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Acetylated Phenylpyridine Derived Iridium (III) Complexes for Orange–Red Electrophosphorescent Light-Emitting Diodes

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The photophysical characteristics of $(Actpy)_2Ir(acac)$ and $(Acpsf)_2Ir(acac)$ were investigated to determine their suitability as candidates for red light-emitting materials for organic light emitting diodes (OLEDs) [where Actpy = 3-acetyl-6-(4-tert-butylphenyl)pyridine, acac = acetoylacetonate, and Acpsf = 3-acetyl-6-(9,9'-spirobi[fluorene]-2-yl)pyridine]. To explore the electroluminescent properties of these materials, multilayered OLEDs were fabricated the device structure of ITO/NPB/CBP: 8% Ir(III) complexes/BCP/Alq₃/Liq/Al. Particularly, device 1 using complex (Actpy)₂Ir(acac) as a dopant material furnished a maximum luminance of 41,800 cdm⁻² at 15 V, and luminous efficiency and power efficiency of 5.70 cdA⁻¹ and 2.70 lmW⁻¹ at 20 mAcm⁻², respectively. Additionally, this device showed orange-red emission with CIE coordinates of (0.57, 0.42) at 10.0 V.

Keywords Acetylated Ir(III) complexes; phosphorescent OLEDs; spirobifluorene; *tert*-butyl group

1. Introduction

Since Forrest and Thompson's group reported the first example of electrophosphorescence at room temperature [1], PHOLED materials and devices have been intensively studied because of high external quantum efficiencies (EQEs) in their electroluminescence devices [2,3]. The $Ir(ppy)_3$ derivatives have shown a number of advantages such as easy tuning emission energy by functionalized ppy ligand with electron donating and withdrawing moieties. In this way, a variety of red Ir(III) complexes have been developed as electrophosphorescent emitter [4–6]. Among those are Ir(III) complexes based on the carbonylated phenyl–pyridine ligands [7–9]. For example, we have reported orange–red emitters based on carbonylated phenyl–pyridine derivative-Ir(III) complexes, (Acppy)₂Ir(acac) in organic

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light emitting diodes (OLEDs) with high luminous efficiency and brightness were reported [10]. Also, in our recent work, a highly efficient red OLED was obtained by adopting 5-benzoyl-2-phenylpyridine derived Ir(III) complex with *tert*-butyl moiety in benzoyl unit [11].

In this article, as a continuing effort to develope the efficient phosphorescent iridium complexes based on the carbonylated phenylpyridine ligand for OLED, two orange-red phosphorescent Ir(III) complexes based on acetylated phenylpyridine ligand, (Actpy)₂Ir(acac) and (Acpsf)₂Ir(acac) are synthesized and their electrophosphrescent properties are investigated. Also, in (Actpy)₂Ir(acac) and (Acpsf)₂Ir(acac), the different bulky groups of electron-donating *tert*-butyl and spirobifluorene were introduced in the ligands of Ir(III) complexes to study the substituent effect on the electroluminescent properties. As will be seen in below, the electroluminescent properties of devices using (Actpy)₂Ir(acac) and (Acpsf)₂Ir(acac) as dopants are highly sensitive to the structural features of the orange-red emitters.

