

New Host Material for High-Performance Blue Phosphorescent Organic Electroluminescent Devices

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In recent years, a considerable amount of attention has been paid toward the development of organic emissive materials for potential applications in full-color flat-panel displays and lighting sources. A number of red and green light-emitting materials have already been identified for use in phosphorescent organic light-emitting diodes (PhOLEDs). PhOLEDs based on these materials are characterized by high luminous efficiency, reasonable color purity, and long lifetimes. However, the development of blue phosphorescent OLEDs with high electroluminescence (EL) efficiency remains a challenge because a high bandgap energy and high triplet energy are required for blue emitters. To avoid reverse energy transfer from the guest to the host and to confine triplet excitons in the emitting layer, the singlet and triplet levels of the host should be higher than those of the phosphorescent dopants. This is still a rather challenging task for material researchers. Additionally, the large triplet bandgap materials have to have high thermal, electrochemical, and morphological stability to improve operational stability and maintain charge transport in devices; they must also have sufficiently high singlet and triplet energy levels to confine them both (e.g., ≥ 2.7 eV for Firpic) in the emitting zone.^[1–8] 1,3-Bis(carbazolyl-9-yl)benzene (mCP) has been widely used as a host material in blue PhOLEDs owing to its highly efficient hole transport properties and a wide triplet bandgap of 2.90 eV for efficient energy transfer. However, its relatively low thermal and morphological stability may hinder its application as a host in a blue PhOLED ($T_g = 65$ °C). Silicon-based wide triplet bandgap materials have been used for highly efficient blue PhOLEDs. However, the highest occupied molecular orbital (HOMO) level of UGHs is around 7.0 eV.⁴ Such a deep HOMO level makes it difficult to balance holes and electrons in the emitting zone. To improve the morphological stability, Chen and Wang independently reported an mCP derivative with a silane moiety, 3,5-di(*N*-carbazolyl)tetrakisphenylsilane (SimCP) ($T_g = 101$ °C).^[9,10] An OLED device fabricated using SimCP as a host, exhibited a maximum power efficiency of 11.9 lm/W and an external quantum efficiency of 14.4% when using the Firpic dopant (iridium(III)bis[(4,6-di-fluorophenyl)-pyridinato-*N,C2'*]

picolate). The higher performance of a SimCP-based device, compared to an mCP-based device (12.3%, 9.3 lm/W), was attributed to a hindering effect on the packing of the molecular condensed phase that occurs because of the bulky and rigid structure of SimCP, thus alleviating T1–T1 annihilation.^[10]

In addition, a new electrochemically stable host material for blue PhOLEDs, 9-(4-*tert*-butylphenyl)-3,6-bis(triphenylsilyl)-9H-carbazole (CzSi), was synthesized by directly linking two triphenylsilyl moieties to the C3 and C6 positions of carbazole, which are electrochemically active sites.^[11] The device using CzSi as a host achieved a maximum external quantum efficiency of 15.7%, corresponding to a current efficiency of 30.6 cd/A and a power efficiency of 26.7 lm/W.^[11] Other than these, there have been many other reports on the development of blue PhOLEDs, thus demonstrating their significance. However, despite constant efforts to accomplish high device efficiency, the development of blue PhOLEDs with high electroluminescence (EL) efficiency remains a challenge because of difficulties in obtaining materials with a wide triplet bandgap, the proper energy levels, and mobility for balanced charge injection.^[12,13]

Herein, we describe the design and synthesis of a new carbazole derivative that has two carbazole moieties on the C3 and C6 positions of carbazole and a triphenylsilane moiety directly linked to the N of the carbazole. This new carbazole derivative is expected to have improved thermal properties, while maintaining its advantageous electronic and optical characteristics. The non-planar molecular structure of a large gap and the non-conjugated triphenylsilane end-capping group would prevent close packing of the molecules in the solid state and enable the formation of smooth and pinhole-free thin films.^[14]

Scheme 1 (in the Supporting Information (SI)) shows the synthetic route to 9'-triphenylsilylanyl-9'-H-[9,3',6',9']tercarbazole (SitCz). Specifically, the *N*-unsubstituted carbazole (CzG2on)^[15] is treated with 2.5 M *n*-BuLi at -78 °C to give a lithiated intermediate, which is subsequently quenched with chlorotriphenylsilane to give the desired SitCz in 76% yield after column chromatography. The structure was confirmed by ¹H NMR spectroscopy, ¹³C NMR spectroscopy, and mass spectrometry (MS).

