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Current Issue

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Organic Soluble Deoxyribonucleic Acid (DNA) Bearing Carbazole Moieties and Its Blend with Phosphorescent Ir(III) Complexes

MIN JU CHO,¹ U RA LEE,¹ YOUN SUN KIM,¹ JICHEOL SHIN,¹ YOUNG MIN KIM,² YOUNG WOOK PARK,² BYEONG-KWON JU,² JUNG-IL JIN,¹ DONG HOON CHOI¹

¹Department of Chemistry, Advanced Materials Chemistry Research Center, Korea University, 5 Anam-dong, Sungbuk-gu, Seoul 136-701, Korea

²Display and Nanosystem Laboratory, College of Engineering, Korea University, 5 Anam-dong, Sungbuk-gu, Seoul 136-701, Korea

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ABSTRACT: A functionalized deoxyribonucleic acid (Cz-DNA) was prepared with carbazolyl ammonium lipid as a triplet host material for phosphorescent material system. It is soluble in organic solvents, which facilitates the sample preparation for the absorption and luminescent properties in solid states. A highly soluble iridium complex, Ir(Cz-ppy)₃ with carbazolyl-substituted 2-phenyl-pyridine ligands was employed for studying the phosphorescence in Cz-DNA. There is a good overlap between the photoluminescence spectrum of Cz-DNA and the metal-to-ligand charge transfer (MLCT) absorption bands of the iridium complex. This overlap enables efficient energy transfer from the excited state in the host to the MLCT band of Ir(Cz-ppy)₃. In addition, photoluminescence quantum yield of Cz-DNA was found to be rela-

INTRODUCTION In the recent times, some interesting applications of deoxyribonucleic acid (DNA), which is a naturally occurring material with unique electronic,^{1,2} optoelectronic,^{3,4} photonic,⁵ and magnetic properties,⁶ have been discovered.

In particular, double-stranded (ds) DNA obtained from salmon sperm are interesting biodegradable and environmentally friendly biomacromolecules. Because of the regular sequence of the four base pairs in ds-DNA and the abundance of π electrons, ds-DNA can be used in novel electronic devices as an electroactive material.⁷⁻¹² However, the hydrophilic/hygroscopic nature of natural ds-DNA interferes with the study of electronic properties under ambient conditions even in an inert atmosphere.

Nevertheless, Adachi et al. reported the fabrication of DNAlipid complex-based organic light-emitting diodes (OLEDs) and characterized their properties. A DNA-CTMA (cetyltrimethylammonium) lipid complex was used as an active carrier transport layer by doping *fac*-tris(2-phenylpyridinato- C^2 ,N)iridium(III). However, poor electron injection from the cathode side hampered the improvement of the device efficiency due tively larger than the copolymer (PCzSt) with vinylcarbazole and styrene. Thus, Cz-DNA was employed as a triplet host material for fabricating multilayered electrophosphorescence devices via modification of its property by doping 5,4-*tert*-butylhexyl-1,3,4-oxadiazole (PBD). After doping 30 wt % PBD and 10 wt % lr(Cz-ppy)₃ into Cz-DNA, we achieved much improvement in electron injection/transport from an adjacent carrier transport layer, resulting in much improved device performances. © 2010 Wiley Periodicals, Inc. J Polym Sci Part A: Polym Chem 48: 1913–1918, 2010

KEYWORDS: biopolymers; carbazole; electrophosphorescence; iridium complex; light-emitting diodes (LED); luminescence; photoluminescence

to the presence of shallow lowest unoccupied molecular orbital (LUMO) level. 13

Steckl et al. also demonstrated the use of DNA-CTMA complex films as electron blocking layers (EBL) in organic lightemitting devices (OLEDs). The use of DNA-CTMA complex films led to significant enhancement of luminance and device efficiency over conventional device structures.^{2–4}

A few report about the use of organically functionalized DNA as an emitting host material for electrophosphorescent (EL) devices has been made in the literature. It might be due to the fact that those ds-DNAs inherently do show poor charge transport properties due to the non-conjugated DNA backbone and long alkylene spacers. Nevertheless, in this study carbazole-containing DNA (Cz-DNA) was used as a triplet host material for fabricating multilayered electrophosphorescence devices via modification of its property by doping 5,4-*tert*-butylhexyl-1,3,4-oxadiazole (PBD) in this study.