2. Experimental Details

5-Acetyl-2-bromopyridine, 4-tert-buthylphenylboronic acid, spirofluorenyl-2-boronic acid, and acetylacetone were used as received from Aldroch or TCI. All reactions were performed under nitrogen. Solvents were carefully dried and distilled from appropriate drying agents prior to use. Commercially, available reagents were used without further purification unless otherwise stated. ¹H- and ¹³C-NMR spectra were recorded using a Varian (unity Inova 300Nb or 500NB) spectrometer at 300 or 500 MHz. FT-IR spectra were recorded using a VERTEX 70 spectrometer. Low- and high-resolution mass spectra were recorded using either a Jeol JMS-AX505WA spectrometer in FAB mode or a Jeol JMS-600 spectrometer in EI mode. The UV-vis absorption measurements of these blue-emitting materials in dichloromethane (10^{-5} M) were acquired with a Sinco S-3100 in a quartz cuvette (1.0 cm path). The photoluminescence (PL) spectra were measured on an Amincobrowman series 2 luminescence spectrometer. The phosphorescence quantum yields were determined in CH₂Cl₂ solutions at 293 K against Ir(ppy)₃ as a reference ($\Phi = 0.40$) [12]. The HOMO (highest occupied molecular orbital) energy levels were determined using a low energy photoelectron spectrometer (Riken-Keiki, AC-2). The energy band gaps were determined from the intersection of the absorption and PL spectra. The lowest unoccupied molecular orbital (LUMO) energy levels were calculated by subtracting the corresponding optical band gap energies from the HOMO energy values.

2.1. Synthesis and Characterization

2.1.1. General Procedure for the Synthesis of Ir(III) Complexes. Actppy (0.88 g, 4.46 mmol) was dissolved in 2-ethoxyethanol (18.0 mL) in a 50 mL round-bottom flask. IrCl₃·3H₂O (0.60 g, 2.01 mmol) and water (6.0 mL) were then added to the flask. The mixture was stirred under nitrogen at 120°C for 24 h and cooled to room temperature. The precipitate formed in the mixture was collected and washed with methanol, hexane, and dried in vacuum to give the corresponding cyclometalated Ir(III)- μ -chloro-bridge dimer. In a 50 mL flask, the dimer complex, acetylacetone (0.31 mL, 3.02 mmol) and Na₂CO₃ (0.64 mg, 6.03 mmol) were mixed with 2-ethoxyethanol (20 mL) and mixture was heated at 100°C for 6h. After cooling at room temperature, the solid precipitate was collected by filtration, washed with ethanol and hexane. The residue was dissolved in dichloromethane

and the solid was filtered off. The solution was concentrated in vacuo and the residue was purified on a silica gel column using dichloromethane and methanol as eluent. The product was obtained after recrystallization from dichloromethane/ethanol. Similar procedures were also employed for the synthesis of other Ir(III) complexes. The yields and spectral data of all Ir(III) complexes are as follows.

Complex 1 (Actppy)₂Ir(acac) (32% Yield). ¹H NMR (300 MHz, CDCl₃): δ ppm 9.09 (d, J = 2.0 Hz, 2H), 8.24 (dd, J = 2.0, 8.6 Hz, 2H), 7.87 (d, J = 8.5 Hz, 2H), 7.54 (d, J = 8.3 Hz, 2H), 6.89 (dd, J = 1.9, 8.3 Hz, 2H), 6.23 (d, J = 1.9 Hz, 2H), 5.27 (s, 1H), 2.60 (s, 6H), 1.85 (s, 6H), 1.03 (s, 18H). ¹³C NMR (125 MHz, CD₂Cl₂): δ ppm 194.3, 185.5, 172.4, 153.6, 149.9, 149.4, 141.5, 136.8, 130.4, 129.6, 125.7, 119.5, 118.0, 101.1, 34.6, 30.9, 30.8, 28.4, 26.4. IR (KBr): $\nu = 2959$, 2866, 1687, 1584, 1517, 1480, 1454, 1387, 1358, 1265, 1113, 1061, 815, 756 cm⁻¹. FAB-MS (*m*/*z*): 796 [M⁺]. HRMS-FAB⁺ calcd for C₃₉H₄₃IrN₂O₄: 796.2855, found: 796.2852.