The thermal behavior of SitCz was evaluated by means of (a) differential scanning calorimetry (DSC) and (b) thermogravimetric analysis (TGA) under a nitrogen atmosphere. A 5% weight loss was observed at 402 °C (see SI, Figure S1(b)). The melting point (T_m) of SitCz was found to be 346 °C and the glass transition temperature (T_g) was 168 °C (SI, Figure S1). In general in OLEDs, amorphous thin films that possess high T_g values are less vulnerable to heat. This results in more stable device performances owing to the retention of the film morphology during the operation of the devices.^[16] SitCz is therefore more thermally stable than mCP or SimCP. To confirm the morphological stability against thermal stress, we also carried

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out an atomic force microscopy (AFM) study of a vacuum-deposited SitCz film (see SI, Figure S1(c)). These data indicate that SitCz is stable enough to endure the high temperature at which vacuum vapor deposition is carried out.

To determine the optimized geometry and the frontier molecular orbital levels, density functional theory (DFT) calculations were carried out at the B3LYP/6-31G* using Spartan '08. As shown in SI, Figure S2, neither the HOMO nor the lowest unoccupied molecular orbital (LUMO) is localized on the triphenylsilane benzene rings, suggesting that the introduction of the silyl group on CzG2on would not result in a significant change in the HOMO or LUMO levels. The triphenylsilane group therefore serves as an effective spacer and blocks the π -conjugation of the carbazole core from extending to the peripheral substitution. The calculated HOMO and LUMO levels of SitCz were -5.11 and -1.04 eV, respectively.

The electrochemical properties of SitCz were characterized by cyclic voltammetry. The oxidation scans were carried out in a 0.1 M solution of tetraethylammonium tetrafluoroborate in anhydrous dichloromethane. A platinum disk and an Ag/AgCl disk were used as the working and reference electrodes, respectively. The platinum disk was used as the counter electrode. Based on the HOMO energy of the ferrocene/ferrocenium redox system (-4.8 eV), we were able to calculate the HOMO energy values of SitCz, which was equal to -5.54 eV (see SI, Figure S3).

Figure 1 shows the room-temperature UV-vis absorption and PL spectra dissolved in dichloromethane and low-temperature PL spectra of SitCz in a 2-methyltetrahydrofuran (2-MeTHF) solution at 77 K. The absorption spectra of SitCz are nearly similar to those of the unsubstituted carbazole monomer. The bandgap energies (3.24 eV) were measured from the absorption spectra, and the LUMO energies were estimated to be -2.30 eV for SitCz (E_{LUMO} (eV) = $-(E_{\text{HOMO}} - E_{\text{g}}^{\text{opt}})$). The phosphorescence of SitCz was obtained in a frozen 2-methyltetrahydrofuran solution at 77 K. The highest energy phosphorescent emission peak was located at about 414 nm, as the transition energy of $T_1 \rightarrow S_0$ is estimated to be as high as 3.00 eV, which is similar to that

of TCz1.^[17] In general, a higher triplet energy of the host material would facilitate energy transfer from host to dopant in the host-guest system. Thus, we would expect that SitCz would provide effective confinement of the triplet excitons on the guest ((Firpic); $E_{\text{T}} = 2.62$ eV) and provide the consequential prevention of back energy transfer from the dopant (Firpic) to the host (SitCz) material. From these results, we postulate that SitCz can be considered a promising host for a blue phosphorescent OLED.^[18]

