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Phenylquinoline Fused Cyclic Derivatives as Electron Acceptor of Exciplex Forming Host For Solution-Processable Red Phosphorescent OLEDs

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Supplementary Information

Contents

General Procedures:

^{1}H NMR spectra were recorded at 300 or 500 MHz on NMR instruments in Chloroform-*d* or DMSO-d_6 solution, ^{13}C NMR spectra were recorded at 101 MHz on Varian Liquid-State NMR instruments in Chloroform-*d* solution. Mass spectra were recorded on electrospray ionization mass spectrometry (ESI-MS) and a Bruker autoflex matrix assisted laser desorption/ionization time-of-flight (MALDI-TOF). 3-bromo-2-phenylquinoline were prepared exactly the same as previously reported procedure[1] .

The UV-visible spectra were recorded on a SHIMADZU UV-1750 spectrometer. Photoluminescence (PL) spectra were measured on a Hitachi F-4600 fluorescence spectrophotometer. The transient lifetime were measured using on a Horiba JobinYvon FluoroLog-3 spectrometer. The PLQY measurement was carried out using a three-step technique by combination of laser, optical fiber, spectrometer, and integrating sphere.[2] Cyclic voltammetry was performed using a CH Instrument 660E electrochemical analyzer under a nitrogen atmosphere in golvebox. Anhydrous DMF and DCM was used as the solvent for reduction and oxidation, respectively. 0.1 M tetra(*n*-butyl) ammonium hexafluorophosphate was used as the supporting electrolyte. A silver wire was used as the pseudo reference electrode, a Pt wire was used as the counter electrode, and platinum column was used as the working electrode. The redox or oxidation potentials are reported referring to ferrocenium/ferrocene (Fc^+/Fc). The onset potential was determined from the intersection of two tangents drawn at the rising and background current of the cyclic voltammogram. Their HOMO and LOMO energy levels were calculated from the onset of oxidation ($E_{\mathrm{ox,\ onset}}$) and reduction potentials ($E_{\mathrm{re,\ onset}}$) according to the equation of $E_{\mathrm{HOMO/LUMO}} = -(E_{\mathrm{ox/re,\ onset}} + 4.8)$ eV, respectively. Differential scanning calorimetry (DSC) measurements were performed on a NETZSCH DSC 200 PC unit at a heating rate of 10 $^{\mathrm{o}}$C min^{-1} from 30 to 270 $^{\mathrm{o}}$C under argon. The glass transition temperature (T_g) was determined from the second heating scan. Thermogravimetric analysis (TGA) measurements were undertaken with NETZSCH STA 449C instrument. The thermal stability of the samples under nitrogen atmosphere was determined by measuring their weight loss (5 %) when heating at a rate of 10 $^{\mathrm{o}}$C min^{-1} from 30 to 550 $^{\mathrm{o}}$C.

The equilibrium geometries of the ground state (S_0) were optimized by density functional theory

(DFT) method using B3LYP functional with 6-31G(d) basis set. Frequency calculations were performed to ensure structures had no imaginary frequencies. Time-dependent density functional theory (TD-DFT) was implemented to offer detailed transition properties in excitation process. All calculations were carried out with Gaussian 09.

OLED Fabrication. The prepatterned indium tin oxide (ITO) substrates were cleaned by ultrasonic acetone bath, followed by ethanol bath. Afterwards, the substrates were dried with N_2 and then loaded into a UV-Ozone chamber. After UV-ozone treatment for 20 min, The PEDOT:PSS layer was directly spin-coated on the ITO substrate as the hole-injecting layer, and then annealed at 120 °C for 10 min inside the N_2-filled glovebox. The emitting layer was also prepared by spin-coating directly on the hole-injecting layer, and then annealed at 50 °C for 10 min. The electron-transporting material and the cathode material were consecutively thermally evaporated onto the emitting layer in a vacuum chamber. Before being taken out of the glovebox, all the devices were encapsulated with UV curable epoxy. The voltage−current−luminance characteristics and the EL spectra were simultaneously measured with PR735 SpectraScan Photometer and Keithley 2400 sourcemeter unit under ambient atmosphere at room temperature.