Complex 2 (Acpsf)₂Ir(acac) (44% Yield). ¹H NMR (300 MHz, CDCl₃): δ ppm 9.11 (d, J = 2.0 Hz, 2H), 8.16 (dd, J = 2.0, 8.3 Hz, 2H), 7.84 (d, J = 7.7 Hz, 4H), 7.68 (d, J = 8.7 Hz, 2H), 7.38–7.31 (m, 6H), 7.17 (td, J = 1.0, 7.5 Hz, 2H), 7.13–7.02 (m, 6H), 6.98 (td, J = 1.0, 7.5 Hz, 2H), 6.79 (d, J = 7.6 Hz, 2H), 6.70 (s, 2H), 6.65 (d, 7.6 Hz, 2H), 6.59 (d, J = 7.6 Hz, 2H), 5.32 (s, 1H), 2.64 (s, 6H), 1.87 (s, 6H). ¹³C NMR (125 MHz, CD₂Cl₂): δ ppm 194.4, 185.6, 171.7, 150.1, 149.9, 149.4, 149.3, 149.1, 144.2, 143.9, 142.4, 142.0, 141.9, 141.2, 136.8, 130.6, 128.6, 128.2, 128.0, 127.9, 127.7, 125.4, 124.3, 123.9, 123.8, 121.1, 121.0, 120.4, 120.3, 118.8, 101.1, 65.8, 28.5, 26.5. IR (KBr): $\nu = 3057$, 1688, 1591, 1518, 1488, 1439, 1396, 955, 754, 733 cm⁻¹. HRMS-FAB⁺ calcd for C₆₉H₄₇IrN₂O₄: 1160.3165, found: 1160.3167.

2.1.2. Synthesis of Actppy. A mixture of 5-acetly-2-bromopyridine (0.95 g, 4.75 mmol), 4-*tert*-butylphenylbronic acid (0.93 g, 5.23 mmol), tetrakis(triphenylphosphine)palladium (0.22 g, 0.19 mmol), toluene (20 mL), ethanol (10 mL), and 2M Na₂CO₃ (10 mL) was refluxed at 120°C for 5 h. After reaction mixture was cooled at room temperature, it was poured into water and extracted with ethyl acetate. The combined organic layer was dried with anhydrous MgSO₄ and evaporated to dryness. The crude product was further purified by silica gel column chromatography. The product was a white solid with an isolated yield of 74% (0.89 g). ¹H NMR (300 MHz, CDCl₃): δ ppm 9.22 (dd, *J* = 0.8, 2.3 Hz, 1H), 8.28 (dd, *J* = 2.3, 8.4 Hz, 1H), 8.02 (d, *J* = 8.4 Hz, 2H), 7.83 (dd, *J* = 0.8, 8.4 Hz, 1H), 7.53 (d, *J* = 8.4 Hz, 2H), 2.66 (s, 3H), 1.37 (s, 9H). ¹³C NMR (125 MHz, CDCl₃): δ ppm 196.7, 161.2, 153.8, 150.4, 136.5, 135.6, 130.6, 127.4, 126.2, 120.0, 35.1, 31.5, 26.9. IR (KBr): ν = 2961, 2868, 1680, 1458, 1414, 1384, 1359, 1117, 1019, 953, 830, 765, 735 cm⁻¹. HRMS-EI⁺ calcd for C₁₇H₁₉ON: 253.1467, found: 253.1466.

2.1.3. Synthesis of Acpsf. The reaction described for the synthesis of Actppy was carried out starting with 5-acetly-2-bromopyridine (0.60 g, 3.00 mmol) and spirofluorenyl-2-boronic acid (1.19 g, 3.30 mmol). The product was a white solid with an isolated yield of 87% (1.14 g). ¹H NMR (300 MHz, CDCl₃): δ ppm 9.10 (d, J = 2.1 Hz, 1H), 8.15 (td, J = 7.8, 8.6 Hz, 2H), 7.97 (d, J = 7.8 Hz, 1H), 7.91–7.86 (m, 3H), 7.64 (d, J = 8.7 Hz, 1H), 7.42–7.35 (m, 4H), 7.14–7.08 (m, 3H), 6.78–6.72 (m, 3H), 2.58 (s, 3H). ¹³C NMR (125 MHz, CDCl₃): δ ppm 196.6, 160.9, 150.2, 149.9, 149.8, 148.6, 144.2, 142.1, 141.1, 137.9, 136.4, 130.6, 128.8, 128.2, 128.1, 127.8, 124.4, 124.3, 123.2, 120.8, 120.7, 120.4, 66.3, 26.9. IR (KBr): $\nu = 3061$, 1683, 1585, 1447, 1375, 1273, 909, 755, 730 cm⁻¹. HRMS-EI⁺ calcd for C₃₂H₂₁ON: 435.1623, found: 435.1623.