We fabricated two types of multilayer structures: ITO/HATCN (50 Å)/NPB (550 Å)/TAPC (100 Å)/mCP: 10 wt% Firpic (250 Å)/TmPyPB (300 Å)/LiF (5 Å)/Al (1000 Å) (Device I) and ITO/HATCN (50 Å)/NPB (550 Å)/TAPC (100 Å)/SitCz: 10 wt% Firpic (250 Å)/TmPyPB (300 Å)/LiF (5 Å)/Al (1000 Å) (Device II). We used strongly electron-deficient and disk-shaped depyrazino[2,3-f:2',3'-h]quinoxaline-2,3,6,7,10,11-hexacarbonitrile (HATCN) as the hole-injection layer (HIL).^[19–22] We used N,N'-bis(naphthalene-1-yl)-N,N'-bis(phenyl)-benzidine (NPB) as the hole-transport layer (HTL) and di-[4-(N,N-ditoly-amino)-phenyl]cyclohexane (TAPC) as the electron blocking layer.^[23] TAPC has a HOMO energy level of -5.5 eV and a LUMO energy level of -2.0 eV.^[24] Additionally, TAPC has a higher E_{T} (2.87 eV) than the typical blue phosphorescent guest material, Firpic.^[18] mCP or SitCz with a nearly optimal concentration (10 wt%) of Firpic was used as the emitting layer. 1,3,5-tri[(3-pyridyl)-phen-3-yl]benzene (TmPyPB) was used as the electron-transport layers (ETL), and LiF as the electron-injection layer (EIL) at the LiF/Al cathode interface. TmPyPB (HOMO = -6.68 eV, LUMO = -2.73 eV, and $T_1 = 2.78$ eV) has a wider triplet energy level than the guest (Firpic) and thus effectively confines triplet excitons inside the emitting zone. Additionally, TmPyPB has good electron injection and transport properties because of the three pyridine-containing triphenylbenzene; its electron mobility is 1.0×10^{-3} cm²/V·s at 6.4×10^5 V/cm. This high electron mobility is necessary to obtain a high-efficiency blue PhOLED.^[25,26]

Figure 2 shows the EL spectra of the 10% doped device driven at 10 mA/cm². Both devices, in which mCP and SitCz were used as the blue phosphorescent hosts, gave similar EL spectra. The

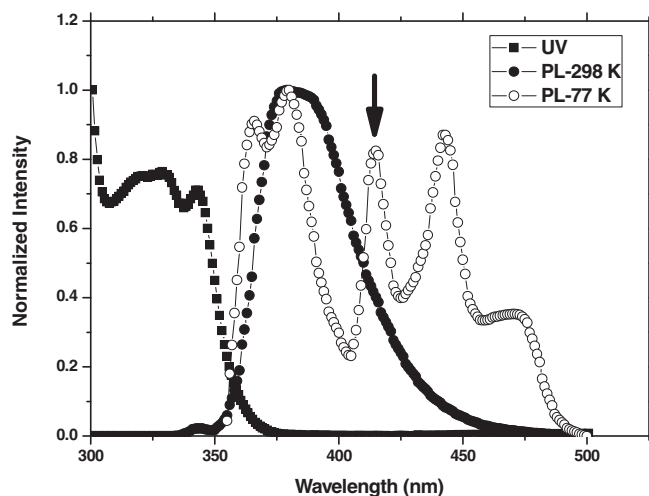


Figure 1. UV-vis absorption and PL spectra of SitCz. Room temperature spectra were taken in dichloromethane and 77 K emission spectrum (open circle) in 2-MeTHF frozen glass.

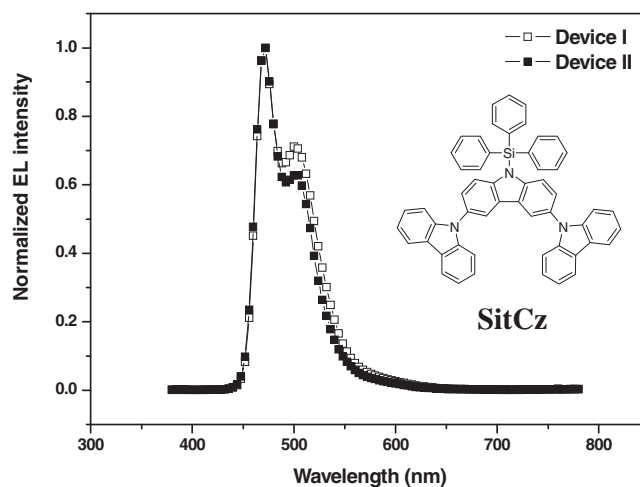


Figure 2. EL spectra of devices I and II along with the structures of SitCz.

Table 1. Summary of the characteristics of devices I and II with mCP and SitCz as hosts.

