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Highly Efficient Orange and Warm White Phosphorescent OLEDs Based on a Host Material with a Carbazole–Fluorenyl Hybrid

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Abstract: A new carbazole–fluorenyl hybrid compound, 3,3′-(2,7-di(naphthaline-2-yl)-9H-fluorene-9,9-diyl)bis(9-phenyl-9H-carbazole) (NFBC) was synthesized and characterized. The compound exhibits blue-violet emission both in solution and in film, with peaks centered at 404 and 420 nm. In addition to the application as a blue emitter, NFBC is demonstrated to be a good host for phosphorescent dopants. By doping $Ir(2\text{-}phq)_3$ in NFBC, a highly efficient orange organic light-emitting diode (OLED) with a maximum efficiency of $32\,cd\,A^{-1}$ ($26.5\,Lm\,W^{-1}$) was obtained. Unlike most phosphorescent OLEDs, the device prepared in our study shows little efficiency roll-off at high brightness and maintains current efficiencies of 31.9 and $26.8\,cd\,A^{-1}$ at a luminance of 1000 and $10\,000\,cd\,m^{-2}$, respectively. By using NFBC simultaneously as a blue fluorescence emitter and as a host for a phosphorescent dopant, a warm white OLED with a maximum efficiency of $22.9\,Lm\,W^{-1}$ ($21.9\,cd\,A^{-1}$) was also obtained.

Introduction

Organic light-emitting diodes (OLEDs) have important applications in high-efficiency full-color flat panel displays and solid-state lighting.[1] OLEDs based on fluorescent emitters are theoretically capped to have an internal quantum efficiency (IQE) of about 25 % due to singlet–triplet statistics.[2] By making full use of both the singlet and the triplet states, phosphorescent OLEDs (PHOLEDs) could theoretically achieve an IQE of 100 %.[3] With the rapid development of OLEDs, highly efficient red and green PHOLEDs with a long lifetime have been demonstrated.[4,5] However, there are still several aspects that need to be improved in order to meet the demands of commercialization. Firstly, the performance of blue phosphorescent emitters is still considerably lower than those of their red and green counterparts.[6] Furthermore, phosphorescent emitters have often a poor lifetime, which is another obstacle for their commercialization. To address the inadequacy of blue phosphors, fluorescence/phosphorescence (F/P) hybrid OLEDs using blue fluorescent but red and green phosphorescent emitters have been proposed.[7] On the other hand, the significant efficiency roll-off in PHOLEDs is another challenge for its application in solid-state lighting. Many researchers have paid significant efforts into this issue and have demonstrated some effective strategies such as using dual-emitting layers (D-EMLs),[8] a co-host system, and new host materials.[9,10]

In this work, we designed a new blue-violet emitter, 3,3′-(2,7-di(naphthaline-2-yl)-9H-fluorene-9,9-diyl)bis(9-phenyl-9H-carbazole) (NFBC). An OLED using NFBC as a non-doped fluorescent emitter gives blue-violet emission with a peak at 428 nm and CIE coordinates of (0.16, 0.05). An orange OLED with a maximum efficiency of $32\,cd\,A^{-1}$ ($26.5\,Lm\,W^{-1}$) has been obtained by doping tris(2-phenylquinoline)iridium [$Ir(2\text{-}phq)_3$] into NFBC. It is interesting that the device presents an extremely low efficiency roll-off and maintains efficiencies of 31.9 and $26.8\,cd\,A^{-1}$ at a luminance of 1000 and $10\,000\,cd\,m^{-2}$ respectively. These performance parameters are much better than those of most reported orange or red/orange phosphorescent OLEDs. Furthermore, by combining the blue-violet fluorescence with the orange phosphorescent emission, an efficient warm white F/P OLED has been obtained with a maximum efficiency of $22.9\,Lm\,W^{-1}$ ($21.9\,cd\,A^{-1}$).

