

Supporting Information

Homoleptic Facial Ir(III) Complexes via Facile Synthesis for High-Efficiency and Low-Roll-Off Near-Infrared Organic Light Emitting Diodes over 750 nm

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KEYWORDS: near-infrared emission, iridium complexes, homoleptic, organic light emitting diodes, density functional calculations.

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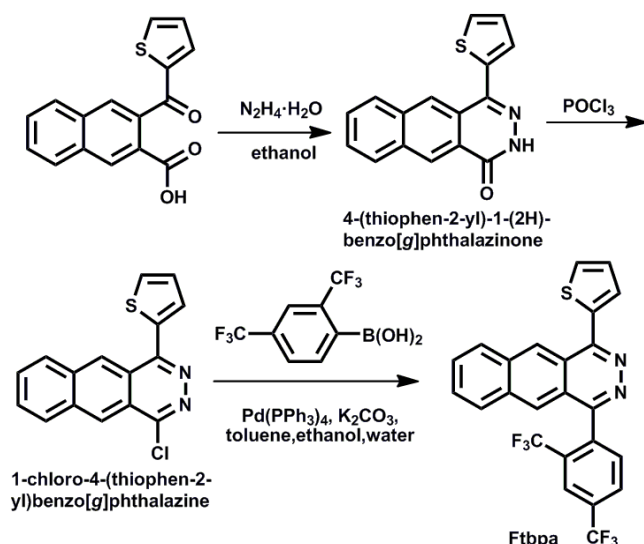
Experimental Section

1. Materials and general information:

General: All commercially available reagents and chemicals were used without further purification. All reactions involving air-sensitive reagents were carried out under an atmosphere of nitrogen. ^1H NMR spectra were measured on a JEOLAL-600 MHz spectrometer at ambient temperature. High resolution mass spectra were recorded using a Thermo-Electron Corporation Finnigan LTQ mass spectrometer (ESI-MS) and LCMS-IT/TOF (HRMS). The laser desorption ionization time-of flight mass spectrometry (LDI-TOF-MS) data were obtained using a Shimadzu AXIMA Performance MALDI-TOF instrument in both positive and negative detection modes with an applied voltage of 25 kV between the target and the aperture of the time-of-flight analyzer. Elemental analyses were performed on a flash EA 1112 spectrometer. High performance liquid chromatography coupled with electrospray ionization ion trap time-of-flight multistage mass spectrometry (HPLC–ESI-IT-TOF-MS) analysis was performed on a Shimadzu LCMS-IT-TOF instrument with methanol-acetonitrile (50:50, v/v) as eluent. Thermogravimetric analysis (TGA) was performed on a STA 409PC thermogravimeter by measuring the weight loss while heating at a rate of $10\text{ }^{\circ}\text{C min}^{-1}$ under nitrogen. Electrochemical measurement were performed with a Potentiostat/Galvanostat Model 283 (Princeton Applied Research) electrochemical workstation, using Pt as working electrode, platinum wire as auxiliary electrode, and a Ag wire as reference electrode standardized against ferrocene/ferrocenium. The reduction/oxidation potentials were measured in

anhydrous DMF solution containing 0.1 M n-Bu₄NPF₆ as supporting electrolyte at a scan rate of 150 mV s⁻¹.

1,4-di(thiophen-2-yl)benzo[*g*]phthalazine (dtbpa)¹ and 3-(2-thenoyl)-2-naphthoic Acid² was synthesized as according to literatures.



Scheme S1.1: Synthesis of the ligand **Ftbpa**.

4-(thiophen-2-yl)-1-(2H)-benzo[*g*]phthalazinone:

4-(thiophen-2-yl)-1-(2H)-benzo[*g*]phthalazinone was synthesized by a modified method of previous reported literature.³ A mixture of 3-(2-Thenoyl)-2-naphthoic Acid (2.82 g, 10 mmol) and 80% hydrazine hydrate (0.75g, 12mmol) in ethanol was refluxed for 16h under nitrogen. Then the reaction mixture was cooled overnight at 0°C. The resulting white precipitate was filtered off and washed with water and methanol to give 4-(thiophen-2-yl)-1-(2H)-benzo[*g*]phthalazinone as white solid. Yield: 80%. ¹H NMR (600 MHz, CDCl₃): δ 10.03 (s, 1H), 9.10 (s, 1H), 8.65 (s, 1H), 8.16 (m, 1H), 8.06 (m, 1H), 7.72 (m, 2H), 7.60 (d, 1H, *J*= 2.7 Hz), 7.56 (d, 1H *J*= 5.1

Hz), 7.28 (m, 1H). HRMS (ESI⁺) m/z: calcd for C₁₆H₁₁N₂OS⁺ [M+H]⁺: 279.0592, found: 279.0564.

1-chloro-4-(thiophen-2-yl)benzo[*g*]phthalazine:

1-chloro-4-(thiophen-2-yl)benzo[*g*]phthalazine was synthesized by a modified method of previous reported literature.⁴ To a 100ml round-bottom flask, 4-(thiophen-2-yl)-1-(2H)-benzo[*g*]phthalazinone (2.78 g, 10 mol) and phosphorus oxychloride (3.06g, 20mmol) were added at once. And the mixture was refluxed for 16h. After cooling to room temperature, the mixture was then poured into ice-water, basified with a solution of ammonia (2 M) and stirred for 15 min. The resulting precipitate was filtered off and washed with water and light petroleum to give 1-chloro-4-(thiophen-2-yl)benzo[*g*]phthalazine as yellow solid. Yield: 96%. ¹H NMR (600 MHz, CDCl₃): δ 9.06 (s, 1H), 8.96 (s, 1H), 8.24 (d, 1H, *J*= 7.9 Hz), 8.16 (d, 1H, *J*= 8.1 Hz), 7.82 (d, 1H, *J*= 3.0 Hz), 7.80-7.73 (m, 2H), 7.67 (d, 1H, *J*= 5.0 Hz), 7.32 (m, 1H). HRMS (ESI⁺) m/z: calcd for C₁₆H₁₀ClN₂S⁺ [M+H]⁺: 297.0253, found: 297.0249.

1-(2,4-bis(trifluoromethyl)phenyl)-4-(thiophen-2-yl)benzo[*g*]phthalazine (Ftbpa):

To a 100ml round-bottom flask, 1-chloro-4-(thiophen-2-yl)benzo[*g*]phthalazine (1.184 g, 4 mmol), 2,4-bis(trifluoromethyl)phenylboronic acid (1.238 g, 4.8 mmol), tetrakis(triphenylphosphine)palladium(0) (0.462 g, 0.4 mmol), potassium carbonate (1.66 g, 12 mmol), toluene (20 mL), ethanol (16 mL) and distilled water (10 mL)

were added. The mixture was heated to reflux under a nitrogen atmosphere for 24 h. After cooling to room temperature, the mixture was extracted with CH₂Cl₂, and the organic layer was washed with water and then dried over MgSO₄ and concentrated. The crude product was purified by column chromatography on silica gel (hexane/dichloromethane = 3:1, v/v). And the crude product was recrystallized from dichloromethane/hexane to give 1-(2,4-bis(trifluoromethyl)phenyl)-4-(thiophen-2-yl)benzo[*g*]phthalazine as yellow solid. Yield: 72%. ¹H NMR (600 MHz, CDCl₃): δ 9.27 (s, 1H), 8.22 (m, 2H), 8.08-8.04 (m, 4H), 7.81-7.72 (m, 4H), 7.40 (t, 1H). HRMS (ESI⁺) *m/z*: calcd for C₂₄H₁₃F₆N₂S⁺ [M+H]⁺: 475.0704, found: 475.0686.