		Voltage [V]	Current Efficiency [cd/A]	Power Efficiency [lm/W]	External Quantum Efficiency [%]	CIE Coordinate (x,y)	Brightness [cd/m ²]
Device I (mCP)	At 100 cd/m ² / At 1000 cd/m ²	3.6 ^{a)} / 4.6/ 5.8	32.7/ 31.6	22.3/ 17.2	17.9/ 17.4	(0.14, 0.33)/ (0.14, 0.32)	4986 (at 10.2 V)
Device II (SitCz)	At 100 cd/m ² / At 1000 cd/m ²	2.9 ^{a)} / 3.9/ 5.0	46.3/ 44.0	36.5/ 27.2	27.2/ 25.6	(0.13, 0.29)/ (0.13, 0.29)	10514 (at 8.4 V)

^{a)}The applied voltage (V_{on}) required for brightness of 1 cd/m².

EL spectra of the mCP- and SitCz-based devices show identical patterns to Firpic emission without any emission from the host and/or adjacent layer. The EL results suggest that the complete energy transfer from mCP or SitCz (host) to Firpic (dopant) takes place upon electrical excitation, and charge carriers and excitons are perfectly confined in the emitting zone. Generally, Firpic is a light-blue emitter (about 450–500 nm) with a typical maximum emission peak around 472 nm and a vibrational peak at 500 nm, with a CIE (x,y) around (0.17, 0.34).^[27,28,11] However, the EL spectra of the Firpic-doped blue phosphorescent devices I and II developed here show more blue-shifted emission with a CIE (x,y) around (0.14, 0.33) and (0.13, 0.29) at 100 cd/m², respectively (Table 1). In the mCP device, the strong vibrational peak at 500 nm degraded the color performance of the Firpic-doped device, whereas the vibrational peak at 500 nm was relatively more suppressed in the SitCz device. The vibrational peak was intensified in the mCP device because of the shifted recombination zone from the HTL side to the ETL side.^[29]

Figure 3 shows the EL performances of devices I and II, and their EL efficiencies are summarized in Table 1. In particular, device I shows a turn-on voltage of 3.6 V at a luminance of 1 cd/m², a current efficiency of 32.7 cd/A, a power efficiency of 22.3 lm/W, and an external quantum efficiency of 17.9% at a corresponding brightness of 100 cd/m² with CIE coordinates of (0.14, 0.33). The performance using an mCP host for a blue phosphorescent OLED is comparable with the previously reported values. Device II, on the other hand, showed a better performance with a very low turn-on voltage of 2.9 V, a current efficiency of 46.3 cd/A, a power efficiency of 36.5 lm/W, and an external quantum efficiency of 27.2% at a corresponding brightness of 100 cd/m² with CIE coordinates of (0.13, 0.29). The performance is the highest values for oligomer carbazole derivatives with Firpic-based devices.^[17,30–32] To further verify the difference between the EL performances of devices I and II, their bandgap energies were examined. Figure 4 shows the HOMO and LUMO energy levels of the organic materials used in each layer. This energy diagram reveals that the energy barrier (0.60 eV) between the HOMO of NPB (5.30 eV) and the HOMO of mCP (5.90 eV) in device I is larger than the difference (0.24 eV) between the HOMO of NPB and the HOMO of SitCz (5.54 eV) in device II. On the other hand, the energy barrier (0.43 eV) from the LUMO of TmPyPB (2.73 eV) to the LUMO of SitCz (2.30 eV) is slightly larger than the barrier (0.33 eV) for electron injection from the LUMO of TmPyPB to the LUMO of mCP. Since mCP is characterized by a lower HOMO than SitCz, hole transportation from the HTL to the 10% doped emission layer is more difficult to achieve in device I. In addition, this difference