Results and Discussions

Scheme 1 depicts the synthetic routes and chemical structure of 3,3′(2,7-di(naphthaline-2-yl)-9H-fluorene-9,9-diyl)bis(9-phenyl-9H-carbazole) (NFBC). The molecular structure of

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NFBC was confirmed by ^{1}H-NMR spectroscopy, elemental analysis, and mass spectrometry. Thermal properties of NFBC were studied by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). NFBC shows a high thermal decomposition temperature (T_d, corresponding to a weight loss of 5 %) of 484 °C and a high glass transition temperature (T_g) of 183 °C (Figure 1).

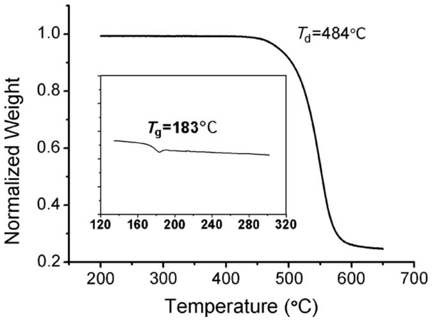


Figure 1. TGA and DSC (inset) measurements of NFBC.

Figure 2 shows the UV/Vis absorption and photoluminescence (PL) spectra of NFBC recorded at room temperature from a dilute CH_2Cl_2 solution as well as a neat film deposited on a quartz substrate. The PL spectra of NFBC show a blue-violet emission with maxima at 404 and 420 nm, respectively, in solution and in the solid film. Remarkably, the compound exhibits a narrow emission with full width at half maximum (FWHM) of about 40 nm in both spectra, which is helpful to obtain a saturated color in OLEDs. The fluorescence quantum yield (Φ) of NFBC in tetrahydrofuran (THF) solution was measured to be 0.5 by using 2-aminopyridine ($\Phi = 0.6$) as a standard, while that in the solid film was measured to be 0.58. NFBC has a HOMO level of 5.9 eV, as determined by UV photoelectron spectroscopy (UPS), and a LUMO level of 2.9 eV, which was obtained by subtracting the HOMO value with its optical gap of 3.0 eV. The triplet energy level of NFBC was determined to be 2.32 eV from its PL characteristics in a 2-methyltetrahydrofuran (2-MeTHF) solution measured at 77 K. This triplet energy is high enough to sensitize a common orange dopant (such as $Ir(2\text{-}phq)_3$, 2.0 eV). Key physical data of NFBC are listed in Table 1. Owing to its highly efficient blue-violet emission and high triplet level, NFBC should be a promising material as a blue-violet emitter and a host for OLED applications.

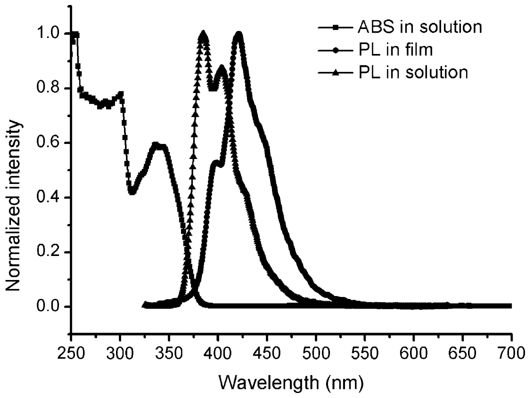


Figure 2. Absorption and photoluminescence spectra of NFBC.

Table 1. Key physical data of NFBC.

T_g [°C]	T_d [°C]	$\lambda_{em, max}$ (film/solution) [nm][a]	HOMO [eV]	LUMO [eV]	E_g [eV]	Φ (film/solution)[b]
183	484	420/404	5.9	2.9	3.0	0.58/0.5

[a] Emission maxima of NFBC in film and in CH_2Cl_2 solution. [b] Fluorescence quantum yield of NFBC in film and tetrahydrofuran (THF) solution using 2-aminopyridine ($\Phi = 0.6$) as a standard.