Tris(1,4-di(thiophen-2-yl)benzo[*g*]phthalazine) iridium(III) (Ir(dtbpa)₃):

1,4-di(thiophen-2-yl)benzo[*g*]phthalazine (1.136 g, 3.3 mmol), IrCl₃·3H₂O (0.352 g, 1 mmol), 2-methoxyethanol (30 mL) and distilled water (10 mL). The mixture was stirred at 100°C for 24 h under nitrogen. After cooling to room temperature, distilled water (40 mL) was added and the precipitate was filtered off and washed with water, ethanol and hexane. The crude product was purified by column chromatography over aluminum oxide using hexane/dichloromethane (1:1, v/v) as the eluent to give Ir(dtbpa)₃ as black solid. Yield: 65%. ¹H NMR (600 MHz, CDCl₃): δ 9.36 (s, 3H), 8.95 (s, 3H), 8.15 (d, *J* = 8.0 Hz, 3H), 8.05 (d, *J* = 8.1 Hz, 3H), 7.65 – 7.56 (m, 9H), 7.41 (d, *J* = 3.3 Hz, 3H), 7.07 (d, *J* = 4.8 Hz, 3H), 6.97 (d, *J* = 4.9 Hz, 3H), 6.91 – 6.86 (m, 3H). HRMS (ESI⁺) *m/z*: calcd for C₆₀H₃₄IrN₆S₆⁺ [M+H]⁺: 1223.0798, found:

1223.0831. Elemental analysis calcd for $C_{60}H_{33}IrN_6S_6$: C, 58.95; H, 2.72; N, 6.87; found: C, 58.78; H, 2.88; N, 6.74.

Tris(1-(2,4-bis(trifluoromethyl)phenyl)-4-(thiophen-2-yl)benzo[*g*]phthalazine)

iridium(III) ($Ir(Ftbpa)_3$):

1-(2,4-bis(trifluoromethyl)phenyl)-4-(thiophen-2-yl)benzo[*g*]phthalazine (1.564 g, 3.3 mmol), $IrCl_3 \cdot 3H_2O$ (0.352 g, 1 mmol), 2-methoxyethanol (30 mL) and distilled water (10 mL). The mixture was stirred at 100°C for 24 h under nitrogen. After cooling to room temperature, distilled water (40ml) was added and the precipitate was filtered off and washed with water, ethanol and hexane. The crude product was purified by column chromatography over aluminum oxide using hexane/dichloromethane (2:1, v/v) as the eluent. It was further purified by sublimation at 300-320 °C and 7×10^{-4} Pa to give $Ir(Ftbpa)_3$ as black solid. Yield: 35%. 1H NMR (600 MHz, DMSO): δ 9.47-9.33 (m, 3H), δ 8.47-9.39 (m, 3H), δ 8.17-7.55 (m, 20H), δ 7.04-6.53 (m, 7H). HRMS (MALDI-TOF): calcd for $C_{72}H_{33}F_{18}IrN_6S_3 [M]^+$: 1612.1271, found: 1612.3755. Elemental analysis calcd for $C_{72}H_{33}F_{18}IrN_6S_3$: C, 53.63; H, 2.06; N, 5.21. Found: C, 53.95; H, 2.12; N, 5.32.

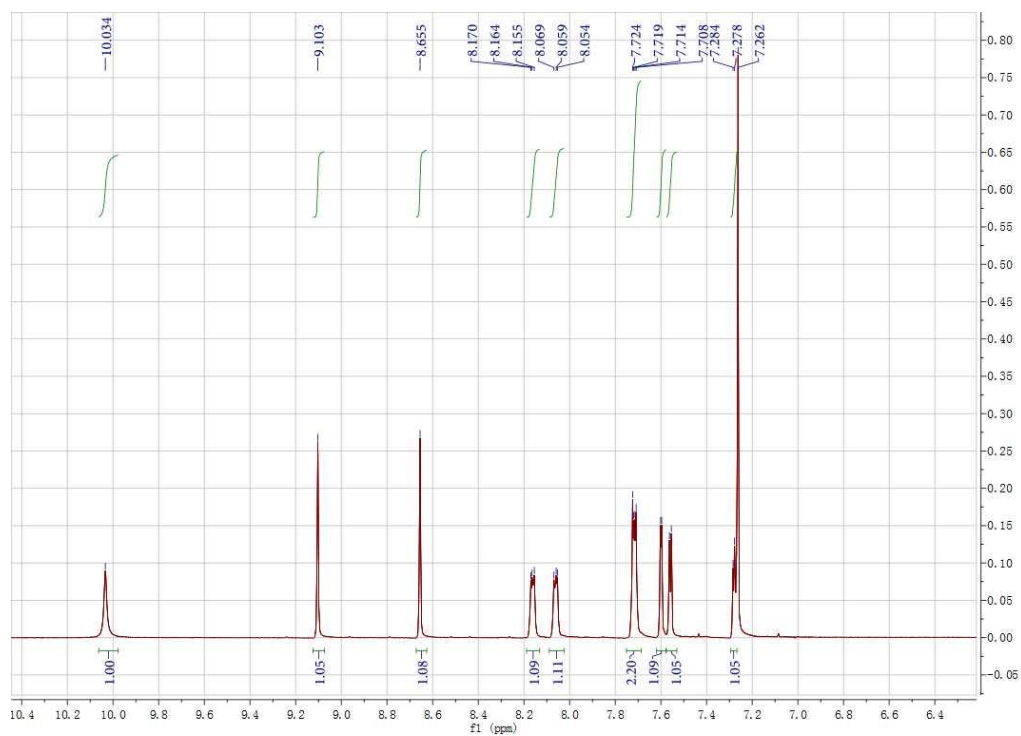


Figure S1.1. ¹H NMR spectrum of 4-(thiophen-2-yl)-1-(2H)-benzo[g]phthalazinone.

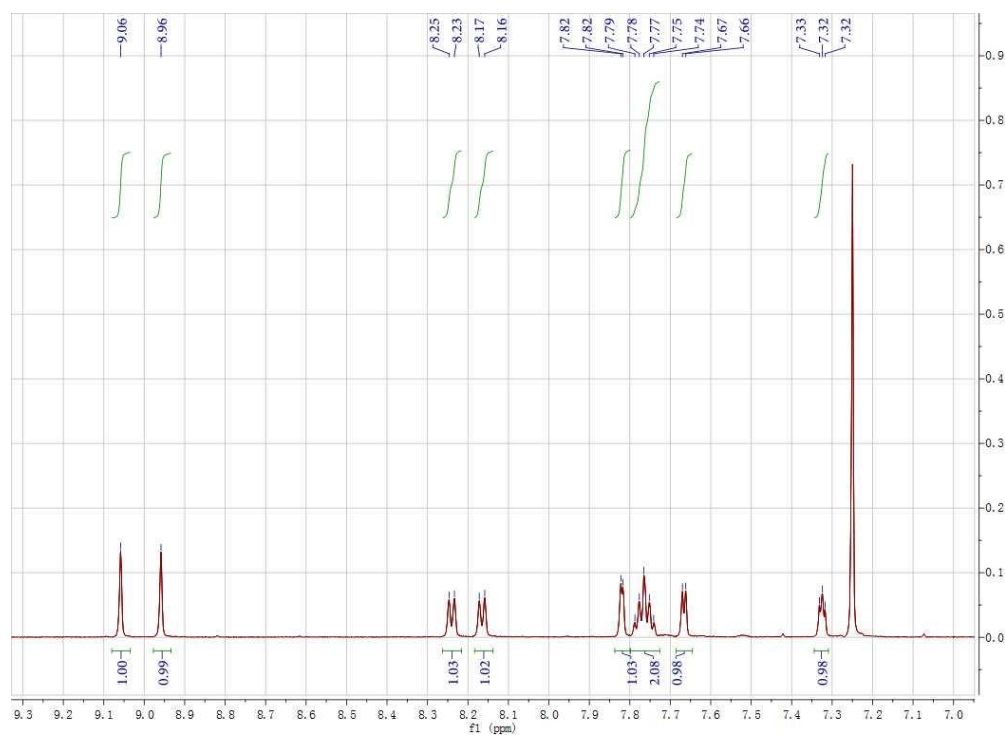


Figure S1.2. ¹H NMR spectrum of 1-chloro-4-(thiophen-2-yl)benzo[g]phthalazine.