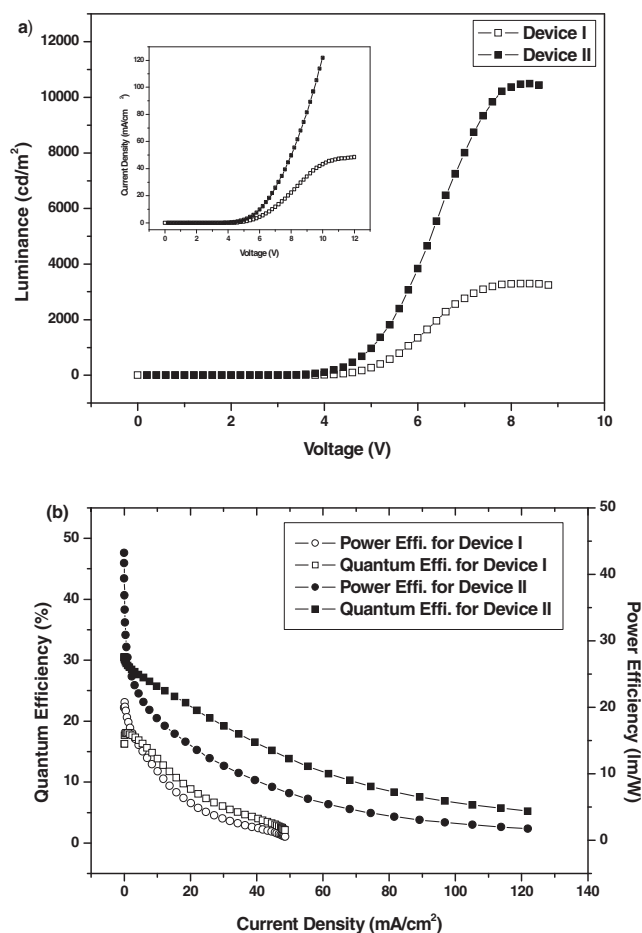


Figure 3. EL performance of devices I and II. (a) Luminance and voltage characteristics of device I and II. Inset: Current density vs. voltage. (b) Quantum efficiency-power efficiency-current density characteristics of devices I and II.

can explain why the electron-hole recombination may be unbalanced in the emitting zone of device I. This unbalanced charge recombination also shifts the blue light-emitting zone to a region close to the ETL, and therefore, device I shows a poor performance.

The maximum efficiency in these devices was achieved with a current efficiency of 47.1 cd/A, a power efficiency of 47.7 lm/W, an external quantum efficiency of 29%, and a brightness of 10514 cd/m². Moreover, when the brightness reached from 100 cd/m² to 1000 cd/m², the performance of

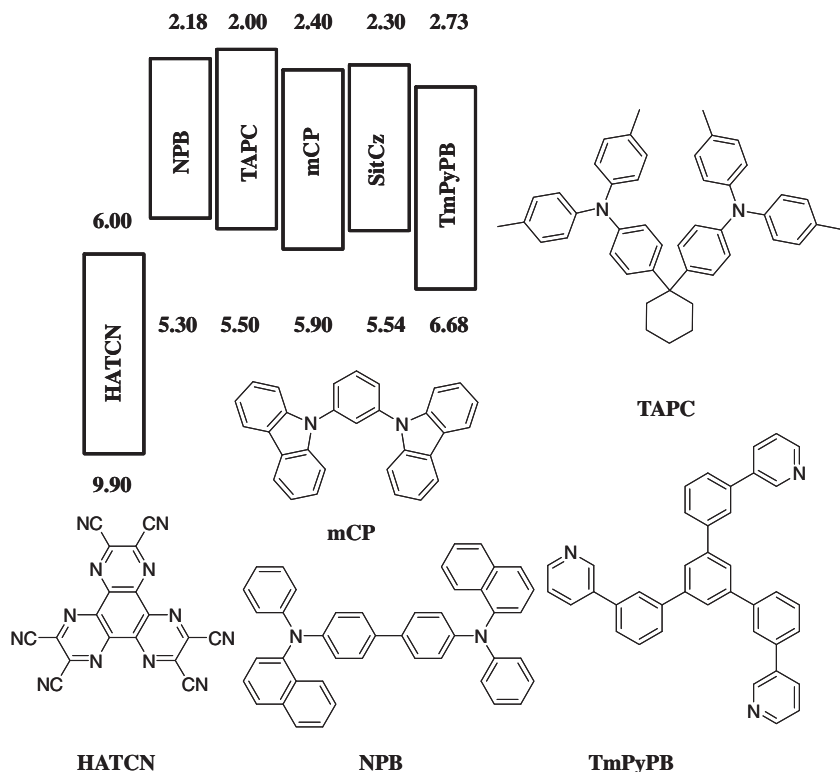


Figure 4. Energy diagram and chemical structures of materials used for fabrication of devices I and II.

device II was still higher than device I with a current efficiency of 44 cd/A, a power efficiency of 27.2 lm/W, and an external quantum efficiency of 25.6% at CIE coordinates of (0.13, 0.29). To the best of our knowledge, these performances are the best among blue PhOLEDs, when using a Firpic dopant, which have been reported. The improved performance can be attributed to the charge balance of electron and hole fluxes in the emitting zone because the internal quantum efficiency of the EL device is directly related to the balance of electrons and holes in the device.^[33,34] Thus, we expect that host materials that have a high triplet state (E_T) with a triphenylsilane moiety directly attached to the carbazole N would possess potential for effective charge balance and morphological stability in the emitting zone.