Synthesis and Characterization

Diphenyl(2-phenylquinolin-3-yl)methanol

To a flask was added with 3-bromo-2-phenylquinoline (0.2 mmol, 57 mg) and 1 ml anhydrous diethyl ether, the reaction mixture was stirred at -78 ºC for 10 min and then added dropwise with 0.2 ml of 1.6 M *n*-BuLi. After another stirring for 1 h, benzophenone (35mg, 0.2mmol) dissolved in 2 ml anhydrous diethyl ether under N_2, was introduced into the reaction system via syringe injection. The mixture was stirred at -78 ºC for a few of minutes and then kept stirring at rt overnight. The reaction was quenched with NH_4Cl aqueous and extracted with ethyl acetate (5 mL x 3). The combined organic phase was washed successively with water, brine and dried with anhydrous Na_2SO_4. The filtrate was concentrated under vacuum and purified through column chromatography to give a white solid. (30 mg, 41% yield). ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.91 (d, *J* = 8.4 Hz, 1H), 7.80 – 7.67 (m, 3H), 7.56 – 7.49 (m, 1H), 7.21 – 7.11 (m, 10H), 7.08 – 7.01 (m, 1H), 6.99 – 6.90 (m, 2H), 6.90 – 6.82 (m, 2H), 6.34 (s, 1H). MS (ESI): 388.18 $[M+H]^+$

Diphenyl(2-phenylquinolin-3-yl)methanol (DPIQ)

Diphenyl(2-phenylquinolin-3-yl)methanol (78 mg, 0.2 mmol) was dissolved in 1 mL dry dichloromethane, and then methane sulfonic acid (0.5 ml) was added to the solution. The resulting mixture was stirred at room temperature overnight, and then quenched with $NaHCO_3$ solution. The organic layer was washed with water and dried with Na_2SO_4. The filtrate was concentrated under vacuum and then purified through column chromatography to give desired product as a white solid.(68 mg, 92% yield) ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.42 (s, 1H), 8.22 – 8.15 (m, 1H), 8.12 (d, *J* = 8.2 Hz, 1H), 7.98 (d, *J* = 8.2 Hz, 1H), 7.80 – 7.72 (m, 1H), 7.62 – 7.51 (m, 4H), 7.35-7.25 (m, 6H), 7.24 – 7.15(m, 4H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 160.68, 152.84, 148.63, 145.65, 143.36, 139.01, 132.82, 131.02, 129.45, 129.25, 128.54, 128.41, 128.37, 128.25, 127.89, 127.09, 126.55, 126.04, 122.33, 63.20 ppm; MS (ESI) 370.16 $[M+H]^+$. HRMS (ESI) (m/z): Calcd. for $C_{28}H_{20}N$ ($M+H^+$): 370.1590, Found: 370.1587.

9-(2-phenylquinolin-3-yl)-9H-fluoren-9-ol

To a reaction flask was added with 3-bromo-2-phenylquinoline (0.2 mmol, 57 mg) and dissolved with 1 ml anhydrous diethyl ether. The reaction mixture was stirred at -78℃ for 10 min and then added dropwise with 0.2 ml of 1.6 M *n*-BuLi. After 1 h, 9-Fluorenone (36 mg, 0.2 mmol) dissolved in 2 ml anhydrous diethyl ether under N_2 was introduced into the reaction system via syringe injection. The mixture was stirred at -78 °C for a few minutes and kept stirring at rt overnight. The reaction was quenched with NH_4Cl aqueous and extracted with ethyl acetate (5 mL x 3). The combined organic phase was washed successively with water and brine, and dried with anhydrous Na_2SO_4. The filtrates were concentrated under vacuum and purified through column chromatography to give a white solid. (50 mg, 65% yield). ^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.31 (s, 1H), 8.22 (d, *J* = 6.0 Hz, 1H), 7.88 (d, *J* = 8.2 Hz, 1H), 7.79 – 7.73 (m, 1H), 7.71 – 7.65 (m, 1H), 7.30 – 7.07 (m, 8H), 6.94 – 6.84 (m, 1H), 6.68 – 6.56 (m, 2H), 6.37 (s, 1H), 5.92 (d, *J* = 7.5 Hz, 2H). MS (ESI): 386.15 $[M+H]^+$.