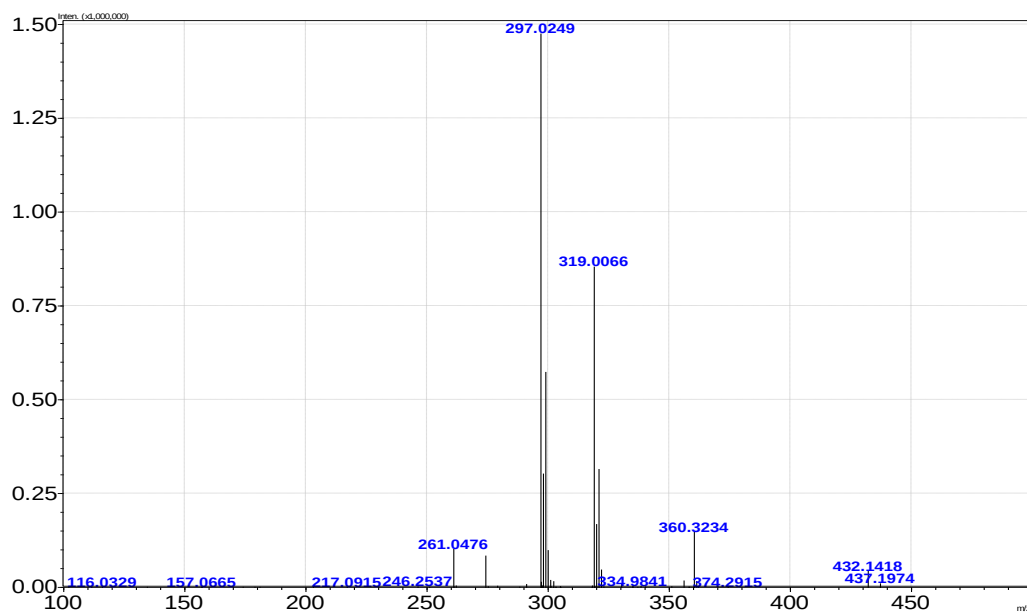


Figure S1.3. HRMS-ESI⁺ spectrum of 1-chloro-4-(thiophen-2-yl)benzo[*g*]phthalazine.

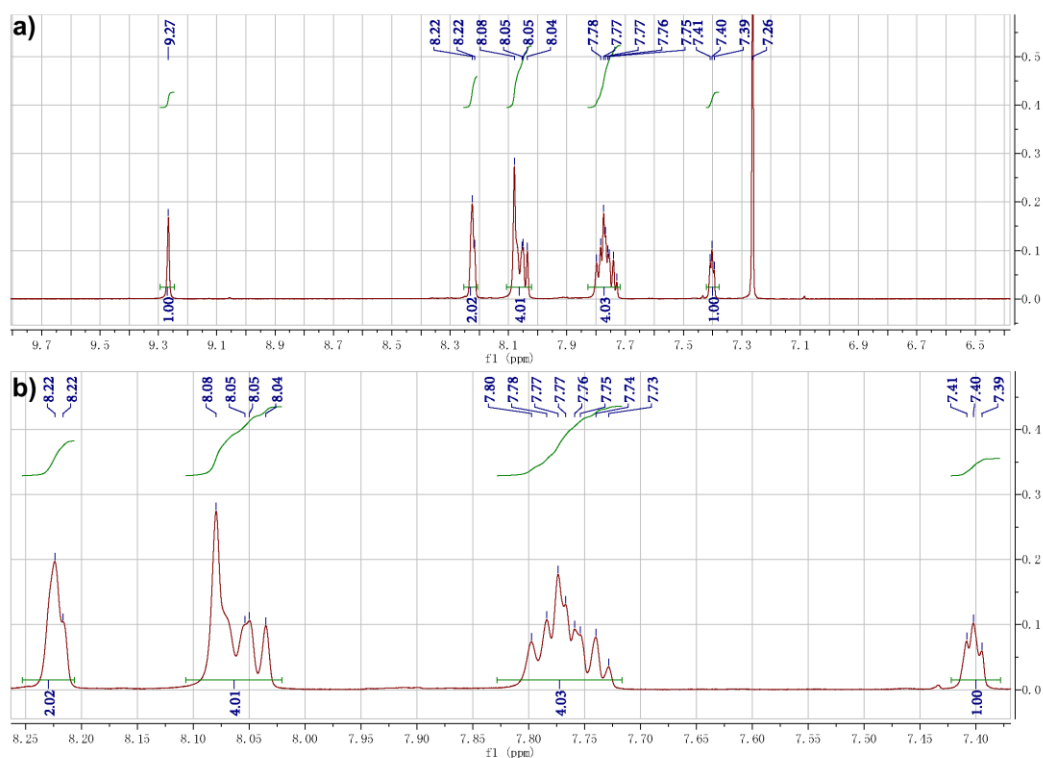


Figure S1.4. a) ¹H NMR spectrum of Ftbpa. b) The ¹H NMR spectrum of Ftbpa with range from 8.25 ppm to 7.36 ppm.

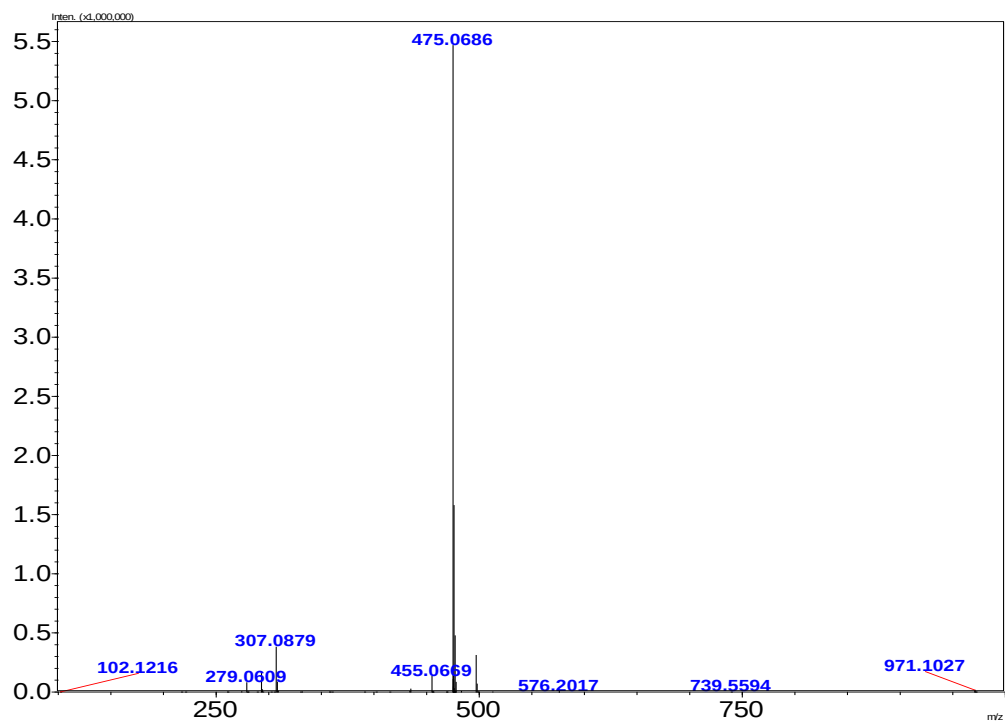


Figure S1.5. HRMS-ESI⁺ spectrum of Ftbpa.