To investigate the blue-violet emission property of NFBC, a non-doped device with a configuration of ITO/NPB (40 nm)/TCTA (10 nm)/NFBC (20 nm)/TPBi (30 nm)/LiF (1 nm)/Al (80 nm) was fabricated. In the device, 4,4′-bis[N-(1-naphthyl)-*N*-phenylamino]biphenyl (NPB) is the hole-transporting layer (HTL); 4,4′,4″-tris(*N*-carbazolyl)triphenylamine (TCTA) is used as the electron-blocking layer (EBL), while 1,3,5-tris(*N*-phenylbenzimidazol)benzene (TPBi) works as an electron-transporting layer (ETL) as well as a hole-blocking layer (HBL) and NFBC is the emitting layer (EML). As shown in Figure 3, the device gives blue-violet emission with a maximum at 428 nm with CIE coordinates of (0.16, 0.05), which is shifted slightly over the brightness range of 10–1000 cd m^{-2}. The current density–voltage–luminescence (J–V–L) characteristics are shown in the inset. The device shows a turn-on voltage (defined as the driving voltage at a brightness of 1 cd m^{-2}) of 3.4 V. Figure 4 shows plots of the current efficiency and the external quantum efficiency as a function of luminance of the blue-violet device. The obtained maximum external quantum efficiency ($\eta_{ext, max}$) of 2.8 % and the maximum current efficiency of 0.8 cd A^{-1} (0.6 Lm W^{-1}) at a luminance of

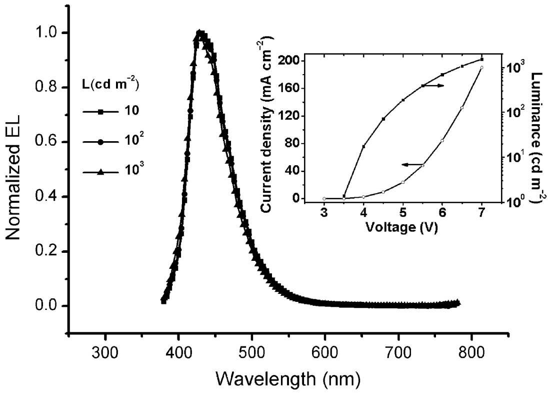


Figure 3. EL spectra at different luminance and J–V–L curve (inset) of the blue-violet device.

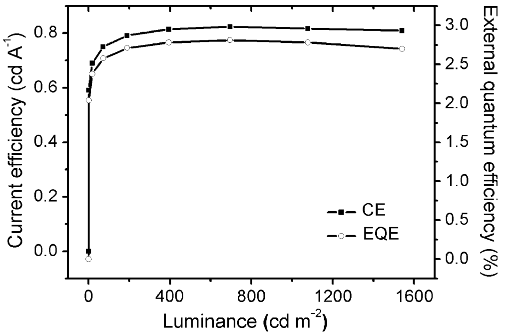


Figure 4. Current efficiency–luminance–EQE curve of the blue-violet device.

700 cd m^{-2} with a mild efficiency roll-off is comparable with those of reported devices with CIEy < 0.055.[12]

To evaluate the potential of NFBC as a host material for phosphorescent devices, a device using Ir(2-Phq)$_3$ as an orange dopant was fabricated with a configuration of ITO/NPB (30 nm)/TCTA (10 nm)/NFBC:2 % Ir(2-phq)$_3$ (30 nm)/TPBi (40 nm)/LiF (1 nm)/Al (80 nm). Figure 5 shows the EL spectra and the current density–voltage–luminance (J–V–L) characteristics (inset) of the device. The device has a turn-on voltage of 3.7 V and shows an orange emission with a maximum at 588 nm. The emission spectrum remains essentially unchanged over a wide range of brightness, thus suggesting that the exciton generation zone has been successfully confined. This is attributed to the high triplet energy levels of TCTA (2.76 eV) and TPBi (2.6 eV). Figure 6 shows efficiencies of the device at different brightness outputs. The device has a maximum current efficiency of 32 cd A^{-1}, a maximum power efficiency of 26.5 Lm W^{-1}, and a maximum external quantum efficiency of 15.3 %. It is noteworthy that the device shows a small efficiency roll-off and maintains current efficiencies of 31.6, 31.9, and 26.8 cd A^{-1}, respectively at 100, 1000, and 10 000 cd m^{-2}. This may be attributed to the sufficient triplet energy level of NFBC resulting in a complete energy transfer from NFBC to Ir(2-phq)$_3$. The high emission efficiency and mild efficiency roll-off of this device are comparable to the best values reported for orange or orange-red phosphorescent OLEDs with similar device structures (Table 2).[13]

