission layer can exhibit a deep-blue color with CIE coordinates of (0.15,0.09), very close to the standard NTSC blue; however, the maximum efficiency of this nondoped OLED device was only about 0.8 cd A<sup>-1</sup>. In addition, the low glass-transition temperature of **OF(2)-NPh** ( $T_o = 91 \,^{\circ}$ C) could limit its practical use as a nondoped emission layer. An effective way to significantly enhance OLED device performance is to adopt the guest-host emission system as reported previously.<sup>[12]</sup> Herein, we report

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the synthesis and investigation of novel deep-blue-emitting diphenylamino and triphenylamino end-capped oligofluorenes. We demonstrate for the first time that highly efficient deepblue OLEDs, using these oligofluorenes as a fluorescent dopant emitter and 4,4'-N,N'-di(carbazolyl)-biphenyl (CBP) as a host, have been developed.

#### 2. Results and Discussion

The molecular structures of the deep-blue emitting oligofluorenes are shown in Scheme 1. Syntheses of OF(3)-NPh was reported previously.<sup>[11a]</sup> The synthetic routes for SOF(3)-NPh and OF(2)-PhNPh are outlined in Scheme 2. Amination of

Scheme 1. Molecular structures of the deep-blue-light-emitting materials in this work.

diphenyl-2,7-dibromofluorene<sup>[13]</sup> (1) with 1 equiv diphenylamine in the presence of Pd(OAc)<sub>2</sub>:2P(o-tolyl)<sub>3</sub>, as catalyst, afforded the mono-amination product 2 in 45 % yield and diamination product in 25 % yield. Transformation of the bromide 2 into the corresponding boronic acid 3 was achieved in 86 % yield by lithium-bromide exchange at -78 °C, followed by reaction with trimethyl borate at room temperature and subsequent acid hydrolysis. Double palladium-catalyzed Suzuki cross-coupling of 1 with boronic acid (3) afforded terfluorene **SOF(3)-NPh** in 65 % yield and the mono-coupling product in 21 % yield. Adopting a similar convergent approach, triphenylamino-substituted bifluorene, OF(2)-PhNPh, was synthesized by double palladium-catalyzed Suzuki cross-coupling of 4-diphenylaminophenylboronic acid<sup>[14]</sup> (4) and dibromobifluorene<sup>[11]</sup> (5), which afforded the desirable product in 90% isolated yield. All the newly synthesized oligofluorenes were fully characterized with <sup>1</sup>H NMR, <sup>13</sup>C NMR, high-resolution mass spectrometry (HRMS), and elemental analysis and found to be in good agreement with their structures.

The photophysical, electrochemical, and thermal properties of these oligofluorenes are summarized in Table 1. The absorption spectra of these diphenylamino and triphenylamino endcapped oligofluorenes measured in chloroform are composed of two major absorption bands. The absorption peak appearing around 306 nm is due to the  $n\rightarrow\pi^*$  transition of triarylamine moieties and the absorption maxima are in the range of 375-390 nm, corresponding to the  $\pi \rightarrow \pi^*$  transition of the oligofluorene core. They all exhibit deep-blue emission with an emission maximum around 432 nm, as shown in Figure 1. Their emission spectra are excitation-wavelength-independent, that is, whether excitation occurs at either triarylamine moieties or the oligofluorene core, the emission spectra obtained are iden-

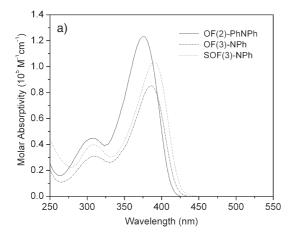
Scheme 2. Synthesis routes for SOF(3)-NPh and OF(2)-PhNPh.



Table 1. Summaries of photophysical, electrochemical, and thermal measurements.

	λ <sup>abs</sup> <sub>max</sub> [a] [nm]	λ <sup>em</sup> <sub>max</sub> [a,b] [nm]	$\Phi_{\sf FL}\left[{\sf a,c}\right]$	τ [a,d] [ns]	E <sub>1/2</sub> [e] [V]	HOMO [f] [eV]	Τ <sub>g</sub> [g] [°C]	<i>T</i> <sub>dec</sub> [h] [°C]
OF(3)-NPh	386	432	0.93	1.23	0.35, 0.87	-5.15	99	461
SOF(3)-NPh	390	435	0.61	1.07	0.40, 0.96	-5.20	195	585
OF(2)-PhNPh	375	429	0.90	1.37	0.40	-5.20	107	456