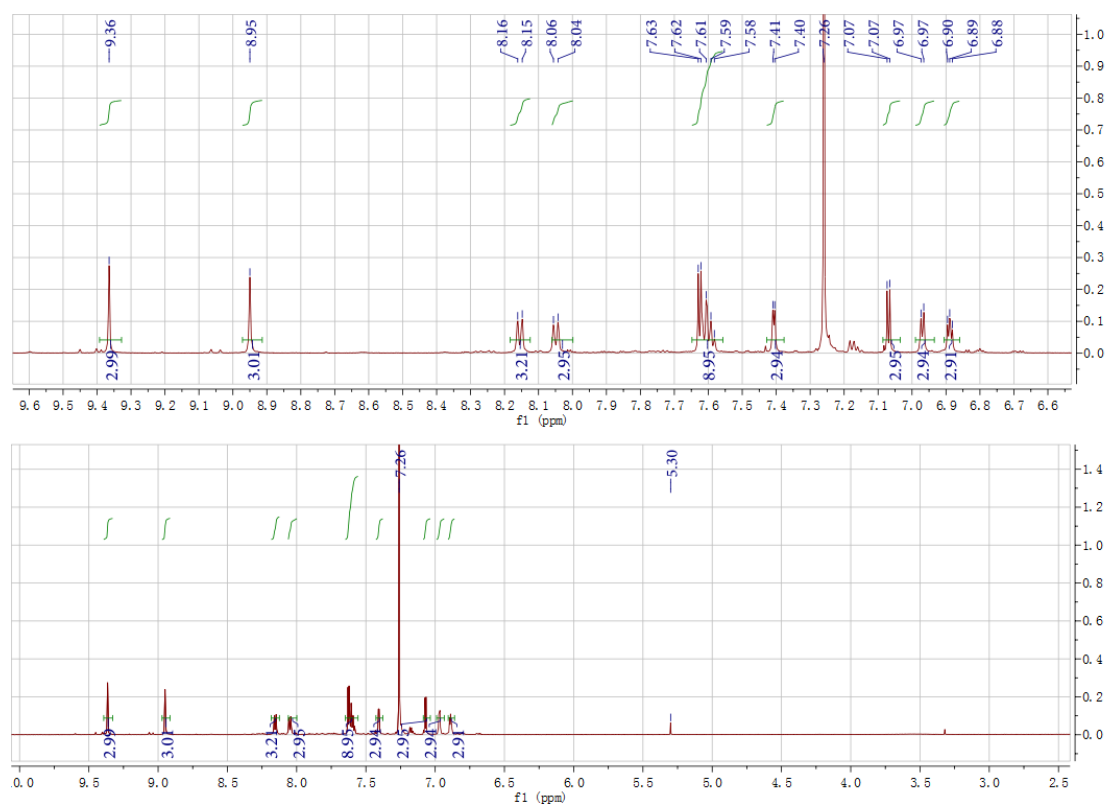


Figure S1.6. ¹H NMR spectrum of Ir(dtbp)₃.

area marked. Inset in (b): qualitative table of the TIC chromatogram. MS spectra of sublimated **Ir(dtbpa)₃** film at retention time = c) 2.525 min and d) 10.353 min corresponding to (a).

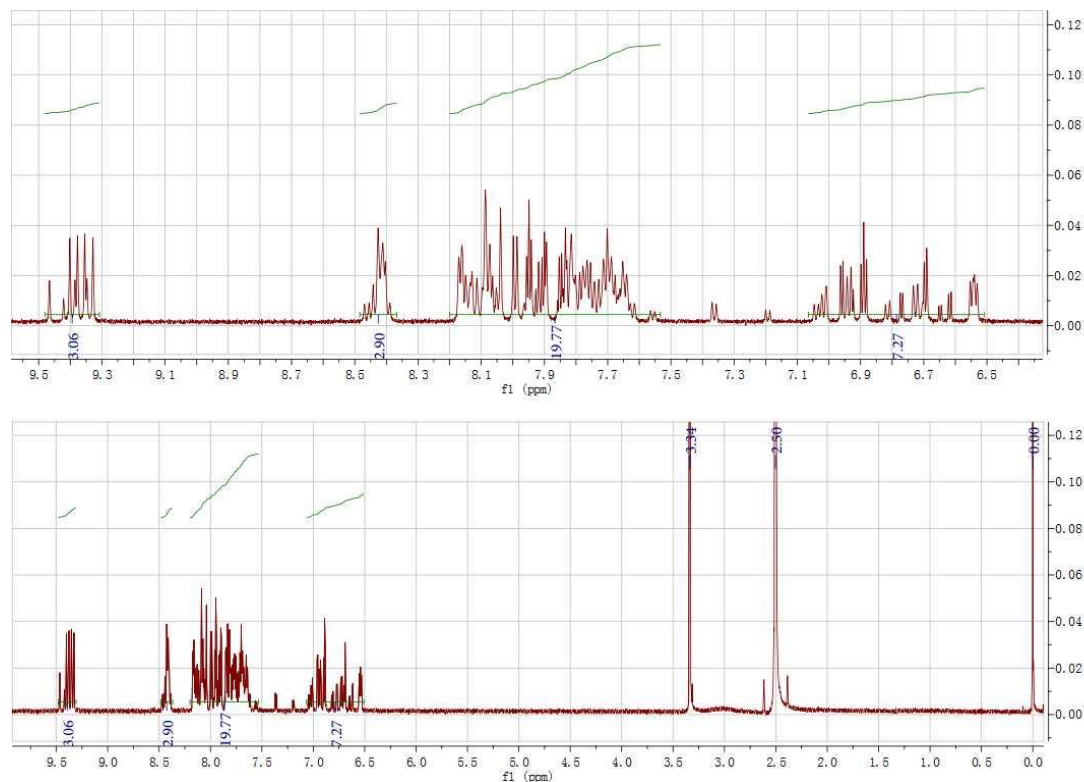


Figure S1.9. ¹H NMR spectrum of **Ir(Ftbpa)₃**.

