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### **Dipyrenylcarbazole Derivatives for Blue Organic Light-Emitting Diodes**

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The use of  $\pi$ -conjugated organic compounds as electroluminescent materials in diode devices was pioneered by van Slyke and co-workers over two decades ago.<sup>[1]</sup> Since then, the development of new polymeric or small molecular  $\pi$ conjugated compounds with superior physical, optical, thermal, and electrochemical properties has become one of the most thriving research areas.<sup>[2-4]</sup> This research is driven inpart by the recent growing demands for organic light-emitting diodes (OLEDs)<sup>[5]</sup> in high-performance electroluminescent materials that have applications in full-color, flat panel, and flexible displays. The simplest configuration of OLEDs, which are composed of a layer of emissive material sandwiched between a cathode and an anode, usually fails to provide satisfactory efficiency and stability for long-term use.<sup>[6]</sup> To circumvent these problems, double- or multi-layer devices have been constructed by incorporating chargetransporting layers, that is, hole-transport and electrontransport layers, into the device. In order to simplify the manufacturing process of material coating, organic compounds that have both light-emitting and charge-transporting properties are therefore highly desirable. A number of polymers or small molecules containing carbazoles are commercially available and have been used as amorphous holetransporting materials, such as poly(vinylcarbazole) (PVK), tris(4-carbazoyl-9-ylphenyl)amine (TCTA), and 4,4'-di(9Hcarbazol-9-yl)biphenyl (CBP). These compounds are known

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for their high glass transition temperatures and good thermal stabilities. Several other examples, such as carbazole derivatives containing diarylamine peripheries<sup>[7]</sup> and triarylamine–carbazole end-capped oligofluorenes<sup>[8]</sup> have also been reported. Recent development in emissive materials has focused on the blue electroluminescence (EL)<sup>[9]</sup> as a number of new fluorescent blue light-emitting materials, such as triphenylfluoranthene,<sup>[10]</sup> oligoquinoline,<sup>[11]</sup> triarylamine,<sup>[12]</sup> fluorene,<sup>[13]</sup> and pyrene derivatives<sup>[14]</sup> have been reported. However, pyrene derivatives are more attractive to us owing to their strong fluorescence, high quantum efficiency, carrier mobility, and hole-transporting ability.<sup>[14a,15]</sup>

Our design for new electroluminescent materials involved a combination of a hole-transporting carbazole unit and a highly fluorescent pyrene moiety to incorporate desirable physical, optical, and electronic properties. Herein, we report a detailed synthesis of compounds 1-3 and their optical, thermal, and electrochemical properties (Scheme 1). A preliminary investigation of OLED device fabrication is also reported. We began with a regioselective diiodination of carbazole using KI/KIO<sub>3</sub> in refluxing acetic acid, followed by a Suzuki coupling with the commercially available pyrene-1boronic acid to afford dipyrenylcarbazole 5 in 67% yield (Scheme 2). The nitrogen atom was then substituted with a para-tolyl group by reacting with 4-iodotoluene under the copper-catalyzed C-N coupling conditions developed by Buchwald and co-workers.<sup>[16]</sup> After a purification by column chromatography on silica gel, compound 1 was obtained in a fair yield of 56%. However, we found that 1 had low solubility in most organic solvents, which is considered to be an undesirable property of materials for solvent-casting techniques. Therefore, we decided to substitute the nitrogen atom in 5 with a flexible glycolic chain. Along this line, it would be interesting to see the effect of a combination of multiple units of 5 into a single molecule. Upon treatment of 5 with K<sub>2</sub>CO<sub>3</sub> and 1,2-bis(chloroacetoxy)ethane,<sup>[17]</sup> we obtained diester 2 in 42% yield. This compound is highly soluble in most organic solvents, such as ethyl acetate, dichloromethane, and tetrahydrofuran. We also opted to incorporate a triphenylamine group into the molecule as it is well-



Scheme 1. Structures of dipyrenylcarbazole derivatives (1-3).



