

含氟绿光铱配合物的设计、合成及其高效磷光电致发光器件

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摘要: 以 2-(3-(2',4'-二氟苯基)苯基)吡啶(Hdfbppy)为环金属 C^N 配体, 乙酰丙酮(Hacac)为辅助配体, 设计合成了一种绿色磷光铱配合物(Ir(dfbppy)₂(acac)); 研究了此配合物的光物理性质及其电致发光器件性能。室温下, 配合物 Ir(dfbppy)₂(acac)的二氯甲烷溶液的最大发射波长为 520 nm, 量子效率为 71%, 寿命为 381 ns。将此配合物掺杂在 4,4'-N,N'-二咔唑基二联苯(CBP)中, 作为发光层制备了有机发光二极管器件。结果显示, 该器件在 7.2 V 电压下呈现的最大亮度为 68 324 cd·m⁻², 最大电流效率约为 53 cd·A⁻¹, 最大功率效率为 37 lm·W⁻¹, 色坐标为(0.33, 0.62)。

关键词: 绿光材料; 铱配合物; 光电特性; 有机电致发光二极管; 磷光

中图分类号: O614.82⁵ 文献标识码: A 文章编号: 1001-4861(2015)05-1034-07

DOI: 10.11862/CJIC.2015.123

A Fluorine-Containing Phosphorescent Iridium(III) Complex for High-Efficiency Green Organic Light-Emitting Device

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Abstract: A green-light emitting iridium(III) complex Ir(dfbppy)₂(acac) with 2-(3-(2',4'-difluorophenyl)phenyl)pyridine (Hdfbppy) as C^N ligands and 2,4-pentanedione (Hacac) as an ancillary ligand was designed and synthesized for phosphorescent organic light emitting diodes, and its photophysical and electroluminescent properties were investigated. The title complex exhibits a peak emission at 520 nm, a high PL quantum yield of 71%, and a relatively short phosphorescence emission lifetime of 381 ns in CH₂Cl₂ at room temperature. An

收稿日期: 2014-12-22。收修改稿日期: 2015-03-12。

国家自然科学基金青年基金(No.21201104), 江苏自然科学基金面上项目(No.BK20141422), 江苏省有机电子与信息显示重点实验室提升项目(No.BM2012010), 江苏高校优势学科建设工程资助项目(No.YX03001), 教育部创新团队(长江学者和创新团队发展计划)(No.IRT1148), 南京邮电大学引进人才启动基金(No.NY213096)资助项目。

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organic light-emitting diode using this complex with 4,4'-*N,N'*-dicarbazolylbiphenyl (CBP) as the host shows a green color with CIE coordinates of (0.33, 0.62), accompanied by a rather excellent performance with a maximum luminance of $68\,324\text{ cd}\cdot\text{m}^{-2}$ at 7.2 V, the luminous and power efficiencies are $53\text{ cd}\cdot\text{A}^{-1}$ and $37\text{ lm}\cdot\text{W}^{-1}$, respectively.

Key words: green emitting materials; iridium(III) complex; optoelectronic performances; organic light emitting devices; phosphorescence

In the past two decades, organic light-emitting diodes (OLEDs) have been intensively investigated because of their strong potential for the next generation of display and solid state lighting applications with high color quality, low power consumption, etc^[1-6]. In particular, phosphorescent organic light emitting diodes (PHOLEDs) based on transition-metal complexes have undergone tremendous development because they exhibit excellent device performances^[7-18]. For example, the internal quantum efficiencies of PHOLEDs can reach as high as 100% in theory owing to the harvest of both singlet and triplet excitons for photon emission, which accelerates the mass production of OLEDs^[19-28]. However, further developments in highly efficient PHOLEDs are desirable, such as device lifetimes and efficiency roll-off. Therefore, it is of great importance to develop novel phosphorescent materials.