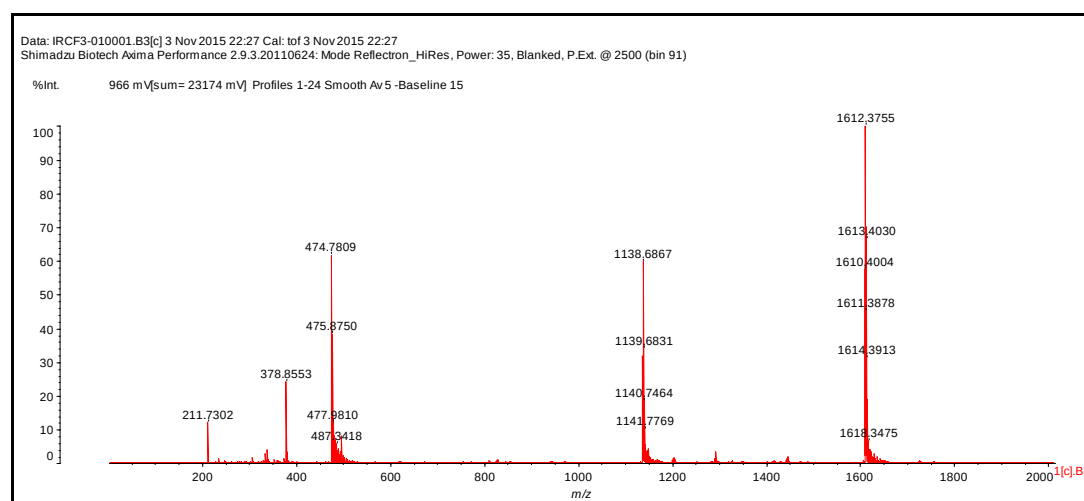


Figure S1.10. MALDI-TOF-MS spectrum of **Ir(Ftbpa)₃**.

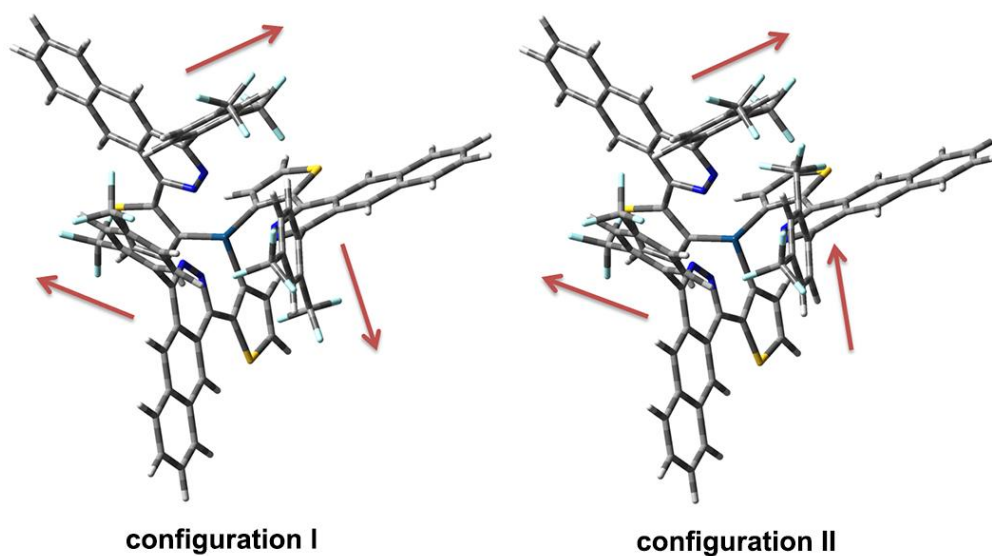


Figure S1.11. Possible configurations of facial **Ir(Ftbpa)₃** with red arrows denoting the direction of trifluoromethyl groups at ortho-position.

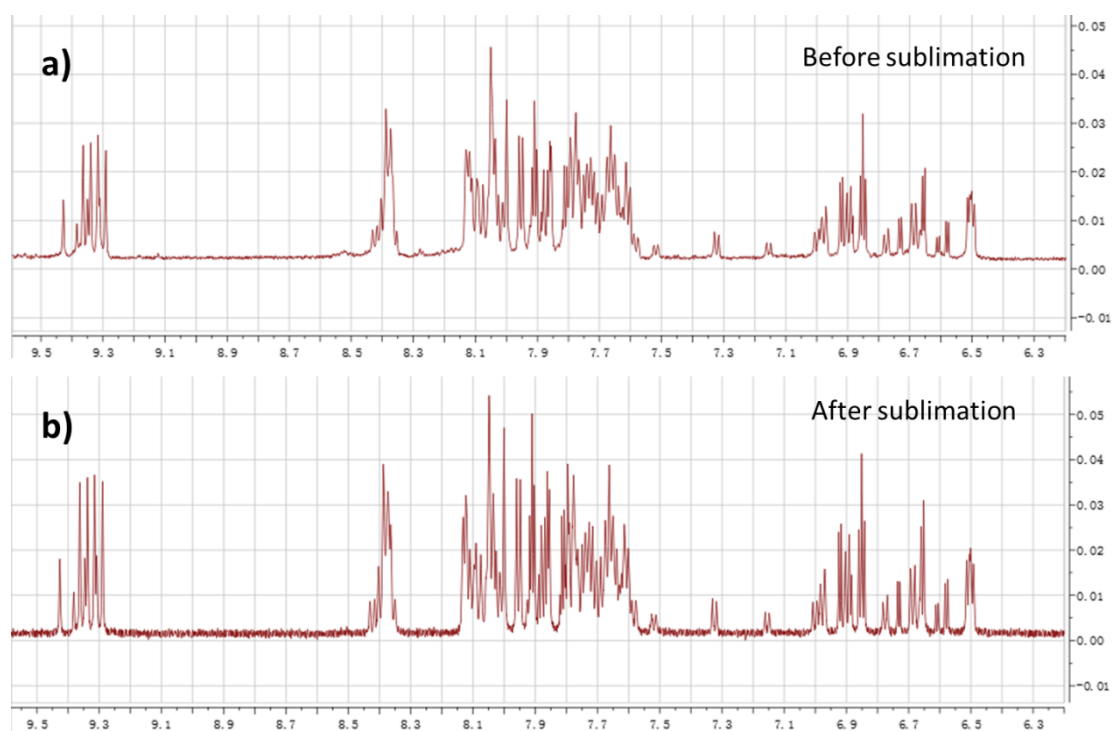


Figure S1.12. ¹H NMR spectrum of **Ir(Ftbpa)₃** a) before and b) after sublimation.

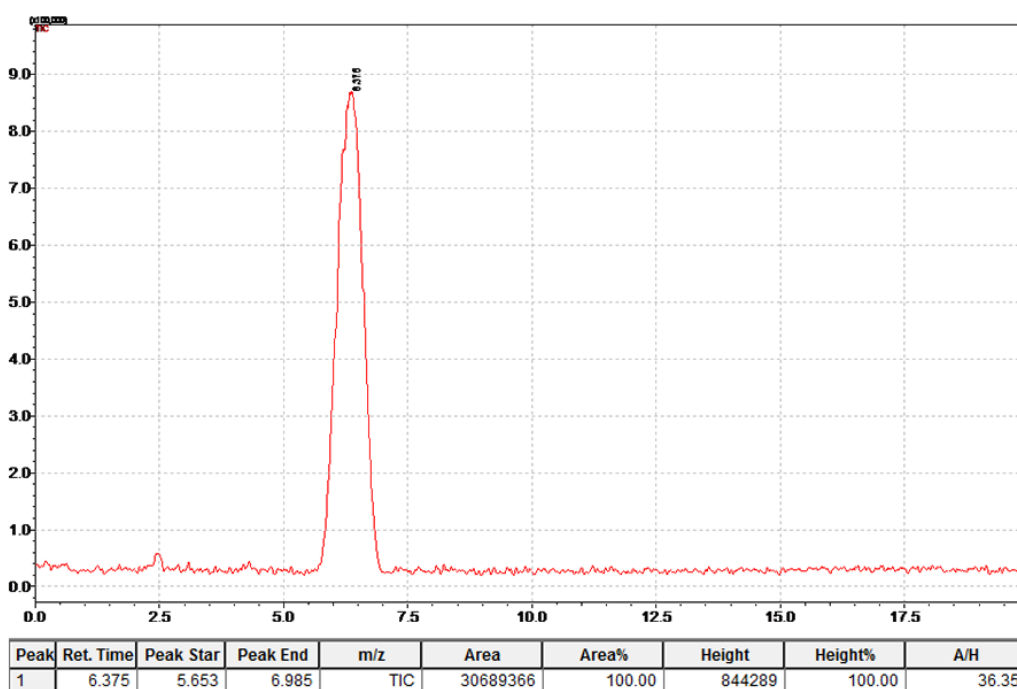


Figure S1.13. TIC chromatogram of **Ir(Ftbpa)₃** with retention time marked and qualitative table provided.