Scheme 2. Synthesis of dipyrenylcarbazole derivatives (1–3). DMF = N,N-dimethylformamide, DMSO = dimethyl sulfoxide.

known for its hole-transporting properties. 4-Bromo-*N*,*N*-diphenylaniline, pre-prepared from reacting triphenylamine with *N*-bromosuccinimide,<sup>[18]</sup> successfully underwent a

copper-catalyzed C–N coupling with 5 to give 3 in 70% yield.

The photophysical properties of 1, 2, and 3 (0.1  $\mu$ M in dichloromethane) were investigated and the results were summarized in Table 1. The absorption spectra of all three compounds appeared to be very similar, showing bands at 250, 280, and 350 nm. Although these absorption bands were in the same region as the free carbazole and pyrene, their relatively broader shapes indicated a strong electronic coupling between these two  $\pi$ -conjugated units. This electronic communication was also evident from the fact that the emission wavelengths of 1-3 were significantly longer than that of pyrene.<sup>[14a,19]</sup> Compound 2, which contained two dipyrenylcarbazole units connected with a flexible glycolic chain, showed an emission shoulder at 470 nm that corresponded to the pyrene excimer.<sup>[20]</sup> For compound 3, which contained a triphenylamine group, the maximum emission peak appeared at a longer wavelength (465 nm), while its absorption band appeared in the same location as 1 and 2. These observations suggest that the incorporation of a triphenylamine moiety promotes the electron-vibration coupling in the electronic excited state of 3, thereby resulting in a geometrically relaxed exciton with lower energy.

The solid-phase absorption spectra of the thin films of all three compounds (obtained by spin-casting techniques) exhibited significant bathochromic shifts, thus indicating better  $\pi$ -electron delocalization in the conjugated systems and greater planarity between the carbazole and pyrene units caused by solid-state packing.<sup>[21]</sup> Subsequently, the emission spectra of 1 and 2 also showed substantial bathochromic shifts. As both the absorption and emission spectra of the three compounds in the solid state were very similar in shape and wavelength, it may be assumed that the dipyrenylcarbazole is the photoactive module in these compounds, regardless of the substituent on the nitrogen atom. Interestingly, the solid-state emission spectrum of 3 exhibited a hypsochromic shift compared to the solution-phase spectrum. This result may also be attributed to the aforementioned solid-state packing force which precludes the electron-vibration coupling between the triphenylamine substituent and the photoactive unit.

Next, the thermal properties of 1-3 were examined by differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) under a nitrogen atmosphere (for thermograms, see the Supporting Information). Those results suggested that all three compounds were thermally stable, with 5% weight-loss temperatures  $(T_d^{5\%})$  well above 390°C. During the first heating cycle in the DSC experiments, there were sharp endothermic peaks for 1 and 2, representing their melting temperatures  $(T_m)$  at 256 and 220 °C, respectively. In the second heating cycle, compound 1 showed an endothermic baseline shift owing to glass transition  $(T_g)$  at 165 °C. Further heating of this sample resulted in exothermic peaks at 294 and 357 °C, corresponding to the crystallization temperature  $(T_c)$  and  $T_m$ , respectively. For compound 2, only a  $T_{\rm g}$  at 199°C was observed and there were no signals for crystallization or melting upon further heating. For com-

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Table 1. Photophysical, thermal, and electronic properties of 1-3.

Cmpd	Absorption		Emission <sup>[c]</sup>		$\Phi_{\text{F}}^{[d]}$	$T_g \left[ {}^{\bullet}\!C \right]^{[e]}$	$T_{s\%}\;[^{\bullet}C]^{[f]}$	$E_{1/2}/V$ $(E_{onset}/V)^{[g]}$	$E_g[eV]^{[h]}$	HOMO [eV] <sup>[i]</sup>	LUMO [eV] <sup>[j]</sup>
	$\lambda_{\max} \text{ [nm] } (\log \varepsilon)$ Solution <sup>[a]</sup>	$\lambda_{\max} [nm]$ Thin film <sup>[b]</sup>	$\lambda_{max}$ Solution	[nm] Thin film							
1	347 (4.87)	359	427	456	0.79	165	459	1.09 (1.06)	3.06	-5.50	-2.44
2	347 (5.15)	360	425	460	0.79	199	414	1.03 (1.00)	3.06	-5.44	-2.38
3	345 (4.94)	356	465	453	0.73	166	398	0.91 (0.85)	2.73	-5.29	-2.56