2.2. Fabrication and Characterization of Device

OLED fabrication of the indium-tin-oxide (ITO) thin films coated on glass substrates were used, which were 12 Ω sq⁻¹ (emitting area was 3 mm² × 3 mm²) of the sheet resistivity and a 1000 Å of thickness. The ITO-coated glass was cleaned in an ultrasonic bath by the following sequence: acetone, methyl alcohol, distilled water, followed by storage in isopropyl alcohol for 20 min and drying with a N_2 gas gun. The substrates were treated with O_2 plasma argon environment. Organic layers were deposited by thermal evaporation from resistively heated alumina crucibles onto the substrate at a rate of 1.0 Å s⁻¹. All organic materials and metal were deposited under high vacuum (5.0×10^{-7} Torr). The OLEDs were fabricated in the following sequence: ITO/bis[N-(1-(naphthyl)-Nphenyl-amino]biphenyl (NPB) (65 nm)/4,40,N,N0-dicarbazolebiphenyl (CBP): 8% Ir(III) complexes/bathocuproine (BCP) (10 nm)/tris-(8-hydroxyquinoline) aluminum (Alq₃) (25 nm)/lithium quinolate (Liq) (2 nm)/Al. The current density (J), luminance (L), luminous efficiency (LE), and the CIE chromaticity coordinates of the OLEDs were measured with Keithly 2400, Chroma meter CS-1000A. Electroluminance was measured using a Roper Scientific Pro 300i.

3. Result and Discussion

Scheme 1 outlines the structures and synthetic processes of the Ir(III) complexes (Actppy)₂Ir(acac) and (Acpsf)₂Ir(acac). The Ir(III) complexes were obtained by cyclometalation of the ligands with $IrCl_3$ and subsequent reaction with acetylacetone [13]. After conventional purification, such as column chromatography and recrystallization, these synthesized orange-red emitting materials (Actppy), Ir(acac) and (Acpsf), Ir(acac) were purified further by train sublimation at reduced pressure, below 10^{-3} torr, and fully characterized with ¹H- and ¹³C-NMR, infrared (IR), and low- and high-resolution mass spectrometry.

The UV-vis absorption and PL spectra of (Actppy)₂Ir(acac) and (Acpsf)₂Ir(acac) were shown in Fig. 1, and their data summarized in Table 1. The absorption spectra of Ir(III) complexes (Actppy)₂Ir(acac) and (Acpsf)₂Ir(acac) below 360 nm can assigned to the spin-allowed ${}^{1}\pi$ - π^{*} transition involving Actppy and Acpsf ligands, and the band around 400 nm to a spin-allowed metal charge transfer (¹MLCT) band. These orange-red phosphors show outstanding strong absorption band 450–550 nm regions, which can be assigned to spin-forbidden (³MLCT) band. The maximum emission wavelength (λ_{max}) of (Actppy)₂Ir(acac) and (Acpsf)₂Ir(acac) appeared at 600, and 612 nm, respectively. Especially, compared with the PL spectra of (Acppy)₂Ir(acac), the maximum emission wavelength of complex (Acpsf)₂Ir(acac) with spirobifluornyl moiety in the phenyl unit was red-shifted (ca. 9 nm), respectively, due to increasing of π -conjugation length of ligand. The emission quantum yields of complexes (Actppy)₂Ir(acac) and (Acpsf)₂Ir(acac) were 0.42 and 0.19, respectively, as determined by using $Ir(ppy)_3$ (0.40) used as the reference [12]. Interestingly, compound (Actppy)₂Ir(acac), bearing bulky *tert*-butyl groups in the phenyl unit, had quantum yields higher than that of Ir(ppy)₃. The HOMO and LUMO energy levels of complexes (Actppy)₂Ir(acac) and (Acpsf)₂Ir(acac) are shown in Table 1. The respective HOMO energy levels of $(Actppy)_2 Ir(acac)$ and $(Acpsf)_2 Ir(acac)$ were measured at -5.51and -5.54 eV by a low-energy photoelectron spectrometer (Riken-Keiki AC-2). The optical energy band gaps (E_g) of $(Actppy)_2 Ir(acac)$ and $(Acpsf)_2 Ir(acac)$ were 2.24 and 2.17 eV, as determined from the absorption spectra and emission spectra. The LUMO energy levels calculated by subtraction of the optical energy band gap from the HOMO levels were