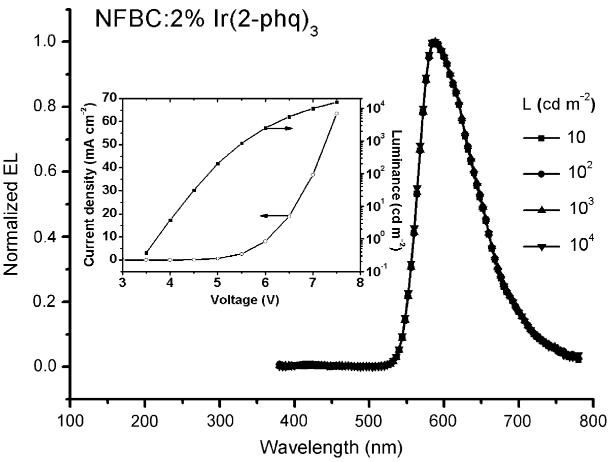


Figure 5. EL spectra at different luminance and J–V–L characteristics of the orange device.

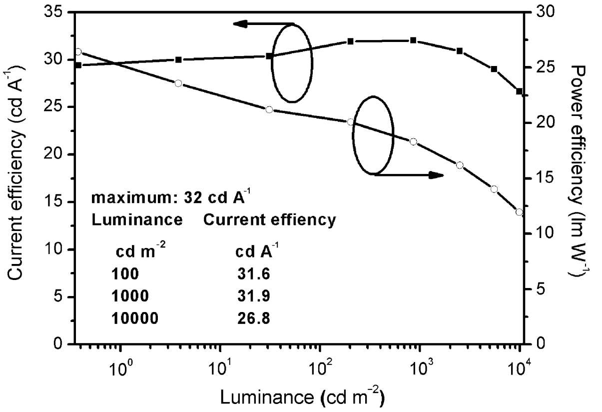


Figure 6. Current efficiency–luminance–power efficiency characteristics of the orange device.

Considering its blue-violet fluorescence and good performance as a host material, the merits of NFBC were exploited in F/P hybrid white OLEDs (WOLEDs) with configurations of: device I: ITO/NPB (30 nm)/NFBC: 2 % Ir(2-phq)$_3$ (5 nm)/NFBC (15 nm)/TPBi: 8 % Ir(ppy)$_3$ (20 nm)/TPBi (15 nm)/LiF (1 nm)/Al (80 nm) and device II: ITO/NPB (30 nm)/NFBC: 2 % Ir(2-phq)$_3$ (5 nm)/NFBC (20 nm)/TPBi:

Table 2. Summary of the emission characteristics of representative orange OLEDs.

Emission layer	EQE[a] [%]	$\lambda_{\text{em, max}}$ [nm]	$\eta_{\text{c.max}}$[b] [cd A^{-1}]	$\eta_{\text{c.100}}$[c] [cd A^{-1}]	$\eta_{\text{c.1000}}$[d] [cd A^{-1}]	$\eta_{\text{c.10000}}$[e] [cd A^{-1}]	CIE coordinates	Ref.
NFBC:Ir(2-phq)$_3$	15.3	588	32	31.6	31.9	26.8	(0.57,0.42)	This work[h]
DADBT:Ir(2-phq)$_3$	16.7	584	32.7	31.8	29.6	–	(0.56,0.43)	[13a][h]
BPPI:Ir(2-phq)$_3$	12.4	581	23.9	22.5	23	21.5	(0.61,0.39)	[13b][h]
DFBC:Ir(2-phq)$_3$	–	584	20.1	–	–	–	(0.55,0.42)	[11a][h]
CBP:(pbi)$_2$Ir(biq)	–	586	20.2	20	17.5	9.2	(0.53,0.46)	[13c][p]
CBP:Ir(TBT)$_2$(acac)	9.1	588	15.8	15.8	10.9	–	(0.58,0.41)	[13d][p]
CBP:(impy)$_2$Ir(acac)	12.7	568	34.2	32	25	10.5	(0.52,0.48)	[13e][p]
OTPD:x-emitter	–	570	18.4	18.4	15	–	(0.55,0.44)	[13f][p]
Bebq2:Ir(phq)$_2$acac	21.0	605	26.5	26	20.53	17.6	(0.62,0.37)	[13g][h]