Cyclometalated iridium(III) complexes are the most widely used phosphorescent materials^[29-36]. The radiative transition from triplet exciton to singlet one can be promoted by the strong spin-orbit coupling associated with the heavy-metal atom iridium(III), leading to high phosphorescence quantum yield at room temperature from the triplet metal-to-ligand charge-transfer (³MLCT) states, relatively short triplet emission decay times and a broad range of emission colors^[37-41]. Particularly, bis- and tris-cyclometalated iridium(III) complexes based on C^N ligands of 2-phenylpyridine (ppy) and their derivatives have been widely used as one of the promising electroluminescent materials for the PHOLEDs technology^[42-43]. The HOMO of the cyclometalated iridium(III) complexes is determined by the 5*d* orbital of iridium(III) with the π orbitals of the 2-phenylpyridine ligand^[44]. Thus, by changing the structure of the 2-phenylpyridine ligand,

one can modulate the frontier orbital energy level and band gap of iridium(III) complexes in order to tune the carrier injection and mobility as well as emission colors.

Fluorine atom exhibits strong electron withdrawing effect on the π -system of ligand, which may decrease the LUMO energies of the phosphorescent metal complexes and enhance electron mobility. In addition, it can also provide steric protection around the metal, which will help to reduce concentration quenching of luminescence and then increase the emission intensity, meanwhile increase volatility to favor vapor deposition^[45-47]. The effect of fluorine atoms on the photophysics of homoleptic complexes and heteroleptic iridium complexes has been evaluated by various groups^[48-50]. The fluorine groups in different positions of the ligands show different effects on the photophysical properties. Hence, it is a good strategy to develop excellent phosphorescent materials by introducing fluorine atom in the ligand and comprehensive and deeper studies are still needed. Herein, we designed and synthesized an iridium(III) complex Ir(dfppy)₂(acac) with 2-(3-(2',4'-difluorophenyl)phenyl)pyridine (Hdfppy) as C^N ligands and 2,4-pentanedione (Hacac) as an ancillary ligand with high quantum efficiency. The photophysical properties were investigated in detail. A green-emitting PHOLED with high efficiency was realized using Ir(dfppy)₂(acac) as an emitting material.

1 Experimental

1.1 General

¹H NMR spectra were recorded on a Bruker Ultra Shield Plus 400 MHz NMR instruments in CDCl₃. The elemental analysis was performed using Vario Macro apparatus (Germany). Mass spectrum was acquired

with a Bruker autoflex MALDI-TOF/TOF mass spectrometer. FTIR spectrum was recorded by a Shimadzu IR Prestige-21 spectrometer. Melting point was determined on a Shimadzu DSC-60 spectrometer. The UV-Visible absorption spectrum was recorded on a Shimadzu UV-3600 UV-VIS-NIR spectrophotometer in dilute dichloromethane solution ($10^{-5} \text{ mol} \cdot \text{L}^{-1}$). Photoluminescent spectrum was obtained using an Edinburgh FLS-920 spectrofluorophotometer in dilute dichloromethane solution ($10^{-5} \text{ mol} \cdot \text{L}^{-1}$). The emission lifetime of the iridium(III) complex was performed with an Edinburgh FLS-920 spectrometer in degassed CH_2Cl_2 ($10^{-5} \text{ mol} \cdot \text{L}^{-1}$) with a hydrogen-filled excitation source. The quantum efficiency was measured with an integrating sphere in degassed CH_2Cl_2 ($10^{-5} \text{ mol} \cdot \text{L}^{-1}$) with an Edinburgh FLS-920 spectrometer.

1.2 Materials

All starting reagents were purchased from J&K Scientific, with the exception of $\text{IrCl}_3 \cdot 3\text{H}_2\text{O}$ which was purchased from Shanxi Kaida Chemical. All the reagents, unless otherwise stated, were used as received. All manipulations involving air-sensitive were performed under an atmosphere of dry nitrogen. The solvent THF was purified by routine procedures and distilled before use.

1.3 Synthesis of Ligand and Iridium(III) Complex $\text{Ir}(\text{dfbppy})_2(\text{acac})$

The synthetic route of $\text{Ir}(\text{dfbppy})_2(\text{acac})$ is shown in Scheme 1.