Single-Crystal Structure: The single crystal of **Ir(dtbp)₃** was obtained from diffusion of a chloroform/hexane mixture. The low temperature (104.6K) single-crystals X-ray experiments were performed on a Rigaku RAXIS-SPIDER IP diffractometer with graphite-monochromatized MoK α radiation ($\lambda=0.71073$ Å). Data collection and reduction, cell refinement, and experiential absorption correction for all compounds were performed with the Rigaku RAPID AUTO software package (Rigaku, 1998, Version 2.30). The structure was solved by direct methods and refined against F² by full-matrix least-squares techniques. CCDC 1510452 contains the supplementary crystallographic data for this paper. These data can be obtained free of

charge from The Cambridge Crystallographic Data Centre via

www.ccdc.cam.ac.uk/data_request/cif.

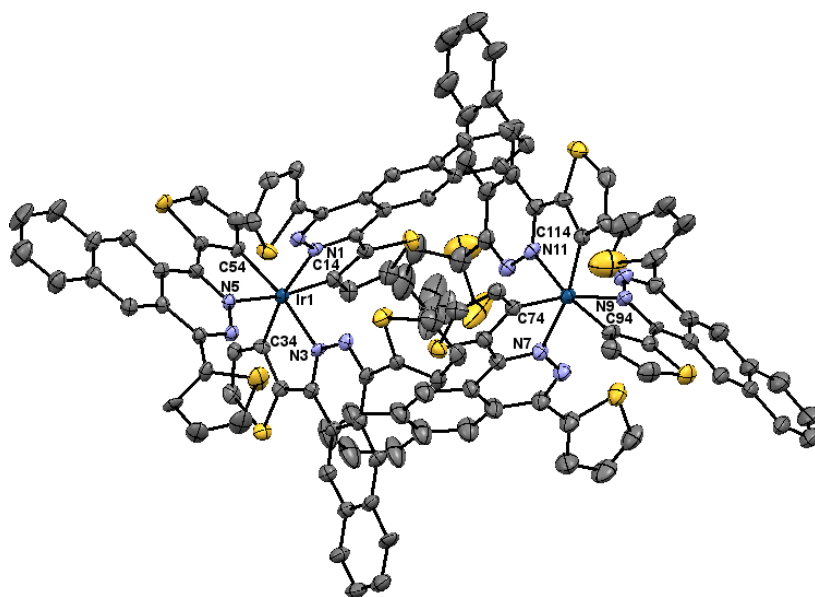


Figure S1.14. Single crystal structure of $\text{Ir}(\text{dtbpa})_3$ with thermal ellipsoids plotted at 50% probability level with solvent molecules hided.

Table S1. Bond lengths and angles of compound **Ir(dtbp_a)₃** from X-ray crystallography data.

	Ir-C bond lengths		Ir-N bond lengths		Angles (°)	
		(Å)		(Å)		
Molecule 1	Ir1-C54	2.019	Ir1-N5	2.105	C54-Ir1-N5	77.88
	Ir1-C14	2.008	Ir1-N1	2.081	C14-Ir1-N1	78.46
	Ir1-C34	2.007	Ir1-N3	2.097	C34-Ir1-N3	78.42
Molecule 2	Ir2-C114	2.013	Ir2-N11	2.102	C114-Ir2-N11	78.22
	Ir2-C94	2.018	Ir2-N9	2.097	C94-Ir2-N9	78.02
	Ir2-C74	2.019	Ir2-N7	2.104	C74-Ir2-N7	78.55
Average ^[a]	Ir-C	2.014	Ir-N	2.098	C-Ir-N	78.26

[a] Average data of Bond lengths and angles of compound **Ir(dtbp_a)₃** from X-ray crystallography data.

2. Quantum chemical calculations:

All of the calculations were carried out with the Gaussian 09 program package using a spin-restricted formalism in the gas phase.⁵ The ground and excited electronic states of the complexes were calculated using density functional theory (DFT) and time-dependent DFT (TD-DFT) at the B3LYP level.⁶ The standard valence double- ξ polarized basis sets were used for C, H, N, F and S (6-31G*) and Ir (LANL2DZ). An effective core potential (ECP) replaces the inner core electrons of Ir, leaving the outer core [(5s)²(5p)⁶] electrons and the (5d)⁶ valence electrons of Ir(III). The ground state structures of all studied Ir(III) complexes were first optimized with DFT without imposing any symmetry restriction. The optimized structures were then used to

calculate the five lowest singlet ($S_0 \rightarrow S_5$) and triplet optical electronic transitions ($S_0 \rightarrow T_5$) using the TD-DFT method. The iridium atom's contributions to frontier molecular orbitals were obtained using the Multiwfn program with Ros–Schuit (SCPA) partition and used to calculate the metal-to-ligand charge transfer (MLCT) percentage in each assignment during the singlet and triplet optical transitions.⁷

Table S2.1. Bond lengths and angles of compound **Ir(dtbp_a)₃** from quantum chemical calculations.

	Ir-C bond lengths (Å)	Ir-N bond lengths (Å)	C-Ir-N Angles (°)
Ligand 1	2.009	2.158	77.67
Ligand 2	2.008	2.160	77.70
Ligand 3	2.006	2.160	77.71
Average ^[a]	2.008	2.159	77.69
Average ^[b]	2.014	2.098	78.26

[a] Average data of Bond lengths and angles of complex **Ir(dtbp_a)₃** from quantum

chemical calculations. [b] Average data of bond lengths and angles of complex

Ir(dtbp_a)₃ from X-ray crystallography data.

Table S2.2. Selected excited states for **Ir(dtbp_a)₃** and **Ir(Ftbp_a)₃** calculated from TDDFT approach.