[a] Maximum absorption wavelength, measured in CHCl<sub>3</sub>. [b] Maximum emission wavelength, excited at the absorption maxima. [c] Using 9,10-diphenylanthrancene ( $\Phi_{360} = 0.9$ ) as a standard. [d]  $\tau$  is the fluorescence lifetime, measured using nitrogen laser as an excitation source. [e]  $E_{1/2}$  (oxidation potential) vs. Fc+/Fc estimated by cyclic voltammetry: a platinum disc electrode as a working electrode, platinum wire as a counter electrode, and a standard calomel electrode (SCE) as a reference electrode with an agar salt bridge connected to the oligomer solution; ferrocene was used as an external standard,  $E_{1/2}$  (Fc/Fc<sup>+</sup>) = 0.45 V (vs. SCE). [f] Highest occupied molecular orbital (HOMO) level, calculated with reference to ferrocene (4.8 eV). [g] Glass-transition temperature, T<sub>e</sub>, determined by differential scanning calorimetry with a heating rate of 10 °C min<sup>-1</sup> under N<sub>2</sub>. [h] Decomposition temperature, T<sub>dec</sub>, determined by thermal gravimetric analysis with a heating rate of 10 °C min<sup>-1</sup> under N<sub>2</sub>.



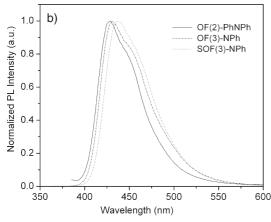


Figure 1. a) Absorption and b) photoluminescence (PL) emission spectra of diphenylamino- and triphenylamino-end-capped oligofluorenes measured in chloroform.

tical. This suggests that the absorbed energy or excitons can efficiently transfer from the triarylamine moieties to the emissive oligofluorene core. It is interesting to note that 9,9-diphenyl-substituted terfluorene, SOF(3)-NPh, consistently exhibits a slight red-shift (3-4 nm) in its absorption and emission spectra, as compared to those of its 9,9-dibutyl-substituted counterparts, OF(3)-NPh (Fig. 1). Importantly, both OF(3)-NPh and OF(2)-PhNPh exhibit very high fluorescence

quantum yields (>90% relative to diphenylanthracene) with the exception of the 9,9-diphenylfluorene-based molecule, **SOF(3)-NPh**. As the measured fluorescence lifetimes  $(\tau)$  are on the nanosecond timescale (1.07-1.37 ns), the origin of the blue emission is consistent with being from the singlet emissive excited state.

Cyclic voltammetry (CV) was carried out in a three-electrode cell set-up with 0.1 m Bu<sub>4</sub>NPF<sub>6</sub> as a supporting electrolyte in CH<sub>2</sub>Cl<sub>2</sub> to examine the electrochemical properties of the oligomers. The results are tabulated in Table 1. All the oligofluorenes exhibit a reversible two-electron oxidation wave with  $E_{1/2}$  in the range of 0.35–0.40 V, corresponding to two triarylamine oxidations. In addition, for terfluorenes, there is an additional (quasi)reversible one-electron oxidation wave, corresponding to the oxidation of the terfluorene  $\pi$ -conjugated core, with  $E_{1/2}$  in the range of 0.87–0.96 V. Because of endcapping with the diphenylamino or triphenylamino groups, these oligomers exhibit very low first ionization potential values, with the highest occupied molecular orbital (HOMO) level around 5.2 eV, as estimated from the electrochemical method. Such a high HOMO level greatly reduces the energy barrier for the hole injection from an indium tin oxide (ITO) cathode to the oligofluorenes and enhances hole transport of the oligofluorenes. Furthermore, these oligomers show distinct higher glasstransition temperatures from the remelt in the range of 99–195 °C, as determined by the differential scanning calorimetry (DSC), and high thermal stabilities with decomposition temperatures  $T_{\rm dec} > 450 \,^{\circ}\text{C}$ , as determined by thermal gravimetric analysis (TGA) (Fig. 2).

To probe the potential application of these blue-emitting oligomers, doped OLED devices with the structure ITO/NPB (45 nm)/TCTA (15 nm)/EML (40 nm)/BPhen (20 nm)/LiF/Al were fabricated (where NPB denotes N, N'-bis-(1-naphthyl)-N,N'-dipheny-1,1'-biphenyl-4,4'-diamine, TCTA denotes 4,4',4"-tris(N-carbazoly)-triphenylamine, and BPhen denotes bathophenanthroline), in which OF(3)-NPh, SOF(3)-NPh, and OF(2)-PhNPh are used as dopant emitter layers (EMLs) doped into the CBP host.