1.3.1 Synthesis of **1**

In a 100 mL reaction flask, a mixture of 1.00 g (4.24 mmol) 1,3-dibromobenzene, 1.08 g (4.24 mmol)

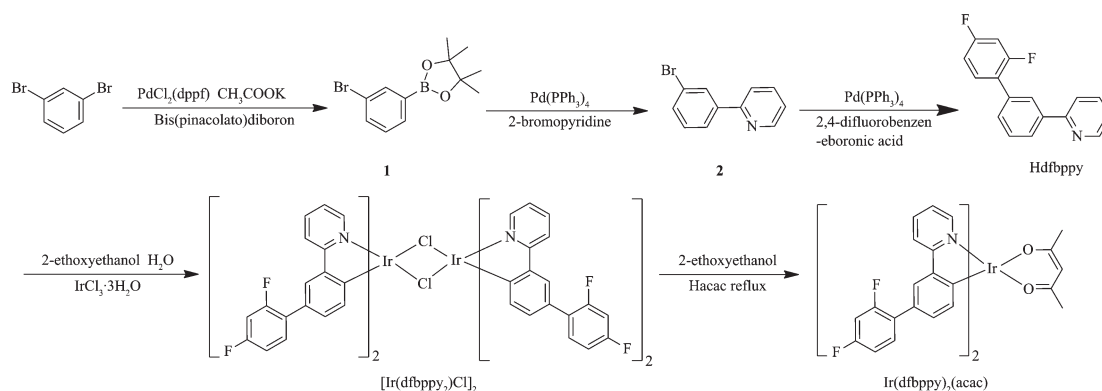
bis(pinacolato) diboron, 0.16 g (0.212 mmol) [1,1'-bis(diphenylphosphino) ferrocene] dichloropalladium(II), 600 mg potassium acetate and 20 mL 1,4-dioxane were refluxed under N_2 atmosphere for 10 h. The mixture was evaporated to dryness and the residue was purified by column chromatography on silica gel to get compound **1** 0.78 g. Yield: 65%. ^1H NMR (400 MHz, CDCl_3) δ =7.93 (s, 1H), 7.71 (d, J =7.60 Hz, 1H), 7.58 (ddd, J =8.00, 2.00, 1.20 Hz, 1H), 7.23 (t, J =7.60 Hz, 1H), 1.34 (s, 12H).

1.3.2 Synthesis of **2**

To a 100 mL reaction flask, 0.22 mL (2.26 mmol) 2-bromopyridine, 0.54 g (2.49 mmol) compound **1**, 0.05 g (0.04 mmol) tetrakis(triphenylphosphine)palladium and 5 mg sodium carbonate were added. 30 mL mixed solvent ($V_{\text{methylbenzene}}:V_{\text{ethanol}}:V_{\text{deionized water}}=3:1:1$) was injected after filling with N_2 . The mixture was refluxed for 6 h. The mixture was evaporated and the residue was purified by column chromatography on silica gel to get the colorless liquid product **2** 0.33 g. Yield: 62%. ^1H NMR (400 MHz, CDCl_3) δ =8.70 (d, J =4.80 Hz, 1H), 8.18 (t, J =1.20 Hz, 1H), 7.91 (d, J =7.60 Hz, 1H), 7.77 (dt, J =8.00, 1.50 Hz, 1H), 7.71 (d, J =8.00 Hz, 1H), 7.54 (dd, J =8.00, 0.80 Hz, 1H), 7.34 (t, J =8.00 Hz, 1H), 7.27 (t, J =6.20 Hz, 1H).

1.3.3 Synthesis of Hdfbppy

In a 100 mL reaction flask, 0.30 g (1.28 mmol) compound **2**, 0.22 g (1.41 mmol) 2,4-difluorophenylboronic acid, 0.03 g (0.03 mmol) tetrakis(triphenylphosphine)palladium and 2.5 mg sodium carbonate were added under N_2 atmosphere. The mixture was stirred and refluxed for 6 h after 30 mL mixed solvent



Scheme 1 Synthetic route to $\text{Ir}(\text{dfbppy})_2(\text{acac})$

($V_{\text{methylbenzene}}:V_{\text{ethanol}}:V_{\text{deionized water}}=3:1:1$) was injected. The mixture was evaporated to dryness and the residue was purified by column chromatography on silica gel to get white product Hdfbppy 0.23 g. Yield, 68%. ^1H NMR (400 MHz, CDCl_3) δ =8.72 (dt, J =4.80, 1.60 Hz, 1H), 8.14 (s, 1H), 8.01~7.98 (m, 1H), 7.77 (dd, J =4.80, 1.20 Hz, 2H), 7.56 (d, J =5.20 Hz, 2H), 7.49 (dt, J =6.40, 8.40 Hz, 1H), 7.29~7.23 (m, 1H), 7.00~6.91 (m, 2H).