In summary, we have designed and synthesized a new host material (SitCz) that has two carbazole moieties on the C3 and C6 positions of carbazole and a triphenylsilane directly linked to the N of the carbazole. The higher T_g value (168 °C) of SitCz compared to mCP or SimCP represents the significantly enhanced morphological and thermal stability of SitCz. The device with the optimized structure, ITO/HATCN (50 Å)/NPB (550 Å)/TAPC (100 Å)/SitCz:Firpic 10 wt% (250 Å)/TmPyPB (300 Å)/LiF (5 Å)/Al (1000 Å), is characterized by high performance with a current efficiency of 46.3 cd/A, a power efficiency of 36.5 lm/W, an external quantum efficiency of 27.2%, and CIE coordinates of (0.13, 0.29) at 100 cd/m². The performance is the best not only among Firpic-doped blue PhOLEDs but also among all the blue PhOLEDs reported to date. Although, mainly, blue

light-emitting devices are studied here, such large gap host materials may also be of use for green and red phosphorescent devices. Further application of the large gap host materials in green, red, and white light-emitting devices is underway and the results will be reported in due course.

Experimental Section

Synthesis of SitCz: CzG2on (2 g, 4.02 mmol) was dissolved in 200 mL of anhydrous tetrahydrofuran (THF) under a flow of N₂. After cooling to -78 °C, *n*-butyllithium (2.5 M, 4.42 mmol, 1.77 mL) was added into the THF solution of CzG2on. Chlorotriphenylsilane (1.78 g, 6.03 mmol) was added dropwise to the lithiated CzG2on at -78 °C for 30 min and stirred for 2 h. The reaction mixture was then allowed to warm to room temperature for 12 h. Finally, 50 mL of water was poured into the mixture to quench the reaction, and the resulting product was extracted with chloroform, purified by silica gel column chromatography (10% chloroform in *n*-hexane used as an eluent), and further purified by recrystallization (yield: 76%).

¹H NMR (300 MHz, CDCl₃, δ): 6.93 (d, 2H, *J* = 8.8 Hz), 7.21–7.39 (m, 14H), 7.50–7.60 (m, 9H), 7.82 (d, 6H, *J* = 6.8 Hz), 8.15 (d, 4H, *J* = 6.8 Hz), 8.23 (s, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 144.51, 141.56, 136.11, 131.84, 131.04, 130.40, 128.65, 127.20, 125.77, 125.15, 123.14, 120.21, 119.62, 118.70, 116.11, 109.755. HRMS (FT MS) *m/z*: calcd. for [C₅₄H₃₇N₃Si + H]⁺: 756.2836, found [M + H]⁺: 756.2834.

Device Fabrication: Depyrazino[2,3-*f*:2',3'-*h*]quinoxaline-2,3,6,7,10,11-hexacarbonitrile (HATCN), *N,N'*-bis(naphthalene-1-yl)-*N,N'*-bis(phenyl)benzidine (NPB), Di-[4-(*N,N*-ditolyl-amino)-phenyl]cyclohexane (TAPC), 1,3-bis(carbazolyl-9-yl)benzene (mCP), and 1,3,5-tri[(3-pyridyl)-phenyl]benzene (TmPyPB) were purchased from Luminescence Technology Corp. OLEDs were fabricated by vacuum deposition onto patterned ITO glass that had been thoroughly cleaned and subsequently treated with oxygen plasma. Blue phosphorescent OLEDs were sequentially fabricated onto the ITO substrates through thermal evaporation of organic layers (evaporation rate: 2 Å/s; base pressure: 3 × 10⁻⁶ Torr). The EL spectra and CIE color coordinates were obtained using a Spectrascan PR650 photometer, and the current–voltage–luminescence (*J*–*V*–*L*) characteristics were measured using a Keithley 2400 source unit.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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