We synthesized organically soluble DNA containing dodecylcarbazolyl side-chain moieties (Cz-DNA) that make the DNA less hydrophilic and improve the host properties such as

Correspondence to: D. H. Choi (E-mail: dhchoi8803@korea.ac.kr)

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FIGURE 1 Chemical structures of Cz-DNA (a), PCzSt (m:n = 1:2.2) (b), and POx-TPACz (c).

carrier mobility, miscibility, and the effect of energy transfer on the guest Ir(III) complex. Cz-DNA showed good film-forming properties under ambient conditions. Vinylcarbazole copolymer (PCzSt) with styrene was synthesized having the identical mass percent of carbzaole unit with that in Cz-DNA. The absorption and emission spectra of *fac*-tris(3-((*9H*-carbazol-9-yl)methyl)-2-phenylpyridinato-C²,N)iridium (III) Ir(Czppy)₃¹⁴ doped samples using two polymer hosts were compared to investigate the DNA effect.

A new photoreactive polymer (POx-TPACz)-bearing oxetane moieties in the side chain was also employed as a material for buffer layer on poly(3,4-ethylenedioxythiophene) poly(styre-nesulfonate) (PEDOT:PSS) layer. We used a double layer of PEDOT:PSS and POx-TPACz in multilayer electrophosphores-cence PLEDs and Ir(Cz-ppy)₃¹⁴ as an emissive carrier recombination center in a device. Much improvement in luminous efficiency and brightness was achieved by doping Ir(Cz-ppy)₃ into Cz-DNA, which was blended with an electron-transport molecule 5,4-*tert*-butylphenyl-1,3,4-oxadiazole (PBD).

EXPERIMENTAL SECTION

Synthesis

Cz-DNA and POx-TPACz were synthesized by following our previous methods.^{15,16} PCzSt was synthesized with the feeding ratio of vinylcarbazole and styrene for coinciding the mass percent with that in Cz-DNA, following the literature method.¹⁷

Characterization

Metal ions and impurities were determined at the Korea Basic Science Institute (Seoul, Korea) using inductively coupled plasma atomic emission spectrometry (ICP-AES; Ultima 2C, Jobin Yvon Horiba). The redox properties of the synthesized compounds were examined by cyclic voltammetry (Model: EA161 eDAQ). The electrolyte solution employed was 0.10 M tetrabutylammonium hexafluorophosphate (Bu₄NPF₆) in a freshly dried dichloromethane. The Ag/AgCl and Pt wire (0.5 mm in diameter) electrodes were utilized as reference and counter electrodes, respectively. The scan rate was at 100 mV/s.

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Absorption and Photoluminescence Spectroscopy

Absorption spectra of samples in solution state were obtained using a UV-VIS spectrometer (HP 8453, photodiode array type) in the wavelength range 190–1100 nm. Photoluminescence (PL) spectra of the solutions at room temperature were acquired on a Hitachi F-7000 FL Spectrophotometer.

Electroluminescence Measurement

The PLEDs had a structure of ITO/PEDOT: PSS (30 nm)/ POx-TPACz (10 nm)/Cz-DNA: PBD with Ir(III) complex (20, 30, 40 nm)/TAZ (15 nm)/4,7-diphenyl-1,10-phenanthroline (Bphen) (25 nm)/LiF (0.8 nm)/Al (500 nm) (see Fig. 4). An attempt at forming the emitting layer was then made by spin coating the solution (solvent: chloroform (45 wt %), monochlorobenzene (45 wt %) and butanol (10 wt %)) onto photocrosslinked POx-TPACz. For multi-layer devices, 5-(4-tertbuthylphenyl)-3-(4-biphenyl)-4-phenyl-1,2,4-triazole (TAZ) and 4,7-diphenyl-1,10-phenanthroline (Bphen) layer were vacuum-deposited onto the emitting polymer layer. Finally, LiF (0.8 nm)/Al (500 nm) electrodes were deposited onto the Bphen layer. Current density-voltage characteristics were measured with a Keithley 2400 sourcemeter. The brightness and electroluminescence (EL) spectra of the devices were measured with Spectra Colorimeter PR-650.