Scheme 1. Synthetic route to new Ir(III) complexes (Actppy)₂Ir(acac) and (Acpsf)₂Ir(acac).

-3.27 eV and -3.37 eV, respectively. In case of $(Acpsf)_2Ir(acac)$ with spirobifluornyl group, the HOMO and LUMO energy levels lower than those of $(Actppy)_2Ir(acac)$. The energy diagrams for complexes $(Actppy)_2Ir(acac)$ and $(Acpsf)_2Ir(acac)$, as well as for devices using other materials, are shown in Fig. 2.



Figure 1. (a) The absorption, and (b) emission spectra of complexes (Actppy)₂Ir(acac) and (Acpsf)₂Ir(acac).

Complexes	Absorption (293K) $\lambda_{abs}{}^a$ (nm)	λ_{em}^{a} (nm)	HOMO/LUMO (eV)	Eg ^b (eV)	Q.Y ^c
(Actppy) ₂ Ir(acac)	289(3.37), 322(3.78), 513(0.47)	600	-5.51/-3.27	2.24	0.42
(Acpsf) ₂ Ir(acac)	300(4.36), 311(4.25), 362(4.81), 530(0.67)	612	-5.54/-3.37	2.17	0.19

 Table 1. Photophysical properties

^aMeasured in CH₂Cl₂ at a concentration of 10^{-5} M and molar extinction coefficient (ϵ) values ($10^4 \text{ M}^{-1} \text{ cm}^{-1}$) are shown in parentheses.

^bBand-gap energies estimated from the red edge of absorption spectra and emission spectra.

^cDegassed CH₂Cl₂ relative to *fac*-[Ir(ppy)₃] ($\Phi_p = 0.40$), $\lambda_{ex} = 400$ nm.

The electroluminescent properties of complexes $(Actppy)_2Ir(acac)$ and (Acpsf)₂Ir(acac) were explored by complexes as dopant materials in the fabrication of multilayered OLEDs. The device configuration was ITO/NPB (65 nm)/CBP: 8% Ir(III) complexes/BCP (10 nm)/Alq₃ (25 nm)/Liq (2 nm)/Al (100 nm). The EL characteristics of devices 1 and 2 are summarized in Table 2. Figure 3 shows the EL spectra of the devices 1 and 2 exhibited orange-red emissions with maximum peaks of 578 nm and 604 nm, respectively. The CIE coordinates versus voltage of the devices based on complexes (Actppy)₂Ir(acac): (0.57, 0.42), and (Acpsf)₂Ir(acac): (0.57, 0.37) are presented in Fig. 4. This trend of the CIE coordinates of devices is well compatible to the PL spectra of Ir(III) complexes (Actppy)₂Ir(acac) and (Acpsf)₂Ir(acac). Compared to device 1 using (Actppy)₂Ir(acac), the bathochromic shifted CIE coordinate of device 2 using $(Acpsf)_2 Ir(acac)$ can be attributed mainly to the increasing of π -conjugation length of dopant (Acpsf)₂Ir(acac). The current density-voltage-luminance (J-V-L) characteristics and luminous efficiencies of devices 1 and 2 are shown in Fig. 4(a) and (b). The luminance



Figure 2. Energy diagram of devices 1 and 2.