1-(2-phenylquinolin-3-yl)cyclohexan-1-ol (SFIQ)

1-(2-phenylquinolin-3-yl)cyclohexan-1-ol (30 mg, 0.078 mmol) was dissolved in 1.5 mL dry dichloromethane, and then CH_3SO_3H (0.1 mL) was added to the solution. The resulting mixture was kept stirring at room temperature for 30 minutes, and then quenched with aqueous $NaHCO_3$. The organic layer was separated, washed with water and dried with Na_2SO_4. The filtrates were concentrated under vacuum and purified through column chromatography to give a white solid. (28 mg, 95%). ^{1}H NMR (300 MHz, DMSO-*d*$_6$) δ 8.26 (d, *J* = 7.6 Hz, 1H), 8.15 (d, *J* = 8.4 Hz, 1H), 8.08 (d, *J* = 7.6 Hz, 2H), 7.82 (d, *J* = 8.1 Hz, 1H), 7.78 – 7.70 (m, 1H), 7.67 (s, 1H), 7.60 – 7.33 (m, 5H), 7.20 – 7.11 (m, 2H), 6.73 (d, *J* = 7.6 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.88, 150.84, 148.81, 148.73, 141.71, 141.10, 140.38, 131.18, 130.57, 129.37, 129.28, 128.62, 128.21(2C), 128.19, 128.00, 126.03, 124.41, 124.31, 122.19, 120.30, 63.36 ppm. MS (ESI): 368.14 $[M+H]^+$. HRMS(ESI) (m/z): Calcd. for $C_{28}H_{18}N$ $(M+H^+)$: 368.1434, Found: 368.1431

10,10-dimethyl-9 -(2-phenylquinolin-3-yl)- 9,10-dihydroanthracen -9-ol

To a reaction flask was added with 3-bromo-2-phenylquinoline (76 mg, 0.268 mmol) and 2 ml anhydrous diethyl ether. The reaction mixture was stirred at -78 °C for 10 min and then added dropwise with 0.3 ml of 1.6 M *n*-BuLi. After 1 h, 10, 10-dimethylanthracen-9 (10 H)-one (56.4 mg, 0.254 mmol) dissolved in 1ml anhydrous diethyl ether under N_2 was introduced into the reaction system via syringe injection. The mixture was stirred at -78°C for a few of minutes and kept stirring at rt overnight. The reaction was quenched with NH_4Cl aqueous and extracted with ethyl acetate (5 mL x 3). The combined organic phase was washed successively with water, brine and dried with anhydrous Na_2SO_4. The filtrates were concentrated under vacuum and purified through column chromatography to give a white solid. (30 mg, 30% yield). ^{1}H NMR (300 MHz, DMSO-d_6) δ 9.33 (s, 1H), 8.27 (d, *J* = 9.0, 1.6 Hz, 1H), 7.90 (d, *J* = 6.0 Hz, 1H), 7.82 – 7.66 (m, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.31 – 7.21 (m, 2H), 7.15 – 7.06 (m, 2H), 6.99 – 6.89 (m, 3H), 6.71 – 6.62 (m, 2H), 6.09 (s, 1H), 5.79 (d, *J* = 7.5 Hz, 2H), 1.43 (s, 3H), 1.05 (s, 3H). MS (ESI): 428.21 $[M+H]^+$.

10,10-dimethyl-10H-spiro[anthracene-9,11'-indeno[1,2-b]quinoline] (DM-DHAIQ)

To a sealed tube was added raw material (43 mg, 0.1 mmol), appropriate PPA and 2 ml toluene. The mixture was heated at 120 °C for 4 hours. After cooling to rt, the mixture was poured into ice water and extracted with ethyl acetate. The organic phase was washed successively with water and $NaHCO_3$ solution, dried with anhydrous Na_2SO_4. The filtrates were concentrated under vacuum and purified through column chromatography to give a white solid. (38 mg, 92.7% yield). ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.24 (d, *J* = 7.4 Hz, 1H), 8.15 (d, *J* = 8.5 Hz, 1H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.82 – 7.70 (m, 4H), 7.57 – 7.47 (m, 2H), 7.44 – 7.39 (m, 1H), 7.28 – 7.19 (m, 2H), 6.96 (d, *J* = 7.6 Hz, 1H), 6.92 – 6.85 (m, 2H), 6.20 (d, *J* = 8.0, 2H), 1.97 (s, 3H), 1.92 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.27, 158.73, 149.66, 148.53, 143.19, 139.07, 135.31, 131.94, 131.81, 129.30, 129.27, 129.16, 128.34, 128.31, 128.18, 127.17, 127.13, 126.53, 126.01, 125.93, 121.92, 55.74, 37.66, 36.23, 35.93 ppm; MS (ESI): 410.19 $[M+H]^+$. HRMS(ESI) (m/z): Calcd. for $C_{31}H_{24}N$ $(M+H^+)$: 410.1903, Found: 410.1899.