[a] Maximum external quantum efficiency. [b] Maximum current efficiency. [c,d,e] Current efficiency at 100 cd m^{-2}, 1000 cd m^{-2}, and 10 000 cd m^{-2}, respectively. [h] New host materials for orange OLEDs. [p] New dopant for orange OLEDs.

8 % Ir(ppy)$_3$ (20 nm)/TPBi (15 nm)/LiF (1 nm)/Al (80 nm). Here, Ir(2-phq)$_3$ and tris(2-phenylpyridine)iridium (III) (Ir(ppy)$_3$) were used as orange and green phosphorescent emitters, respectively. Due to the high triplet energy of Ir(ppy)$_3$ (2.40 eV), TPBi was used as the host. Firstly, TPBi has a high enough triplet energy of 2.74 eV when compared with that of Ir(ppy)$_3$, which is beneficial for energy transfer. Furthermore, TPBi has good electron-transporting and hole-blocking properties, and thus the use of TPBi as the host is helpful for charge transport and recombination. The undoped NFBC layer was used for blue fluorescent emission. The structure and energy level diagram of the device are shown schematically in Figure 7. Figure 8 shows the EL spectra of the two devices at different luminance. As the luminance increases, CIE coordinates of the devices are slightly blue-shifted, which is attributed to a shift of the exciton generation zone. Figure 9 shows the corresponding current efficiency–luminance–power efficiency plots and J–V–L curves (inset) of devices I and II. Device I has a lower turn-on voltage of 3.1 V than that of device II (3.7 V). Device I shows a warm white emission with CIE coordinates of (0.45, 0.40) and a maximum power efficiency of 22.9 Lm W^{-1}, current efficiency of 21.9 cd A^{-1}, and an external quantum efficiency of 8.2 %, which is comparable or better than that of many reported F/P hybrid WOLEDs.[14] To further tune the color coordinates of the device, we increased the thickness of the NFBC layer. The optimized device (device II) shows CIE coordinates of (0.36, 0.34), which is very close to the standard white emission of (0.33, 0.33). The maximum efficiency values of the device are 13.4 Lm W^{-1}, 14.9 cd A^{-1}, and 5.7 % ($\eta_{\text{ext, max}}$).

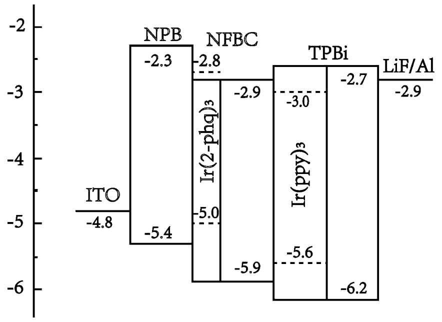


Figure 7. The device structure of the WOLED and energy levels of the materials.

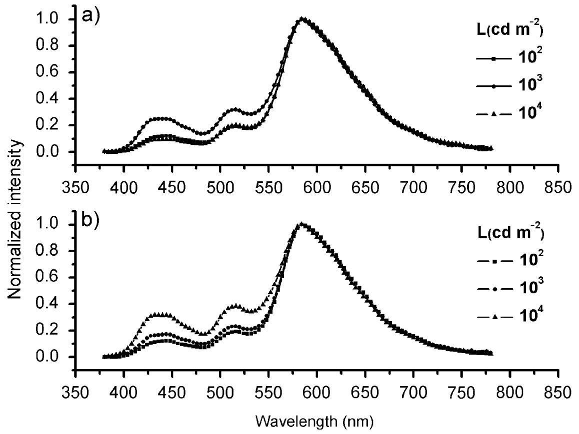


Figure 8. EL spectra of a) device I and b) device II at different luminance.