As shown in Figure 3, all the electroluminescence (EL) spectra, which are voltage-independent, show an emission peak around 428-432 nm, which corresponds well with the same solution's photoluminescence (PL); that is, the emission origi-



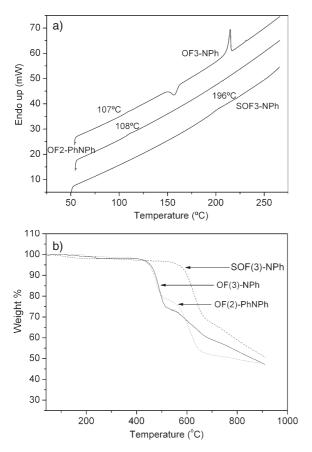


Figure 2. a) DSC traces recorded from the second heating cycle and b) TGA traces of oligofluorenes measured at 10 °C min<sup>-1</sup>.

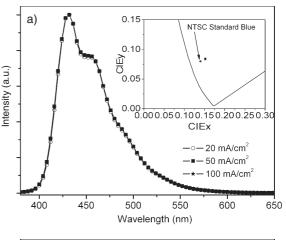
nates from singlet-excited states of the oligomers. More importantly, all these EL specta exhibit saturated blue emission with  $CIE_{x,y}$  1931 coordinates of (0.152,0.083), (0.151,0.099), and (0.153,0.072) for **OF(3)-NPh-**, **SOF(3)-NPh-**, and **OF(2)-PhNPh**-based devices, respectively, all of which are very close to the NTSC standard blue color. The performances of these OLED devices at the drive current density of 20 mA cm<sup>-2</sup> are listed in Table 2.

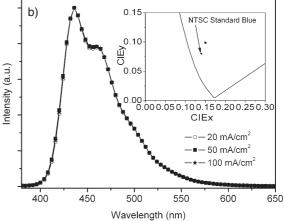
All of these dopant emitters show promising EL efficiencies in the current density range of 1 to 100 mA cm<sup>-2</sup> (Fig. 4). The maximum efficiency achieved is 2.9 cd A<sup>-1</sup> at 2 mA cm<sup>-2</sup> with

Table 2. Performance of OLED devices doped with oligofluorenes (@ 20 mA cm<sup>-2</sup>) [a]. LE = luminance efficiency, CE=current efficiency, EQE=external quantum efficiency.

EML (CBP host)	V [V]	CE [cd/A]	LE [lm/W]	EQE [%]	CIE (x,y)
OF(3)-NPh	6.9	2.24	1.01	3.1	(0.152,0.083)
SOF(3)-NPh	7.0	2.7	1.24	3.2	(0.151,0.099)
OF(2)-PhNPh	6.8	1.75	0.8	2.9	(0.153,0.072)

[a] Device structure: ITO/NPB (45nm)/TCTA (15nm)/EML (40nm)/BPhen (20nm)/LiF/Al. (NPB = N, N'-Bis-(1-naphthyl)-N, N'-diphenyl-1, 1'-biphenyl-1, 1'-biphenyl-1,4,4'-diamine; TCTA = 4,4',4"-tris (N-carbazoly)-triphenylamine; BPhen = bathophenanthroline; EML=4 % of dopant emitter in 4,4'-N,N'-dicarbazole-biphenyl (CBP) host.)





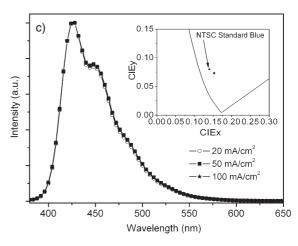


Figure 3. EL spectra of a) OF(3)-NPh, b) SOF(3)-NPh, and c) OF(2)-PhNPh doped into CBP at current densities of 20, 50, and 100 mAcm<sup>-</sup> Inset: CIE 1931 coordinates of the devices, along with the NTSC blue standard.

an external quantum efficiency (EQE) of 4.1% when OF(3)-NPh was used as a dopant emitter, which is attributed to the high fluorescence quantum yield of this oligofluorene. The maximum luminance is more than 5000 cd m<sup>-2</sup>. It is worth mentioning that this is the best current efficiency reported so far for deep-blue OLEDs, with a CIE coordinate of y < 0.10, which matches closely to the NTSC blue standard. [15] Although

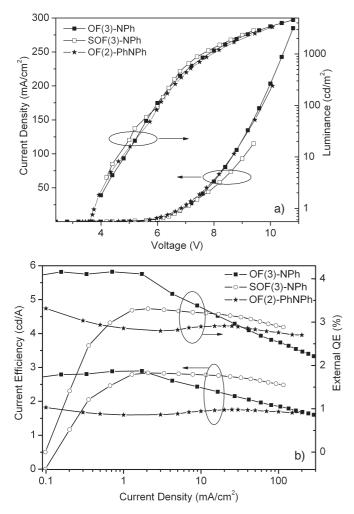


Figure 4. a) Current density-voltage-luminance plot and b) efficiency as a function of current density of oligofluorene-based OLED devices.

there is slight roll-off in EL efficiency at high current density for the OF(3)-NPh-based device, the overall performances of these oligofluorene-based devices are still among the best that have been reported in the literature for the saturated deepblue OLEDs with a CIE coordinate of y < 0.10.