RESULTS AND DISCUSSION

Synthesis and Characterization

The synthesis of Cz-DNA is well described in our recent report.¹⁵ 12-(9*H*-carbazol-9-yl)-*N*,*N*,*N*-trimethyldodecan-1-aminium bromide was used to prepare the Cz-DNA homopolymer (see Fig. 1). To confirm the completeness of the reaction, inductively coupled plasma atomic emission spectrometry (ICP-AES) was performed to measure the concentration of sodium ions bound to phosphate anions. The concentration of sodium in natural Na-DNA was 56,770 ppm; after reaction with 12-(9H-carbazol-9-yl)-*N*,*N*,*N*-trimethyldodecan-1-aminium bromide, the concentration of sodium in Cz-DNA decreased to less than 15 ppm. Carbazoyl side chains were substituted into the phosphate anions at a rate of up to 99.9%. The resultant modified Cz-DNA is insoluble in water but is soluble



FIGURE 2 UV-Vis absorption (solid line) and PL spectra (dotted line) of Cz-DNA (a), PczSt (b), and $Ir(Cz-ppy)_3$ (c) in the solution state (chloroform:MCB:BuOH = 0.45:0.45:0.1 wt ratio).

in alcohol or mixed solvents. To investigate the absorption and emission behavior of $Ir(Cz-ppy)_3$ doped Cz-DNA, we prepared the control polymer which is a random copolymer bearing vinylcarbazole and styrene monomers by radical polymerization in the presence of azobisisobutyronitrile (AIBN). The carbazole concentration (~25 wt %) in PCzSt was made to be identical with that in Cz-DNA for eliminating the significant excimer emission.

 $Ir(Cz-ppy)_3$ was synthesized by following the method described in our previous report.¹⁴ For fabricating multilayered electrophosphorescence devices, we prepared a photocrosslinkable polymer POx-TPACz, which has good holetransporting properties (see Fig. 1). The resulting POx-TPACz was found to have good self-film-forming properties, and was easily soluble in various organic solvents such as chloroform, toluene, monochlorobenzene, and tetrahydrofuran. The detailed synthetic procedure was already reported elsewhere.¹⁶

It should be noted that the PEDOT:PSS layer in PLEDs is slightly soluble in the solvents (e.g., alcohols) for Cz-DNA.

Therefore, the solution (solvent: chloroform (45 wt %), monochlorobenzene (45 wt %), and butanol (10 wt %)) containing Cz-DNA and $Ir(Cz-ppy)_3$ cannot be deposited on PEDOT:PSS directly by spin coating. Deposition of a protective layer on PEDOT:PSS improves the interfacial contact between HTL and EML. As the HOMO level of POx-TPACz is close to that of PEDOT:PSS, hole-transport behavior was not affected. The role of photocrosslinked POx-TPACz should be emphasized as a hole-transporting interlayer in this study.

DSC measurement was performed at a heating (cooling) scan rate of 10 (-10) °C/min under nitrogen with the highest temperature limited to below the decomposition temperature. The POx-TPACz exhibited no distinct crystalline-isotropic transitions in the range of 25–220 °C but shows clear glass transition temperatures ($T_g^{onset} = 165$ °C). The cationic photocrosslinking reaction was performed in the vicinity of the T_g by illumination of UV light ($\lambda = 254$ nm, I = 20 mW/cm²).

Absorption and Photoluminescent Spectroscopy

The absorption and PL spectra of the solution samples of Cz-DNA, PCzSt, and $Ir(Cz-ppy)_3$ are shown in Figure 2. The absorption bands of the spectrum of Ir(Cz-ppy)₃ below 320 nm are assigned to be the intraligand π - π * transitions originating from the Ir(III) complex, while the absorption at around 340 nm is due to the carbazole moieties. The spectral shapes and characteristic bands of Cz-DNA and PCzSt are almost identical in absorption and PL spectra. As the carbazole units in PCzSt were diluted by styrene monomers, we could not see any evidence of intermolecular interaction, which is usually observed in the emission spectrum of poly(N-vinyl carbazole). In the low-energy region (from 350 to 500 nm), spin-allowed and spin-forbidden metal-to-ligand charge transfer (MLCT) transitions of the Ir(Cz-ppy)₃ were observed. The emission maximum of $Ir(Cz-ppy)_3$ in the solution state was observed at 520 nm. There is a good overlap between the PL spectrum of Cz-DNA and the MLCT absorption bands of the iridium complex. This overlap should enable efficient energy transfer from the excited state in the host to the MLCT band of the guest.