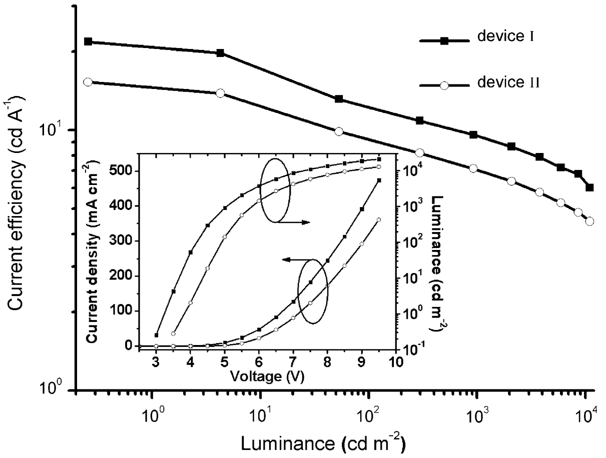


Figure 9. Current efficiency–luminance plot and J–V–L curve (inset) of devices I and II.

Conclusions

In summary, we synthesized and characterized a new carbazole-fluorenyl-based compound, that is 3,3′(2,7-di(naphthaline-2-yl)-9H-fluorene-9,9-diyl)bis(9-phenyl-9H-carbazole) (NFBC), which possesses a high glass transition temperature (T_g) of 183 °C and a decomposition temperature (T_d) of 484 °C. The compound shows blue-violet emissions both in solution and in a film, with peaks centered at 404 and 420 nm. An OLED using NFBC as a non-doped fluorescent emitter gives blue-violet emission with a maximum at 428 nm and CIE coordinates of (0.16, 0.05). Moreover, highly efficient orange emission has been achieved by using NFBC as the host doped with Ir(2-phq)$_3$, with a maximum efficiency of 32 cd A^{-1} (26.5 Lm W^{-1}). It is noteworthy that the device presents an extremely low efficiency roll-off, with efficiencies of 31.6, 31.9, and 26.8 cd A^{-1}, respectively, at100, 1000, and 10000 cd m^{-2}, which is much better than that of most orange or red/orange phosphorescent OLEDs reported recently. Furthermore, an efficient warm white emission has been obtained with a maximum efficiency of 22.9 Lm W^{-1} (21.9 cd A^{-1}) with the F/P device structure. These results suggest that NFBC is a promising multifunctional material for high-performance OLED applications.