1.3.4 Synthesis of $[\text{Ir}(\text{dfbppy})_2\text{Cl}]_2$

The cyclometalated iridium (III) chloro-bridged precursor $[\text{Ir}(\text{dfbppy})_2\text{Cl}]_2$ with Hdfbppy as cyclometalated ligands was synthesized using the same method as that reported by Nonoyama^[51].

1.3.5 Synthesis of $\text{Ir}(\text{dfbppy})_2(\text{acac})$

To a 50 mL reaction flask, 0.3 g (0.2 mmol) $[\text{Ir}(\text{dfbppy})_2\text{Cl}]_2$, 0.05 mL (0.5 mmol) acetylacetone, 30 mL 2-ethoxyethanol and 0.28 g (2 mmol) potassium carbonate were added. The mixture was refluxed for 6 h under N_2 atmosphere. The reaction mixture was then concentrated under vacuum, and the crude product was purified by column chromatography on silica gel to get faint yellow product $\text{Ir}(\text{dfbppy})_2(\text{acac})$ 0.16 g. Yield: 48%. Melting point: >300 °C. IR (KBr): 3 440, 1 576, 1 505, 1 479, 1 475, 1 396, 1 264, 1 140, 1 068, 965, 809, 777, 750, 587. ^1H NMR (400 MHz, CDCl_3) δ =8.55 (d, J =5.20 Hz, 2H), 7.92 (d, J =8.00 Hz, 2H), 7.77 (dt, J =8.40, 1.60 Hz, 2H), 7.69 (t, J =1.60 Hz, 2H), 7.33 (dt, J =8.80, 6.80 Hz, 2H), 7.20 (ddd, J =6.80, 6.00, 0.80 Hz, 2H), 6.89~6.81 (m, 6H), 6.38 (d, J =7.60 Hz, 2H), 5.25(s, 1H), 1.82 (s, 6H). MS (MALDI-TOF) $[m/z]$: 823.679 (Calcd. for 824.16). Anal. Calcd. (%) for $\text{C}_{39}\text{H}_{27}\text{F}_4\text{IrN}_2\text{O}_2$: C, 56.86; H, 3.30; N, 3.40; Found(%): C, 57.06; H, 3.31; N, 3.39.

1.4 OLED fabrication and measurement

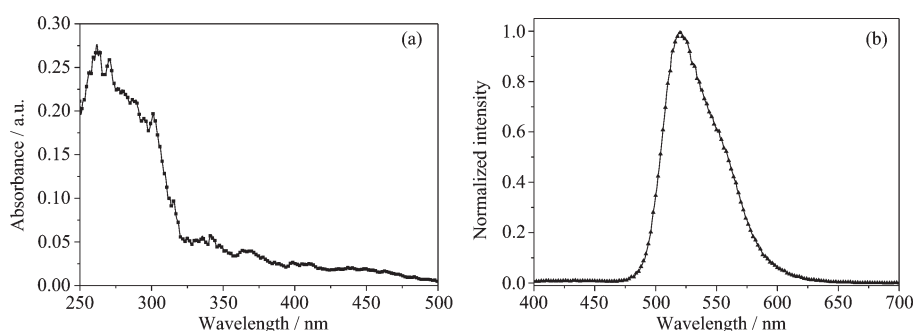
OLED was fabricated onto cleaned indium tin oxide (ITO) glass substrates with sheet resistance of 10~12 $\Omega \cdot \square^{-1}$. Prior to organic layer deposition, the ITO substrates were successively washed with acetone and isopropanol under ultrasonic for 10 min before loading into a deposition chamber. Using the vacuum evaporation method, onto a glass substrate pre-coated with ITO, molybdenum oxide (MoO_3) (3 nm) was