FIGURE 3 PL spectra of PCzSt (A) and Cz-DNA (B) doped with $Ir(Cz-ppy)_3$ dye; *sample: Thin films with an identical thickness (t: ~140 nm). *Inset C: Relative comparison of PL spectra of Cz-DNA (1) and PCzSt (2) doped with $Ir(Cz-ppy)_3$. The concentration of Ir(III) dye is 4 wt %.



FIGURE 4 Energy level alignment in the multilayered EL device. *Dotted line: triplet level of Cz-DNA. [Color figure can be viewed in the online issue, which is available at www. interscience.wiley.com.]

We prepared the thin films of Cz-DNA and PCzSt with the concentration of $Ir(Cz-ppy)_3$ (conc. 1, 2, and 4 wt %) by spin coating method. In Figure 3, the PL spectra of the thin films were illustrated.

As was expected, when increasing the concentration of $Ir(Cz-ppy)_3$ the emission intensity increases monotonically. Very intriguing phenomenon could be observed comparing two sets of spectra. At the same concentration of $Ir(Cz-ppy)_3$, Cz-DNA samples exhibited relatively high PL intensity at 515–518 nm. The inset Figure (C) displays the clear difference of green emission from two samples. The PL intensity of 4 wt % doped Cz-DNA exhibited about 10 times higher emission intensity than the sample of PCzSt.

The Ir(III) dye can exhibit significantly high affinity for association with nucleic acid along a significant change of electronic and photophysical properties upon DNA binding. It can be conjectured that the isolation effect of $Ir(Cz-ppy)_3$ is attributed to groove binding or intercalation through the base pairs. In addition, we measured the PL quantum yield (PLQY) of two carbazole-based polymers using 9,10-diphenylanthracene in solution states (*solvent: chloroform:ethanol = 9:1). From the experiment, we could determine the PLQYs of Cz-DNA and PCzSt to be around 0.09 and 0.04, respectively. Therefore, the energy transfer efficiency can be expected to be much higher yielding the enhancement of emission intensity from Ir(III) complex in Cz-DNA.

Electrochemical Analysis

Before fabricating the multilayered electrophosphorescence devices, we investigated the molecular electronic energy levels of each component. Electrochemical analysis was performed to determine the redox ionization potentials of the synthesized compounds. The oxidation and reduction potentials are closely related to the HOMO and LUMO levels of the analyzed compounds. Cyclic voltammograms were recorded in a solution sample, and the potentials were obtained relative to an internal ferrocene reference (Fc/ Fc⁺). Ir(Cz-ppy)₃ and POx-TPACz in dichloromethane have reversible oxidation peaks $(E_{ox}^{1/2})$ at 0.74 and 0.66 V, respectively. However, cyclic voltammograms of Cz-DNA could not be recorded because it was soluble only in co-solvents. We measured thin film of Cz-DNA that were spun-coat on the Pt plate ($E_{ox}^{onset} = 1.20$ V). The HOMO levels of Ir(Cz-ppy)₃, Cz-DNA, and POx-TPACz were determined to be approximately -5.14, -5.60, and -5.06 eV, respectively. To determine the LUMO levels, we combined the oxidation potential in CV with the optical energy band gap (E_{g}^{opt}) resulting from the absorption edge in the absorption spectrum (see Fig. 4).

Although Cz-DNA exhibited the potential capability of a triplet host material based on the results of absorption, PL spectroscopy and CV analysis in electrophosphorescence device, it should be noted that the carrier injection/transport under an applied electric field is strongly dependent of the electronic nature. Cz-DNA is expected to show the dielectric nature due to nonconjugated DNA backbone and long alkyl chain spacers. The poor electron injection to the DNA-hosted emitting layer was the problem in the early study.¹³ That results in significantly poor EL device performances. Nevertheless, we improved the device performances significantly by doping electron transporting molecules into Cz-DNA in this study. The electrophosphorescence device properties of PBD doped Cz-DNA based Ir(III) dye systems were illustrated in the following section.