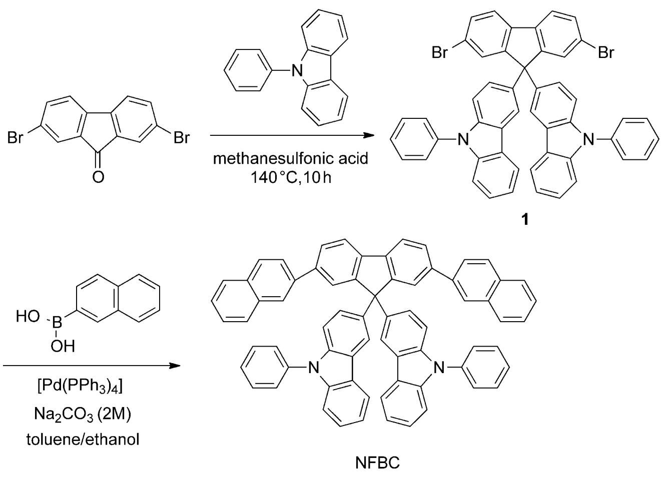
Experimental Section

Chemicals and Instrumentation

All reactions were carried out under a nitrogen atmosphere and all powders and solvents were used as received from the commercial suppliers without further purification. The final products were confirmed by using ^{1}H-NMR spectroscopy, elemental analysis (EA), and mass spectrometry (MS). The glass transition temperature (T_g) was measured by differential scanning calorimetry (DSC) using a PerkinElmer Pyris DSC6 system operated at a heating rate of 10 °C min^{-1}. The decomposition temperature (T_d) was measured by thermogravimetric analysis (TGA) on a TA SDT Q600 instrument at a heating rate of 10 °C min^{-1} under a nitrogen flow. Optical absorption and emission spectra were recorded with a Hitachi U-3010 UV/Vis spectrophotometer and a Hitachi F-4500 fluorescence spectrophotometer, respectively. The highest occupied molecular orbital (HOMO) energy level was measured using ultraviolet photoelectron spectroscopy (UPS). The lowest unoccupied molecular orbital (LUMO) energy level was determined from the difference between the HOMO energy level and the energy gap (E_g) obtained from the optical absorption edge.

Synthesis

3,3′-(2,7-Dibromo-9H-fluorene-9,9-diyl)bis(9-phenyl-9Hcarbazole) (**1**, see Scheme 1) was synthesized according to previously reported methods.[11]



Scheme 1. Synthesis of NFBC.

3,3′(2,7-Di(naphthaline-2-yl)-9H-fluorene-9,9-diyl)bis(9-phenyl-9H-carbazole) (NFBC)

NFBC was obtained through a classical Suzuki cross-coupling reaction: Compound **1** (0.81 g, 1 mmol), 2-naphthaleneboronic acid (0.43 g, 2.5 mmol), $[Pd(PPh_3)_4]$ (0.1 g, 0.087 mmol), aqueous Na_2CO_3 (2.0 M, 20 mL), ethanol (15 mL), and toluene (20 mL) were mixed in a flask. The mixture was degassed three times and then heated under reflux at 100 °C for 24 h under a nitrogen atmosphere. After cooling of the reaction mixture to room temperature, the solvent was evaporated. The resulting residue was then extracted with CH_2Cl_2, washed with water, and dried over $MgSO_4$. The crude product was purified by column chromatography on silica gel (petroleum ether/CH_2Cl_2, 3:1). The product was then further purified by recrystallization with petroleum ether and CH_2Cl_2 to give NFBC as a white solid. Yield: 0.58 g (65 %); ^{1}H-NMR ($CDCl_3$, 300 MHz): $\delta = 7.14$–7.25 (t, 2H), 7.33–7.42 (m, 6H), 7.43–7.64 (m, 16H), 7.66–7.78 (t, 4H), 7.83–7.93 (t, 4H), 7.93–7.98 (t, 4H), 7.98–8.07 (d,2H), 8.12–8.17 (s, 2H), 8.17–8.24 (s, 2H), 8.24–8.35 ppm (s, 2H); MS (ESI$^+$): m/z 900 (M^+); elemental anal. calcd (%) for $C_{69}H_{44}N_2$: C 92.00, H 4.89, N 3.11; found: C 91.57, H 5.09, N 3.06.

Device Fabrication and Measurements

Indium tin oxide (ITO)-coated glass with a sheet resistance of 15 Ω per square were used as substrates. Before device fabrication, the ITO glass substrates were washed with isopropyl alcohol and deionized water, dried in an oven at 120 °C for 2 h, and then treated with UV light-ozone for 30 min before they were loaded into a vacuum deposition chamber with a base vacuum $<10^{-6}$ Torr. Organic layers were successively deposited onto the ITO glass substrates with a rate of 1–2 Å s^{-1}. An electron-injecting LiF layer and an Al cathode were deposited with rates of 0.1 and 10 Å s^{-1}, respectively. Electroluminance (EL) spectra, CIE coordinates, and current density–voltage–luminance (J–V–L) characteristics were measured with a computer-controlled Keithley 2400 source meter and a Spectrascan PR650 photometer under ambient atmosphere.

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Keywords: blue fluorescent host · doping · fluorescence · OLEDs · phosphorescence · warm white emission

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