[a] Measured in a dilute  $CH_2Cl_2$  solution. [b] Measured on a spin-cast film. [c] Excited at 345 nm. [d] Quinine sulfate as a standard in  $CH_2Cl_2$ . [e] Obtained from DSC measurements on the second heating cycle with a heating rate of 10 °C min<sup>-1</sup> under N<sub>2</sub>. [f] Obtained from TGA measurements with a heating rate of 10 °C min<sup>-1</sup> under N<sub>2</sub>. [g] Measured using a platinum disk as a working electrode, a platinum rod as a counter electrode, and a SCE as a reference electrode in  $CH_2Cl_2$  containing  $0.1 \text{ m } n\text{Bu}_4\text{NPF}_6$  as a supporting electrolyte at a scan rate of 50 mV s<sup>-1</sup> under an argon atmosphere. [h] Estimated from the electronic absorption edge ( $E_g = 1240/\lambda_{edge}$  eV). [i] Calculated by the empirical equation:  $HOMO = -(4.44 + E_{onset})$ , where  $E_{onset}$  is the onset potential of the oxidation peak.[j] Calculated from LUMO =  $HOMO - E_g$ .

pound 3, the endothermic peak was not observed in the first and second heating cycles, but a  $T_g$  of 166 °C was observed. These results suggested that the amorphous compounds 2 and 3 have the ability to form molecular glass with a high  $T_g$ .

 $T_{\rm g}$ . The electrochemical properties of the compounds were niques and their redox potentials are shown in Table 1. The HOMO energy levels of 1-3 were calculated from the onset potential of the oxidation  $(E_{onset})$ . The energy band gaps  $(E_g)$  were estimated from the electronic absorption band edges which were used for determination of the LUMO energy levels. The results suggested that the HOMO-LUMO energy gaps for 1 and 2 are of a similar size (3.06 eV), although the HOMO of **1** is slightly lower (0.06 eV). Of the three compounds, **3** possessed the highest HOMO, lowest LUMO, and narrowest energy gap of 2.73 eV. In addition, the electrochemical stability of these compounds was tested by seven subsequent redox cycles (voltammograms are available in the Supporting Information). Compounds 1 and 3, which have aromatic substituents on their carbazole nitrogens, were electrochemically unstable. For compound 1, the oxidation may first take place at the nitrogen atom. Delocalization of the unpaired electron and a loss of hydrogen radical would lead to a benzylic radical-cation that could undergo a dimerization. In the case of 3, oxidative coupling at the *para* position of the phenyl rings in the triphenylamine moiety probably led to electro-polymerization. The observation of these oxidative couplings was in good agreement with results previously reported for the other compounds containing the triphenylamine fragment.<sup>[22]</sup> Multiple cyclic voltammetry scans revealed that 2 had good electrochemical stability.

The high photoluminescence (PL) quantum yields of 1-3 encouraged us to investigate the use of these compounds as emissive material (EM) for OLEDs. We used indium tin oxide (ITO; 4.80 eV) as the anode and LiF (3.5 eV) coated with aluminium as the cathode. Single-layer devices of structure ITO/EM(10 nm)/LiF(0.5 nm)/Al(120 nm) were made using 1-3 as the EM (Scheme 3). The voltage/luminance and voltage/current-density characteristics of the devices are shown in Table 2 and Figure 2. Device III was found to have the best external efficiency of 0.15 with a maximum lumi-



Scheme 3. Band diagrams for devices I-IV.

Table 2. EL performance of devices with **1–3** as EM without HTM (devices I–III) and a device with **3** as EM and PEDOT:PSS as HTM (device IV).

Device	EM	$V_{\rm on}^{\ [a]}$	$L_{\max}^{[b]}$	$\eta_{lum}^{[c]}$	$\eta_{ex}{}^{[d]}$	$J^{[e]}$	$CIE^{[f]}$
Ι	1	5.4	670 (12.0)	0.41	0.07	440	(0.18,0.25)
II	2	6.9	1200 (11.6)	0.48	0.07	770	(0.21, 0.33)
III	3	4.8	1450 (9.8)	0.50	0.15	820	(0.18,0.17)
IV	3	3.8	1600 (8.8)	1.50	0.40	660	(0.17,0.15)

[a] Turn-on voltage (V). [b] Maximum luminance  $(cdm^{-2})$  at the applied potential (V). [c] Luminance efficiency  $(cdA^{-1})$ . [d] External efficiency (%). [e] Current density  $(mAm^{-2})$ . [f] Commission International d'Eclairage coordinates (x, y).