9-(2-phenylquinolin-3-yl)-9H-xanthen-9-ol

To a reaction flask was added with 3-bromo-2-phenylquinoline (0.2 mmol, 57 mg) and dissolved with 1 ml anhydrous diethyl ether. The reaction mixture was stirred at -78 °C for 10 min and then added dropwise with 0.2 ml of 1.6 M *n*-BuLi. After stirring for 1 h, 9H-xanthen-9-one (40 mg, 0.15 mmol) dissolved in 1 ml anhydrous diethyl ether under N_2 was introduced into the reaction system via syringe injection. The mixture was stirred at -78 ^{o}C for a few minutes and kept stirring at rt overnight. The reaction was quenched with NH_4Cl aqueous and extracted with ethyl acetate (5 mL x 3). The combined organic phase was washed successively with water, brine and dried with anhydrous Na_2SO_4. The filtrates were concentrated under vacuum and purified through column chromatography to give a white solid. (25 mg, 41.7% yield). ^{1}H NMR (500 MHz, DMSO-*d*$_6$) δ 9.31 (s, 1H), 8.26 (d, *J* = 8.1 Hz, 1H), 7.90 (d, *J* = 8.3 Hz, 1H), 7.78 (dd, *J* = 7.4, 7.6 Hz, 1H), 7. 74 (dd, *J* = 7.4, 7.6 Hz, 1H), 7.23 (dd, *J* = 7.5, 7.6 Hz, 2H), 7.08 – 7.02 (m, 3H), 7.01 – 6.96 (m, 2H), 6.86 (s, 1H), 6.83 (d, *J* = 8.3, 2H), 6.75 (dd, *J* = 7.5, 7.6 Hz, 2H), 5.87 (d, *J* = 7.6 Hz, 2H). MS (ESI): 402.16 $[M+H]^+$.

Spiro[indeno[1,2-b]quinoline-11,9'-xanthene] (SXIQ)

To a sealed tube was added with 9-(2-phenylquinolin-3-yl)-9H-xanthen-9-ol (30 mg, 0.08 mmol) acetic acid (1 mL) and 35 % hydrochloric acid (0.25 ml). The reaction mixture was kept stirring at 140 ^{o}C for about 4 h and monitored via TLC till completely converted of initial reactant. The reaction mixture was neutralized via adding $NaHCO_3$ aqueous. The organic layer was separated out, which was washed with water and dried with Na_2SO_4, concentrated and further purified with column chromatography to give a white solid as desired product.(25 mg, 89.3% yield) ^{1}H NMR (300 MHz, DMSO-*d*$_6$) δ 8.23 (d, *J* = 7.5 Hz, 1H), 8.15 (d, *J* = 8.3 Hz, 1H), 8.08 (s, 1H), 7.91 (d, *J* = 7.9 Hz, 1H), 7.80 – 7.72 (m, 1H), 7.63 – 7.45 (m, 3H), 7.38 – 7.19 (m, 5H), 6.89– 6. 81 (m, 2H), 6.37 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 160.14, 155.71, 151.40, 148.77, 147.17, 139.03, 132.30, 131.82, 129.60, 129.31, 128.81, 128.60, 128.40, 128.23, 128.20, 126.22, 126.17, 124.73, 123.61, 121.92, 117.05, 51.94 ppm; MS (ESI): 384.14 $[M+H]^+$. HRMS(MALDI-TOF) (m/z): Calcd. for $C_{28}H_{17}NO$ (M^+): 383.1310, Found: 383.1319.

Thermal properties

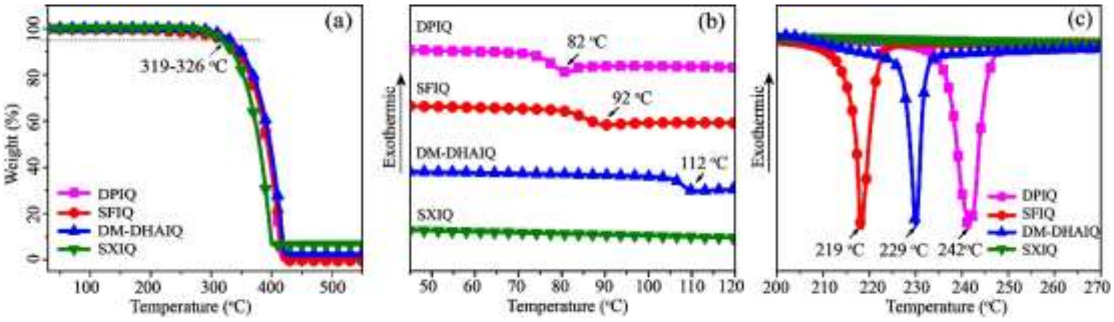


Fig S1. Thermal decomposing curves (a) from TGA, glass transition temperature (b) and melting points (c) from DSC traces recorded at a heating rate of 10 °C min^{-1}.