## 3. Conclusion

In summary, blue light-emitting diphenylamino and triphenylamino end-capped oligofluorenes have been synthesized by a convergent approach using palladium catalyzed Suzuki cross-coupling. All the oligofluorenes exhibit a low first ionization potential, high fluorescence quantum yield, as well as good thermal and amorphous morphological stabilities. Saturated deep-blue OLED devices with excellent performance based on these oligofluorenes as dopant emitters have been demonstrated, with maximum efficiencies up to 2.9 cd A<sup>-1</sup> at 2 mA cm<sup>-2</sup> (EQE of 4.1%) and with CIE coordinates of (0.152,0.08), which is very close to the NTSC standard blue.

## 4. Experimental

2-Bromo-7-diphenylamino-9,9-diphenylfluorene (2): A single-necked 100 mL round-bottom flask was charged with diphenylamine (3.38 g, 20 mmol), sodium tert-butoxide (2.88 g, 30 mmol), palladium(II) acetate (0.11 g, 0.50 mmol), dry toluene (30 mL), 2,7-dibromo-9,9-diphenylfluorene, 1 (9.52 g, 20 mmol), and tri-(o-tolyl)phosphine (0.30 g, 1 mmol), which was heated at 110 °C for 24 h under a nitrogen atmosphere. After cooling to room temperature, the reaction mixture was quenched with water and extracted with dichloromethane  $(3 \times 50 \text{ mL})$ . The combined organic solution was washed with water and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. Evaporation of volatiles left a dark solid, which was separated by silica gel column chromatography, by using petroleum ether/dichloromethane (v/v = 8:1) as eluent, affording compound 2 in 45 % yield as a light-yellow solid and symmetrically disubstituted compound diphenylfluorene in 52 % yield as a yellow solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, δ): 7.47–7.60 (m, 4H), 7.14–7.30 (m, 16H), 7.00–7.10 ppm (m, 6H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>, δ): 152.6, 152.2, 147.8, 147.3, 145.0, 139.0, 133.1, 130.6, 129.3, 129.0, 128.2, 128.0, 126.7, 126.4, 124.2, 124.1, 122.9, 121.1, 120.7, 120.2, 65.6 ppm. MS (fast atom bombardment, FAB): m/z 564.6 (M<sup>+</sup>+1).

7-N,N-Diphenylamino-9,9-diphenylfluorenyl-2-boronic acid (3): To a 100 mL two-necked flask (equipped with a magnetic stirrer) that contained a solution of 2 (1.13 g, 2.0 mmol) in dried tetrahydrofuran (30 mL) under a N<sub>2</sub> purge and in a -78 °C acetone-dry-ice bath, were added 1.5 M of n-butyl lithium (4.0 mL, 6.0 mmol) while maintaining stirring. After stirring for 0.5 h, the temperature was gradually raised to room temperature for another 0.5 h, and then the reaction mixture was cooled to -78 °C again, and trimethyl borate (0.70 mL, 6.0 mmol) was then added. After stirring for 2 h, water was first added to the reaction mixture, and then HCl (6 M) was added in a dropwise fashion until an acidic mixture was obtained. The reaction mixture was poured into water and extracted with dichloromethane ( $3 \times 50$  mL). The combined organic layer was dried with anhydrous Na2SO4 and evaporated to dryness. The crude product was purified by flash column chromatography using dichloromethane:ethyl acetate as a gradient eluent. The product boronic acid 3 was obtained as a yellow solid with an isolated yield of 86 %. <sup>1</sup>H NMR (270 MHz, DMSO-d<sub>6</sub>,  $\delta$ ): 8.05 (s, 2H), 7.78–7.89 (m, 4H), 7.19–7.22 (m, 10H), 6.69–7.05 ppm (m, 12H). <sup>13</sup>C NMR (66 MHz, DMSO-d<sub>6</sub>, δ): 152.3, 149.3, 147.1, 146.8, 145.3, 141.1, 133.9, 133.7, 131.4,  $129.3,\ 128.2,\ 127.6,\ 126.5,\ 123.8,\ 123.1,\ 122.4,\ 121.6,\ 120.6,\ 118.7,$ 64.7 ppm.