FIGURE 5 Molecular structures of Cz-DNA, POx-TPACz, Ir(Cz-ppy)₃, PBD, Bphen, and TAZ. The device configuration of the electrophosphorescent PLED. *PBD: 2-(biphenyl-4yl)-5-(4-*tert*-butylphenyl)-1,3,4-oxadiazole, Bphen: 4,7-diphenyl-1,10-phenanthroline, TAZ: 3-(biphenyl-4-yl)-5-(4-*tert*-butylphenyl)-4-phenyl-4*H*-1,2,4-triazole. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

TABLE 1 Measured Parameters of Electrophosphorescent Devices

Device (Thickness)	Turn-on (V)	Max Luminance/cd m ⁻² (Corresponding <i>J</i>)	Max. Luminous Efficiency/cd A ⁻¹ (Corresponding <i>J</i>)	Max. Power Efficiency/Im W ⁻¹ (Corresponding <i>J</i>)	Max. EQE/η _{ext} (Corresponding <i>J</i>)	EL λ _{max} (nm)	CIE (<i>x, y</i>)
A (20 nm)	4	1135 (114.9), @ 11V	3.41 (3.5) @ 6.5V	1.64 (3.5) @ 6.5V	1.12 (3.5) @ 6.5V	522	0.35, 0.60
B (30 nm)	5	721 (77.1), @ 13V	3.27 (4.8) @ 8.5V	1.26 (3.4) @ 8V	1.05 (3.4) @ 8.5V	520	0.34, 0.60
C (40 nm)	4.5	606 (87.9), @ 13.5V	2.24 (9.3) @ 9V	0.78 (9.3) @ 9V	0.77 (9.3) @ 9V	526	0.46, 0.59
^a D (40 nm)	6	244 (69.3), @ 11.5V	0.74 (13.43) @ 9.5V	0.24 (13.43) @ 9.5V	0.26 (13.43) @ 9.5V	526	0.40, 0.55

^a The device fabricated without adding PBD; The thickness of emitting layer is in the parenthesis.

Electrophosphorescent Properties

The PLEDs had a structure of ITO/PEDOT: PSS (30 nm)/POx-TPACz (10 nm)/Cz-DNA:PBD with Ir(III) complex (20, 30, and 40 nm)/TAZ (15 nm)/Bphen (25 nm)/LiF (0.8 nm)/Al (500 nm) (see Fig. 5). Device A (t = 20 nm), B (t = 30 nm), and C (t = 40 nm) of thicknesses suitable for light emitting layers were fabricated. The emitting layer then was made by spin-coating the solution (solvent: chloroform (45 wt %), monochlorobenzene (45 wt %), and butanol (10 wt %)) onto PEDOT:PSS. The surface of PEDOT:PSS was damaged by butanol, which is essential for preparing a good quality Cz-DNA film.

Therefore, we deposited POx-TPACz on PEDOT:PSS. POx-TPACz layer was crosslinked under UV light irradiation ($\lambda_{max} = 254$ nm, intensity = 40 mW/cm² for 120 s at 150–180 °C) in the presence of 2 wt % cationic photoinitiator [4-[(2-hydroxyte-tradecyl)oxy]-phenyl]phenyliodonium hexafluoro antimonate.

The solution of emitting materials was spin-coated onto the thoroughly dried and photo-cured POx-TPACz layer by using the solution (conc: 1.5 wt %). The TAZ and Bphen layers were vacuum-deposited onto the emitting layer as holeblocking and electron-transporting molecules, respectively. Finally, LiF (0.8 nm)/Al (500 nm) electrodes were deposited onto the Bphen layer.