Device	Dopant (%)	λ _{max} ^{EL} (nm)	$\frac{V_{on}{}^{a}}{(cd\ m^{-2})}$	L^{b} at 15V (cd m ⁻²)	$\frac{\text{LE}^{\text{c, d}}}{(\text{cd } \text{A}^{-1})}$	$\begin{array}{c} PE^{d}\\ (Im \ W^{-1})\end{array}$	CIE ^e (x, y)
1	(Actppy) ₂ Ir(acac) (8%)	578	4.0	41,800	6.88(4 <i>V</i>)/5.70	2.7	(0.57, 0.42)
2	(Acpsf) ₂ Ir(acac) (8%)	604	3.6	17,600	6.06(5.5 <i>V</i>)/5.83	2.5	(0.57, 0.37)

Table 2. EL properties of devices

^aTurn-on voltage at 1 cd m⁻².

^bMaximum luminance.

^cMaximum values. Values in parentheses are the voltages at which the maximum values were obtained.

 d At 20 mA cm⁻².

eCommission Internationale d'Énclairage (CIE) coordinates at a 10 V.



Figure 3. EL spectra of devices 1 and 2.



Figure 4. (a) J-V-L curve, and (b) luminous efficiencies of OLED the devices 1 and 2.

were 41,800 cd m⁻² and 17,600 cd m⁻² at 15 V, and the turn-on voltages were 4.0 V and 3.6 V for devices **1** using (**Actppy**)₂**Ir**(**acac**) and **2** using (**Acpsf**)₂**Ir**(**acac**), respectively. The power efficiencies of devices **1** and **2** were 2.7 lm W⁻¹ and 2.5 lm W⁻¹ at 20 mA cm⁻², and the luminous efficiencies of devices **1** and **2** were 5.70 cd A⁻¹ and 5.83 cd A⁻¹ at 20 mAcm⁻².

Interestingly, compared with device 1 using $(Actppy)_2Ir(acac)$, device 2 using $(Acpsf)_2Ir(acac)$ exhibits, similarly in EL performance, even if $(Acpsf)_2Ir(acac)$ ($\Phi = 0.19$) has quantum yields lower than that of $(Actppy)_2Ir(acac)$ ($\Phi = 0.42$). This observation imply that spirobifluorene group of iridium complex $(Acpsf)_2Ir(acac)$ leads to the suitable energy transfer from host material in device, which improves the EL efficiencies of device using $(Acpsf)_2Ir(acac)$. Although color purity of a red OLED using $(Actppy)_2Ir(acac)$ and $(Acpsf)_2Ir(acac)$ are inadequate for full-color displays, $(Actppy)_2Ir(acac)$ and $(Acpsf)_2Ir(acac)$ could be applied to fabricate the efficient white organic light-emitting diodes (WOLEDs) with a combination of suitable sky blue emitters [14].

4. Conclusion

A series of orange–red phosphorescent Ir(III) complexes based on acetylated pheyl pyridine derivatives was synthesized by cyclometallation of the ligands with IrCl₃ and subsequent reaction with acetylacetone. An OLED using the orange–red dopant (**Actppy**)₂**Ir(acac**) with CBP as the host exhibited the maximum luminance of 41800 cd m⁻² at 15 V. The luminous and power efficiencies were 5.70 cd A⁻¹ and 2.7 lm W⁻¹ at 20 mA cm⁻², respectively. The CIE*x*,*y*-coordinates of (0.57, 0.42) at 10 V. Also, an orange–red OLED using dopant (**Acpsf**)₂**Ir(acac**) with the CIE coordinate (0.57, 0.37) exhibited the maximum luminance of 17,600 cd m⁻² at 9 V. The luminous and power efficiencies were 5.80 cd A⁻¹ and 2.5 lm W⁻¹ at 20 mA cm⁻², respectively. These results indicated that the electrical properties of the OLED devices were very sensitive to the structure of dopant in the emitting layer.

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