2,7''-Bis(diphenylamino)-7,2',7',2''-ter(9,9-diphenylfluorene) (**SOF(3)**-NPh): A mixture of 2,7-dibromo-9,9-diphenylfluorene 1 (381 mg, 0.8 mmol), 7-(diphenylamino)-9,9-diphenylfluorenyl-2-boronic acid 3 (1035 mg, 2.0 mmol), 1:2 palladium(II) acetate/triphenylphosphine (5 mol%), toluene (30 mL), methanol (15 mL), and 2 m K<sub>2</sub>CO<sub>3</sub> (4 mL) was heated at 75 °C and left overnight. After the reaction mixture was cooled to room temperature, it was poured into water and extracted with dichloromethane (3×50 mL). The combined organic layer was dried with anhydrous Na<sub>2</sub>SO<sub>4</sub> and evaporated to dryness. The crude product was further purified by silica gel column chromatography using 3:1 petroleum ether/dichloromethane as eluent, affording SOF(3)-NPh as a yellow solid with an isolated yield of 65 % and a mono-coupled product as a light yellow solid with an isolated yield of 21 %. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>,  $\delta$ ): 7.74 (d, J = 8.00 Hz, 2H), 7.64 (d, J = 8.00 Hz, 2H), 7.49 - 7.57 (m, 10H), 7.16 - 7.21 (m, 40H), 7.03(d, J = 7.60 Hz, 8H), 6.95–6.99 ppm (m, 6H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>, δ): 152.8, 152.0, 151.3, 147.6, 145.9, 145.8, 140.8, 139.8, 139.3, 138.9, 134.1, 129.1, 128.3, 128.2, 126.7, 126.6, 124.7, 124.2, 123.0, 122.8, 121.5, 120.7, 120.3, 119.7, 65.7, 65.5 ppm. MS (FAB): m/z 1285.8 (M<sup>+</sup>). Anal. Calcd for C<sub>99</sub>H<sub>68</sub>N<sub>2</sub>: C, 92.49; H, 5.33; N, 2.18. Found: C, 92.19; H, 5.40; N, 2.34.

9,9-Dibutyl-2,2'-bis(4-diphenylamino-1-phenyl)bifluorene **PhNPh**): To a mixture of 4-(N,N-diphenylamino)-1-phenylboronic acid [13] **4** (347 mg, 1.20 mmol), 2,2'-diiodo-bis[9,9-bis(*n*-butyl)fluorene] [14] 5 (403 mg, 0.50 mmol), Pd(OAc)<sub>2</sub> (11 mg, 5 mol %), and tri(o-tolyl)phosphine (30 mg, 10 mol %) in a 100 mL round-bottom flask were added toluene (20 mL), methanol (10 mL), and 2 m aqueous solution of K<sub>2</sub>CO<sub>3</sub> (2 mL). The reaction mixture was stirred under a nitrogen



atmosphere at 75 °C overnight. After cooling to room temperature, the reaction mixture was poured into cool water and extracted with dichloromethane (3 × 50 mL). The combined organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and evaporated to dryness. The crude product was purified by silica gel column chromatography using petroleum ether/dichloromethane (v/v = 6:1) as eluent affording the desired product as a light-yellow solid (478 mg, 92 % yield). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>,  $\delta$ ): 7.69 (d, J = 7.6 Hz, 2H), 7.63 (d, J = 8.0 Hz, 2H), 7.59–7.61 (m, 8H), 7.27 (d, J = 8.0 Hz, 8H), 7.15 (d, J = 7.6 Hz, 10H), 7.00–7.06 (m, 10H), 1.88–1.99 (m, 8H), 1.07–1.15 (m, 8H), 0.73 ppm (t, J = 7.4 Hz, 20H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>,  $\delta$ ): 119.3, 152.4, 151.4, 148.0, 147.1, 140.0, 139.6, 136.0, 129.1, 125.9, 123.8, 123.7, 123.6, 123.5, 122.5, 122.3, 121.0, 120.3, 119.4, 55.1, 40.0, 26.1, 23.0, 13.9 ppm. MS (FAB): m/z 1040.6 (M<sup>+</sup>). Anal. Calcd for  $C_{78}H_{76}N_2$ : C, 89.95; H, 7.36; N, 2.69. Found: C, 90.00; H, 7.38; N, 2.68.

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