Complex	States ($E[\text{eV}]$) ($\lambda[\text{nm}]$) ^[a]	Dominant excitations ^[b]	Character	Assignme nt	MLCT (%)
Ir(dtbp _a) ₃	T ₁ (1.4940) (830)	H → L (57%)	$\pi(\text{dtbp}_a)/d_\pi(\text{Ir})$ → $\pi^*(\text{dtbp}_a)$	MLCT/IL CT/LLCT	30.32
		H-1 → L+1 (15%)	$\pi(\text{dtbp}_a)/d_\pi(\text{Ir})$ → $\pi^*(\text{dtbp}_a)$		
		H-2 → L+1 (12%)	$\pi(\text{dtbp}_a)/d_\pi(\text{Ir})$ → $\pi^*(\text{dtbp}_a)$		
	T ₂ (1.5099) (821)	H → L+1 (44%)	$\pi(\text{dtbp}_a)/d_\pi(\text{Ir})$ → $\pi^*(\text{dtbp}_a)$	MLCT/IL CT/LLCT	28.94
		H-1 → L (21%)	$\pi(\text{dtbp}_a)/d_\pi(\text{Ir})$ → $\pi^*(\text{dtbp}_a)$		

Ir(Ftbpa) ₃	T ₃ (1.5340) (808)	H-2→L (13%)	π(dtbpa)/d _π (Ir) → π*(dtbpa)	MLCT/IL CT/LLCT	29.05
		H→L+2 (43%)	π(dtbpa)/d _π (Ir) → π*(dtbpa)		
		H-2→L+2 (13%)	π(dtbpa)/d _π (Ir) → π*(dtbpa)		
		H-1→L+2 (11%)	π(dtbpa)/d _π (Ir) → π*(dtbpa)		
		H-2→L (11%)	π(dtbpa)/d _π (Ir) → π*(dtbpa)		
	S ₁ (1.9617) (632)	H→L (100%)	π(dtbpa)/d _π (Ir) → π*(dtbpa)	MLCT/IL CT	35.16
	T ₁ (1.5982) (776)	H→L (51%)	π(dtbpa)/d _π (Ir) → π*(dtbpa)	MLCT/IL CT	32.63
		H-1→L+1 (25%)	π(dtbpa)/d _π (Ir) → π*(dtbpa)		
	T ₂ (1.6143) (768)	H→L+1 (38%)	π(dtbpa)/d _π (Ir) → π*(dtbpa)	MLCT/IL CT/LLCT	31.19
		H-1→L (30%)	π(dtbpa)/d _π (Ir) → π*(dtbpa)		
	T ₃ (1.6277) (762)	H→L+2 (40%)	π(dtbpa)/d _π (Ir) → π*(dtbpa)	MLCT/IL CT/LLCT	31.22
		H-2→L (24%)	π(dtbpa)/d _π (Ir) → π*(dtbpa)		
		H-2→L+2 (20%)	π(dtbpa)/d _π (Ir) → π*(dtbpa)		
	S ₁ (2.0696) (599)	H→L (100%)	π(dtbpa)/d _π (Ir) → π*(dtbpa)	MLCT/IL CT	37.13

[a] Data in parentheses are excitation energies and corresponding wavelengths. [b] H and L denote HOMO and LUMO, respectively; data in parentheses are the contributions of corresponding excitations.

Table S2.3. Contribution of iridium to selected HOMOs of **Ir(dtbpa)₃** and **Ir(Ftbpa)₃**.

Molecular orbital	Contribution of iridium (%)	
	Ir(dtbpa) ₃	Ir(Ftbpa) ₃

HOMO	35.16	37.13
HOMO-1	23.90	30.11
HOMO-2	23.84	30.21
HOMO-3	24.77	16.95
HOMO-4	24.82	17.03
HOMO-5	24.35	17.10

3. Photoluminescence measurements:

The photoluminescence quantum efficiencies were measured using an absolute photoluminescence quantum yield measurement system (Hamamatsu C9920-02G). Absorption spectra were recorded using a UV-vis spectrophotometer (Agilent 8453). The emission spectra and the transient photoluminescence measurements were carried out using a transient spectrometer (Edinburg FL920P). The time-resolved PL spectra were measured on a laser flash photolysis spectrometer (Edinburg LP-920). The 1×10^{-5} M solution was used for photoluminescence spectra measurement. And small-molecule organic films for optical measurements were fabricated by thermal evaporation under high vacuum (10^{-4} Pa) onto clean quartz substrates.

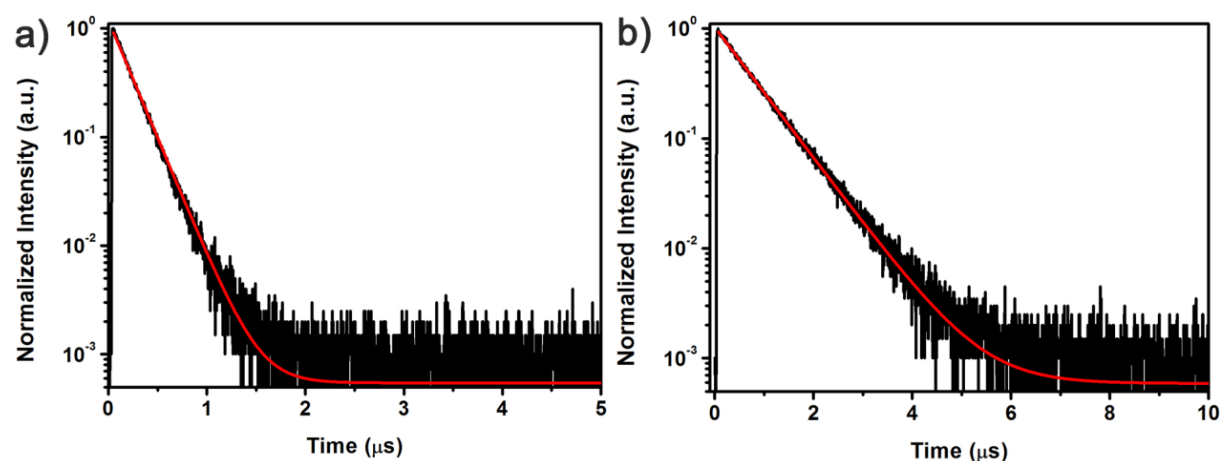


Figure S3.1: a) Transient phosphorescence decay of **Ir(dtbp)₃** in CH₂Cl₂ at 298 K.

b) Transient phosphorescence decay of **Ir(Ftbp)₃** in CH₂Cl₂ at 298 K. Red curves in

(a) and (b) are exponential fitting data.

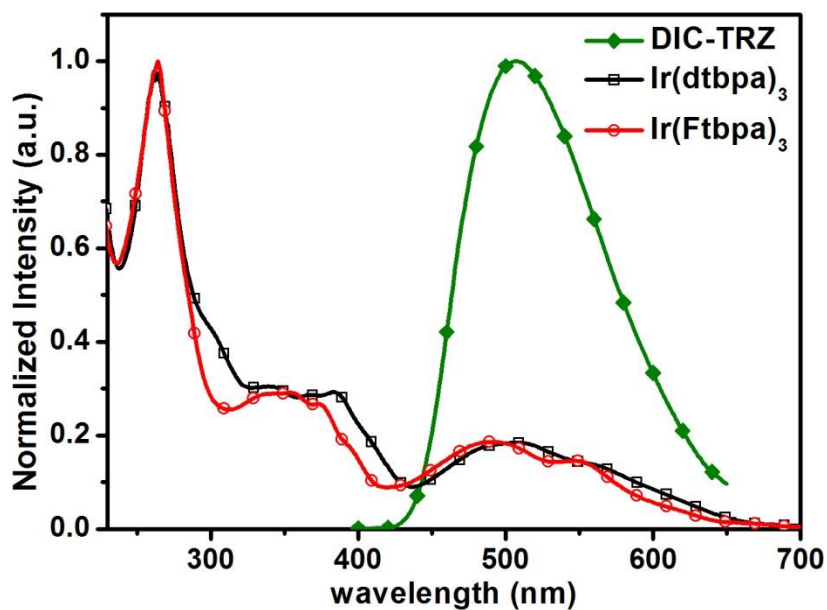


Figure S3.2: Emission spectrum (solid symbol) of the DIC-TRZ and absorption spectra (open symbol) of **Ir(dtbpa)₃** and **Ir(Ftbpa)₃**.