deposited to act as the hole injection layer (HIL), a hole-transporting 4,4',4''-tris (N-carbazolyl)triphenylamine (TCTA) layer was deposited subsequently, moreover, doping MoO_3 into TCTA layer was to reduce the energy barrier between MoO_3 and TCTA. A layer of 4,4'-N,N'-dicarbazolylbiphenyl (CBP) host was subsequently co-deposited with an emitter material $\text{Ir}(\text{dfbppy})_2(\text{acac})$ under high vacuum. Then, 4,7-diphenyl-1,10-phenanthroline (Bphen) was deposited as electron transporting layer (ETL). Finally, a cathode layer of LiF and Al were successively deposited on top of the ETL. The thickness of the corresponding layer was determined with a quartz crystal resonator (deposition rates: TCTA, CBP=0.1 $\text{nm} \cdot \text{s}^{-1}$, MoO_3 , LiF=0.01 $\text{nm} \cdot \text{s}^{-1}$, Al electrode=0.3 $\text{nm} \cdot \text{s}^{-1}$ and a pressure below $\sim 10^{-4}$ Pa). The EL spectrum was obtained with a PR-655 spectrometer. Current-voltage (J - V) and luminance-voltage (L - V) characteristics were recorded on a current-voltage source (Keithley 237) and a luminescence detector (PR-655). All EL measurements were carried out in air at room temperature.

2 Results and discussion

Scheme 1 outlines the structure and synthetic process of the iridium (III) complex. The iridium (III) complex was obtained by cyclometalation of the ligand Hdfbppy with $\text{IrCl}_3 \cdot 3\text{H}_2\text{O}$ and subsequent reaction with Hacac.

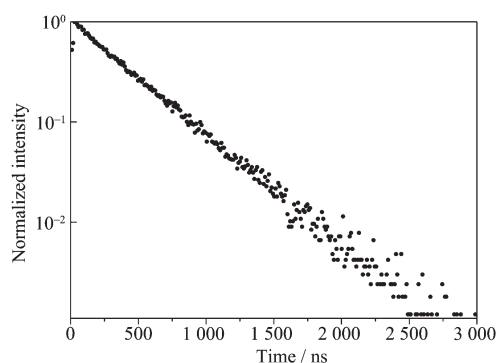
Fig.1 shows the UV-Vis absorption and PL spectra of this new phosphor in dilute dichloromethane solution ($10^{-5} \text{ mol} \cdot \text{L}^{-1}$). The absorption bands below 325 nm are assigned to the spin-allowed $^1\pi-\pi^*$ transitions of the cyclometallated ligands, and the bands above 325 nm are assigned to the spin-allowed $^1\text{MLCT}$ and spin-forbidden $^3\text{MLCT}$ transitions. Highly intensive green luminescence is observed for complex $\text{Ir}(\text{dfbppy})_2(\text{acac})$ with λ_{max} located at 520 nm. Table 1 shows the optical properties of $\text{Ir}(\text{dfbppy})_2(\text{acac})$. We used an Edinburgh FLS-920 spectrofluorophotometer to measure quantum efficiency and emission lifetime of $\text{Ir}(\text{dfbppy})_2(\text{acac})$ in CH_2Cl_2 at room temperature. The complex shows high PL quantum yield of 71% and a phosphorescence emission lifetime of 381 ns in

Fig.1 (a) Absorption and (b) emission spectra of Ir(dfppy)₂(acac) in CH₂Cl₂ solutionTable 1 Optical properties of Ir(III) complexes Ir(dfppy)₂(acac)

Complex	λ_{Abs} / nm	λ_{Em}^a / nm	ϕ^b / %	Lifetime ^c / ns
Ir(dfppy) ₂ (acac)	261, 301, 342	520	71	381

Notes: ^a Measured in CH₂Cl₂ at room temperature with a concentration of 10⁻⁵ mol·L⁻¹; ^b Measured with an integrating sphere in degassed CH₂Cl₂, λ_{ex} =365 nm; ^c Measured in degassed CH₂Cl₂.

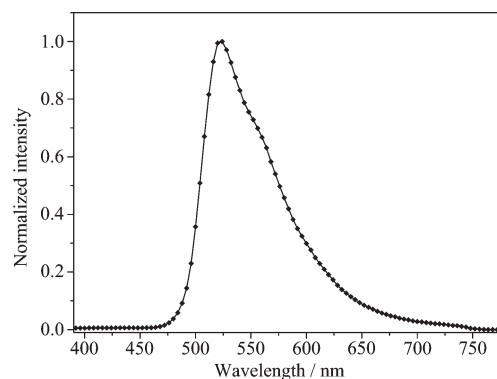
degassed CH₂Cl₂. The high quantum efficiency and relatively short triplet lifetime suggest that the resulting OLED device based on Ir(dfppy)₂(acac) should have highly efficient electroluminescent property. The lifetime decay curve is shown in Fig.2. The lifetime distribution with a single-exponential decay can be observed.