Table 3. EL performance of devices with 1-3 as HTM.

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Device	HTM	$V_{\mathrm{on}}{}^{\mathrm{[a]}}$	$L_{max}^{[b]}$	$\eta_{lum}^{[c]}$	$\eta_{ex}^{[d]}$	$J^{[e]}$
V	1	4.7	1600 (8.8)	0.65	0.03	1950
VI	2	4.8	6900 (9.6)	2.50	0.10	1342
VII	3	4.2	9300 (8.8)	2.00	0.10	1555

[a] Turn-on voltage (V). [b] Maximum luminance  $(cdm^{-2})$  at the applied potential (V). [c] Luminance efficiency  $(cdA^{-1})$ . [d] External efficiency (%). [e] Current density  $(mAm^{-2})$ .

nance of  $1,450 \text{ cdm}^{-2}$  and a turn-on voltage at 4.8 V (Table 2, entry 3). As the most-promising result was obtained from device III, **3** was used in the fabrication of a double-layer device (device IV), which incorporated conductive polymers PEDOT:PSS as hole-transporting materials (HTM). We found that the incorporation of PEDOT:PSS not only increased the maximum luminance to 1600 cd m<sup>-2</sup> but also decreased the turn-on voltage from 4.8 to 3.8 V, which is considered to be one of the lowest turn-on voltages for a blue OLED (Table 2, entry 4). The device also emitted the desirable blue light at CIE coordinate of (0.17, 0.15) along with a significantly improved luminance efficiency and a better external efficiency.

Interestingly, we also observed spectral shifts in the solid phase PL and the device EL for the three emissive compounds (compare Figure 3 with Figure 1, bottom). As device IV emitted light at similar wavelengths to device III, these



Figure 1. Absorption and emission spectra of 1-3 in CH<sub>2</sub>Cl<sub>2</sub> solution (top) and thin film (bottom).

shifts between the solid phase PL and the device EL were irrelevant to the presence of the hole-transporting material, PEDOT:PSS. Notably, the maximum emission wavelenghts for 1 and 2 shifted bathochromically, whilst that of 3 shifted hypsochromically. Therefore, we reasoned that the exciton formation in device III may occur closer to the cathode, where the injected electron has less chance to relax, owing to the greater hole-transporting ability of 3. It is important to emphasize that this blue shift in the EL device is a very welcome property for a blue-light-emitting material.

As the HOMO levels of 1-3 lie between the work function of ITO (4.80 eV) and HOMO of Alq<sub>3</sub> (5.70 eV), these compounds may potentially serve as HTM. To test this hy-



Figure 2. The *I-V-L* curve of devices with **1–3** as EM without HTM (ITO/ EM/LiF/Al) (devices I–III) and with HTM (ITO/PEDOT:PSS/**3**/LiF/Al; devices IV).



Figure 3. EL spectra of devices I–III (insets are cropped photographs of the working devices).

pothesis, double-layer devices of configuration ITO/HTM-(10 nm)/Alq<sub>3</sub>(10 nm)/LiF(0.5 nm)/Al(120 nm) were fabricated using **1–3** as the HTM and Alq<sub>3</sub> as the EM (devices V– VII; Scheme 4). The voltage/luminance and voltage/currentdensity characteristics of these green-light-emitting devices are shown in Table 3 and Figure 4. These results clearly demonstrate the hole-transporting ability of **1–3**. Device VII exhibited the best performance with a moderate maximum luminance of 9,300 cd/m<sup>2</sup> for green OLEDs at 8.8 V and a low turn-on voltage of 4.2 V. Therefore, **3** has the best hole transporting ability among these three materials.

In conclusion, we successfully synthesized three new derivatives of 3,6-dipyrenylcarbazole with the combined characteristics of blue light-emitting and hole-transporting materials. These fluorescent compounds showed emission in the

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Scheme 4. Band diagrams for devices V-VII.