Cyclic voltammetry data

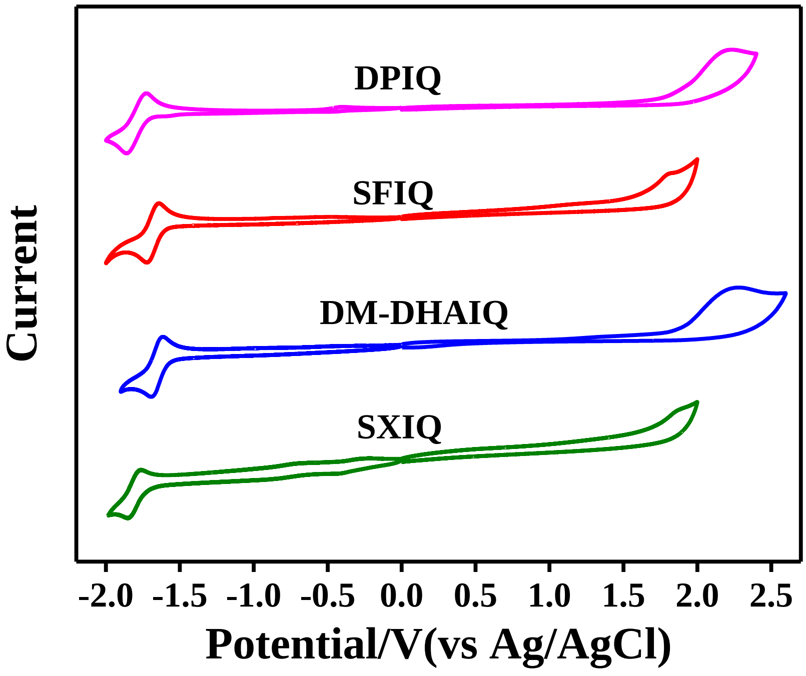
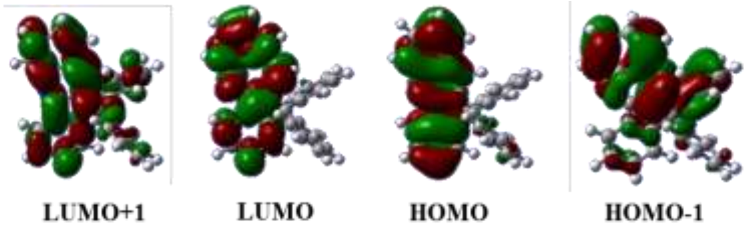


Fig S2. Cyclic voltammograms of DPIQ, SFIQ, DM-DHAIQ and SXIQ. The Oxidation and reduction potentials were measured in DCM and DMF with 0.1 M *n*-Bu_4NPF_6 as the supporting electrolyte, respectively.

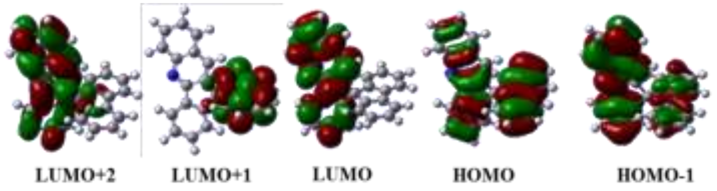
Computational simulations

Table S1 (TD)DFT Calculations of **DPIQ** on S_0 geometry



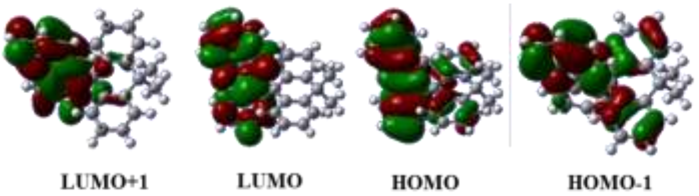
Molecule Orbitals	Energy (eV)	Configurations	Coefficients	Oscillator strength
LUMO+1	−0.77	HOMO-1→LUMO	0.40619	0.2089
LUMO	−1.56	HOMO-1→LUMO+1	0.1618	
HOMO	−5.94	HOMO→LUMO	0.50443	
HOMO-1	−6.14	HOMO→LUMO+1	−0.19414	