Fig.2 PL decay curve of Ir(dfppy)₂(acac) in CH₂Cl₂ at room temperature

In order to explore the electroluminescent properties of complex Ir(dfppy)₂(acac), an OLED device was fabricated with the configuration by employing 6% Ir(dfppy)₂(acac) as a dopant and CBP as the host in the emitting layer, and the whole configuration of device was ITO/MoO₃ (3 nm)/TCTA: MoO₃(25 nm, 15%)/TCTA(10 nm)/CBP: 6% Ir(30 nm)/Bphen(30 nm)/LiF(1 nm)/Al(200 nm).

Fig.3 shows the EL spectrum of the device at a fixed voltage of 7.2 V. The device exhibits green

electroluminescence with a peak emission at 522 nm, and the CIE coordinate value is (0.33, 0.62). The EL spectrum of the device is almost coincident with the PL spectrum of complex Ir(dfppy)₂(acac) in solution, indicating that there is little intermolecular aggregation in the film of this complex. The current density-voltage-luminance (*J-V-L*), current efficiency and power efficiency with respect to the current density of the device are shown in Fig.4. The maximum luminance of the device is 68 324 cd·m⁻² at 7.2 V. The turn-on voltage for device is 3.3 V, which indicates that the device has effective carrier injection. The maximum current efficiency and power efficiency of the device are 53 cd·A⁻¹ and 37 lm·W⁻¹, respectively. This device shows comparable device performance compared with some reported high-efficiency green PHOLEDs previously in a device of

Fig.3 Electroluminescence (EL) spectrum of the device using Ir(dfppy)₂(acac) as an emitter

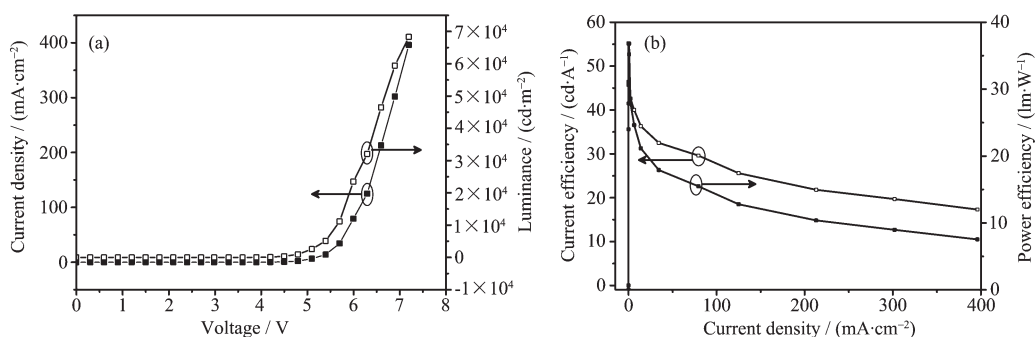


Fig.4 (a) J - V - L and (b) current efficiency and power efficiency versus current density relationship of the device, respectively

similar multi-layer structure^[50,52-54]. This excellent performance may be due to the phosphorescent complex Ir(dfppy)₂(acac) as the emitter which can help to improve the EL performances of OLEDs, demonstrating that the complex Ir(dfppy)₂(acac) shows great potential for application as the green emitting material in OLEDs.

3 Conclusions

A green-light emitting phosphorescent complex Ir(dfppy)₂(acac) containing fluorine atoms on the C^N ligand was synthesized, and its electroluminescent properties were investigated. An OLED using this green dopant with CBP as the host exhibits a maximum luminance of 68 324 cd·m⁻² at 7.2 V, that is greater than many reported values. The luminous and power efficiencies are up to 53 cd·A⁻¹ and 37 lm·W⁻¹, respectively, with CIE coordinates of (0.33, 0.62). These results demonstrate that the phosphorescent iridium(III) complexes with 2-phenylpyridine derivatives containing fluorine atoms as the cyclometalated ligands exhibit potential application as the green-emitting material in OLEDs.

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