Figure 4. The I-V-L curve of devices V-VII with 1-3 as hole-transporting materials (ITO/HTM/Alq<sub>3</sub>/LiF/Al).

blue to green region with high quantum efficiencies of over 70% in the solution phase, and strong luminescence in the solid-phase thin film. All of these compounds were thermally stable and possessed high glass-transition temperatures, well above 160°C. Most importantly, these compounds showed promising potential as either blue-light-emitting materials or hole-transporting materials for Alq3-based OLEDs. Further studies on multi-layered device and device optimization are currently underway.

#### **Experimental Section**

Characterization data for 1: <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 8.37$  (s, 2H), 8.25 (d, J=9.2 Hz, 2H), 8.18 (d, J=7.6 Hz, 2H), 8.02-8.13 (m, 10H), 7.91-7.96 (m, 4H), 7.44 (d, J = 8.40 Hz, 2H), 7.56–7.60 (m, 4H), 7.45 (d, J = 8.0 Hz 4H), 2.49 ppm (s, 3H). <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta = 140.9$ , 138.5, 137.7, 133.1, 131.6, 131.1, 130.7, 130.4, 129.0, 128.2, 127.5, 127.4, 127.2, 127.1, 125.9, 125.7, 125.0, 124.7, 124.6, 123.5, 122.4, 120.4, 109.9, 21.3 ppm. MS(MAL-



DI-TOF) Calcd for C<sub>51</sub>H<sub>31</sub>N: 657.2. Found: 657.1. Anal. Calcd for C<sub>51</sub>H<sub>31</sub>N: C 93.12, H 4.75, N 2.13. Found: C 93.22, H 4.81, N 1.93.

Characterization data for 2: <sup>1</sup>H NMR  $(CDCl_3): \delta = 8.38$  (s, 4H), 8.10-8.14 (m, 8H), 7.88-8.00 (m, 24H), 7.73 (d, J = 9.32 Hz, 4H), 7.67 (d, J = 8.36 Hz, 4H), 7.44 (d, J=8.40 Hz, 4H), 5.11 (s, 4H), 4.55 ppm (s, 4H). <sup>13</sup>C NMR  $(CDCl_3): \delta = 168.5, 140.3, 138.0, 133.2,$ 131.4, 130.8, 130.2, 129.1, 128.6, 128.0, 127.3, 127.2, 127.1, 125.8, 125.3, 124.8, 124.6, 124.5, 123.6, 122.6, 108.4, 62.9, 44.8 ppm. MS(MALDI-TOF) Calcd

for C<sub>94</sub>H<sub>56</sub>N<sub>2</sub>O<sub>4</sub>: 1277.5 Found: 1277.6. Anal. Calcd for C<sub>94</sub>H<sub>56</sub>N<sub>2</sub>O<sub>4</sub>: C 88.38, H 4.42, N 2.19. Found: C 88.58, H 3.96, N 2.11.

Characterization data for 3: <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 8.44$  (s, 2H), 8.32 (d, J=9.10 Hz, 2 H), 8.26 (d, J=7.54 Hz, 2 H), 8.23-8.07 (m, 10 H), 8.06-7.95 (m, 4H), 7.73 (dd, J=22.42, 8.33 Hz, 4H), 7.61 (d, J=7.79 Hz, 1H), 7.45–7.06 ppm (m, 12 H). <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta = 147.6, 147.4, 141.0, 138.5,$ 133.2, 131.6, 131.3, 131.1, 130.4, 129.4, 129.0, 128.9, 128.2, 127.9, 127.5, 127.4, 127.2, 126.0, 125.7, 125.1, 125.0, 124.9, 124.7, 124.7, 124.1, 123.6, 122.7, 122.4, 109.9 ppm. MS(MALDI-TOF) Calcd for C<sub>62</sub>H<sub>38</sub>N<sub>2</sub>: 810.1. Found: 810.4. Anal. Calcd for C<sub>62</sub>H<sub>38</sub>N<sub>2</sub>: C 91.82, H 4.72, N 3.45. Found: C 91.51, H 4.51, N 3.53.

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