We first fabricated three different multilayered devices. Devices A, B, and C were fabricated with the thickness of the emitting layer by doping Ir(Cz-ppy)₃ (10 wt %) into a Cz-DNA:PBD (70:30 wt ratio) host. Device D was fabricated without doping PBD into Cz-DNA to compare the device performances. Performance data of the devices are listed in Table 1. Turn-on voltages for these devices are typical for Ir(III) complex-doped PLEDs and are in the range of 4.0-6.0 V. The maximum brightness of the LEDs was approximately 1135 cd/m^2 (at 114.9 mA/cm^2) for Device A, 721 cd/m² (at 77.1 mA/cm²) for Device B, and 606 cd/m² (at 87.9 mA/cm²) for Device C (see Table 1 and Fig. 6). The brightness of the abovementioned three Devices was much higher than that of Device D ($L_{max} = 244$ cd/m^2). Devices with thin emitting layers had high brightness; this is attributed to a more facile carrier transport and confinement in the emitting layer.¹⁸

The LUMO level of Cz-DNA is too shallow to inject the electron efficiently due to the dielectric nature of the DNA backbone. We can expect better electron injection into carbazole moieties through the added PBD molecules, whose LUMO level is approximately 2.3 eV. The EL emission spectra of the three devices are shown in Figure 7. The spectra of the three devices are similar to the PL solution spectra of the Ir(Cz-ppy)₃. This suggests that the same excited-state species is responsible for both PL and EL emissions, resulting from the triplet emission caused by the Ir(III) complex. No host emission was observed in the fabricated devices. This seems to indicate that energy transfer from the Cz-DNA:PBD host to the three Ir(III) complexes is quite efficient at the given dopant concentration.

Figure 8 shows the dependence of the luminous efficiency and external quantum efficiency on the current density for the three electrophosphorescent devices. The maximum luminous efficiencies of Devices A, B, and C were determined to be 3.41 cd/A ($\eta_{EQE} = 1.12\%$), 3.27 cd/A ($\eta_{EQE} = 1.05\%$), and 2.24 cd/A ($\eta_{\rm EOE} = 0.77\%$), respectively. The inset figure was obtained from the device prepared with Cz-DNA host without PBD. As expected in this case, the device efficiencies are much lower than those of PBD-doped Cz-DNA-based devices due to poor electron-injection and transport properties. The thicker the emitting layer, the lower is the efficiency. Note that a thick emitting layer limits the efficiency of exciton confinement and results in poor carrier transport. This is because the DNA host still bears the ionic species in the anchoring site of the carbazole moieties, so that the carriers can be trapped by cationic or anionic species.



FIGURE 6 Dependence of current density and luminance on the applied voltage Circle (Device A), square (Device B), triangle (Device C). Empty symbol (luminance), filled symbol (current density). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]



FIGURE 7 EL spectra of three devices with emitting layers of thicknesses (a) 20 nm, (b) 30 nm, and (c) 40 nm. [Color figure can be viewed in the online issue, which is available at www. interscience.wiley.com.]

CONCLUSIONS

We prepared an organic soluble DNA (Cz-DNA) bearing carbazole moieties in the side chain of DNA. We also investigated the phosphorescence behavior in $Ir(Cz-ppy)_3$ doped Cz-DNA system by comparing the emission behavior of PCzSt with $Ir(Cz-ppy)_3$. Very intriguing phenomenon could be observed by comparing two sets of PL spectra. At the same concentration of $Ir(Cz-ppy)_3$, Cz-DNA samples exhibited relatively high PL intensity at 515–518 nm.

Although Cz-DNA is expected to show dielectric nature due to a non-conjugated DNA backbone and long alkylene spacers, it was successfully employed as a triplet host material for electrophosphorescence devices via modification of its property by doping PBD. A new Cz-DNA-based electro-



FIGURE 8 Dependence of luminous efficiency and external quantum efficiency on the current density. Circle (Device A), triangle (Device B), square (Device C). *Filled symbol: Luminous efficiency, empty symbol: external quantum efficiency (EQE). *Inset: luminous efficiency and external quantum efficiency vs the current density of Device D. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

phosphorescent device with Ir(Cz-ppy)₃ and PBD exhibited much improved performance compared to the device only with Cz-DNA. Although the performance of a Cz-DNA-based PLED fabricated using the solution technique is much inferior to that of a device fabricated using a PVK host, Cz-DNA was firstly demonstrated to be used as a triplet host material for electrophosphorescence devices. Our study unambiguously describes a new Cz-DNA:PBD host for Ir(III) complex, which takes advantage of improved electron transport.

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