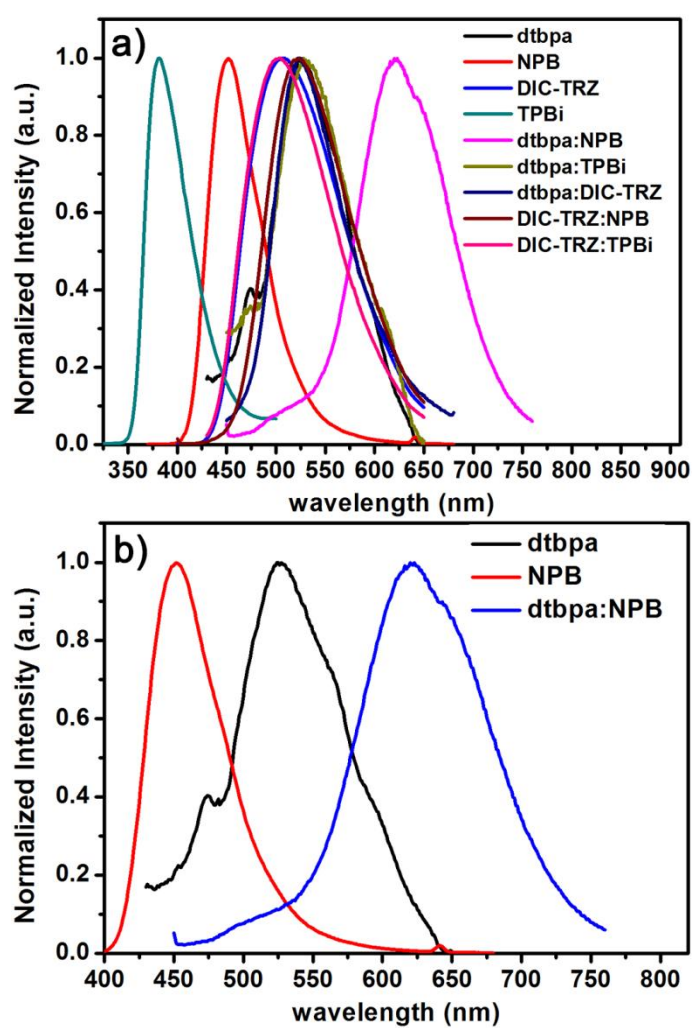


Figure S3.3: a) Emission spectra of the pure films and 1:1 blend films. b) The selected emission spectra of **dtbpa**, **NPB** and **dtbpa:NPB** (1:1) blend films from (a).

4. Device fabrication and measurements:

The devices were fabricated by thermal evaporation under high vacuum (ca. 7×10^{-4} Pa) onto ITO-coated glass substrates. The substrates were carefully cleaned and treated with UV ozone for 10 min before vacuum thermal deposition. All OLED devices were encapsulated in a standard dry nitrogen glove box after fabrication and then measured in ambient. The current–voltage characteristics were measured with a Keithley 4200 semiconductor characterization system and optical power was determined using a Newport 1936-C power meter coupled to a calibrated Newport 918D-UV-OD3 detector with a spectral response range from 200 to 1100 nm. Electroluminescent spectra were collected with a Jobin Yvon FluoroMax-3 fluorospectrophotometer. The EQE of the NIR electroluminescence was determined according to the literature method, by measuring the light intensity in the forward direction and assuming the external emission profile to be Lambertian.⁸

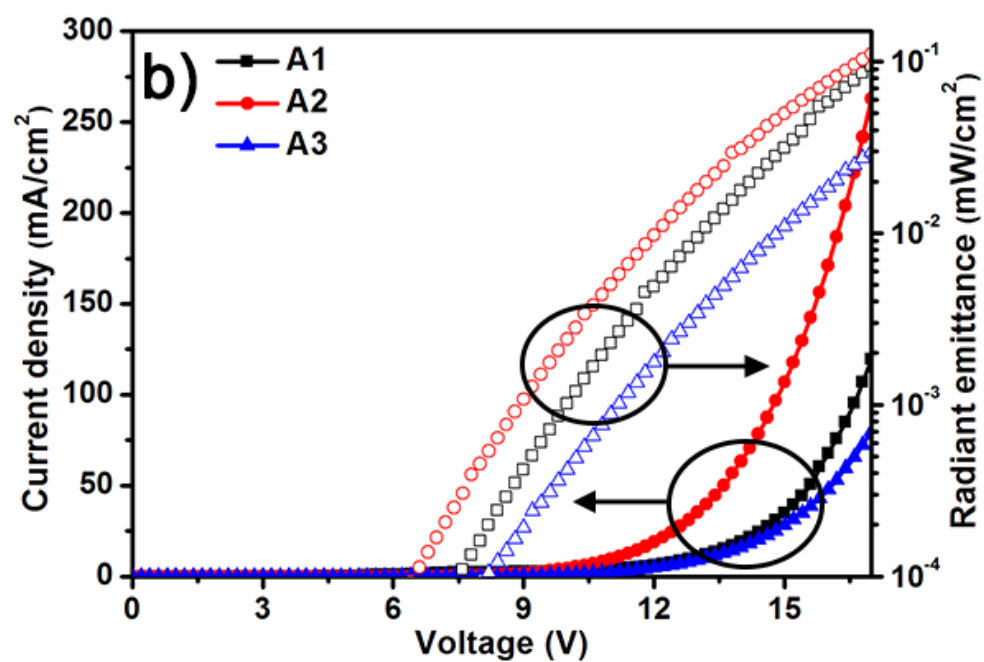


Figure S4.1: Current density (J)–voltage (V)–radiant emittance (R) characteristics of devices based on $\text{Ir}(\text{dtbpa})_3$.

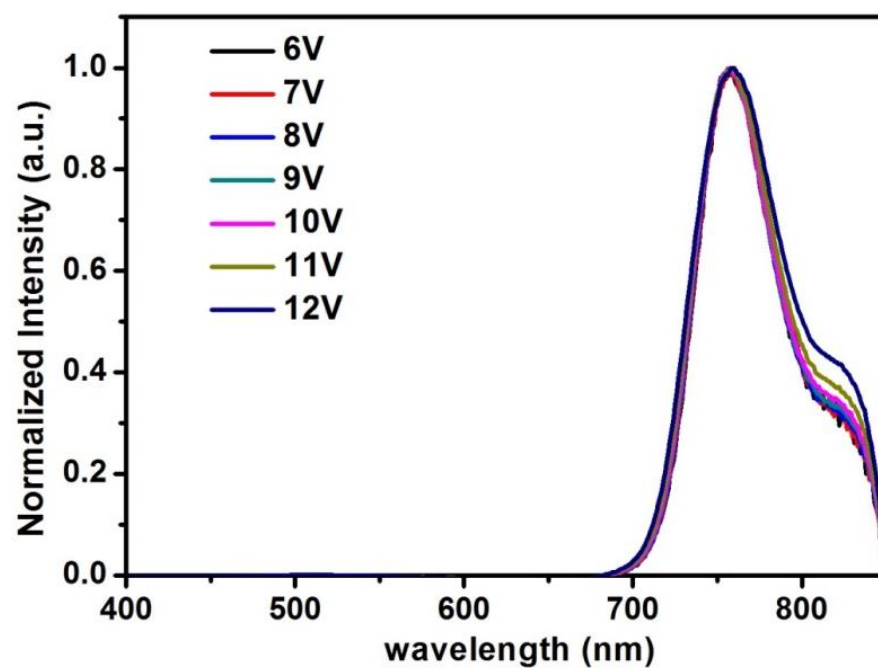


Figure S4.2: Electroluminescent spectra of device B2 recorded at various voltages.

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