Table S2 (TD)DFT Calculations of **SFIQ** on S_0 geometry



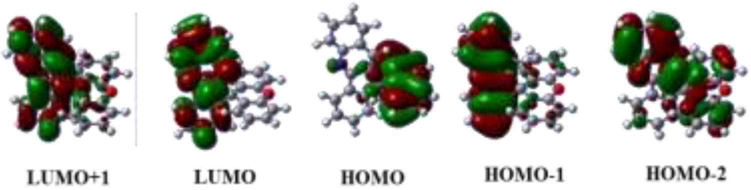
Molecule Orbitals	Energy (eV)	Configurations	Coefficients	Oscillator strength
LUMO+2	−0.70	HOMO−1→LUMO+2	0.11679	0.0805
LUMO+1	−0.90			
LUMO	−1.54			
HOMO	−5.80	HOMO→LUMO	0.68682	
HOMO−1	−5.97			

Table S3 (TD)DFT Calculations of DM-DHAIQ on S_0 geometry



Molecule Orbitals	Energy (eV)	Configurations	Coefficients	Oscillator strength
LUMO+1	−0.66	HOMO−1→LUMO	0.30817	0.2168
LUMO	−1.54	HOMO−1→LUMO+1	0.19107	
HOMO	−5.88	HOMO→LUMO	0.59061	
HOMO−1	−6.05	HOMO→LUMO+1	−0.11375	

Table S4 (TD) DFT Calculations of **SXIQ** on S_0 geometry



Molecule Orbitals	Energy (eV)	Configurations	Coefficients	Oscillator strength
LUMO+1	−0.75	HOMO→LUMO	0.69795	0.0006 Electronic state 1
LUMO	−1.60	HOMO−1→LUMO+1	−0.16994	0.2343 Electronic state 2
HOMO	−5.76	HOMO−1→LUMO	0.54334	
HOMO−1	−5.98	HOMO−2→LUMO+1	−0.18604	
HOMO−2	−6.15	HOMO−2→LUMO	−0.36511	

Photophysical properties

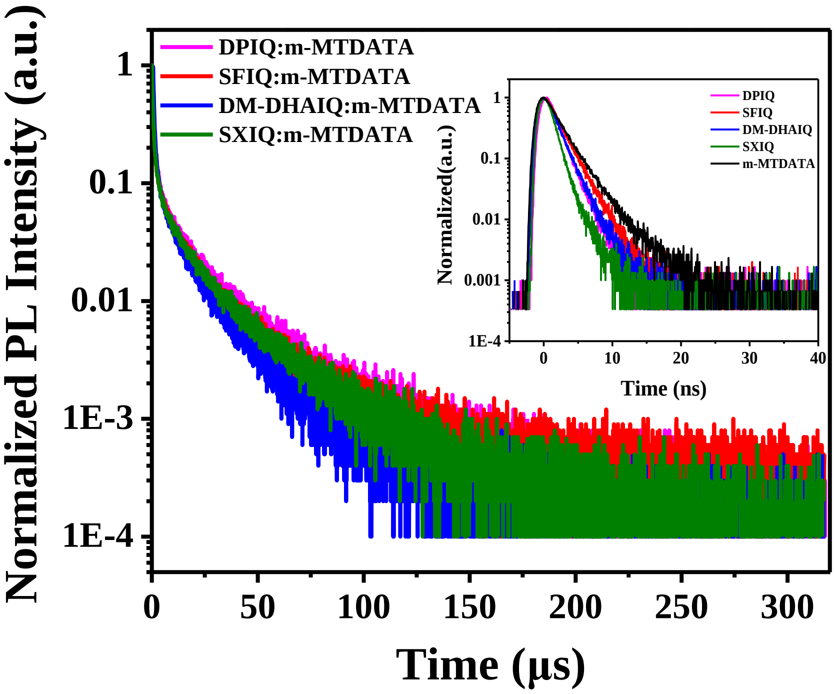


Figure S3. Transient decay curves of the exciplex mixtures and their components (insert) at room temperature.

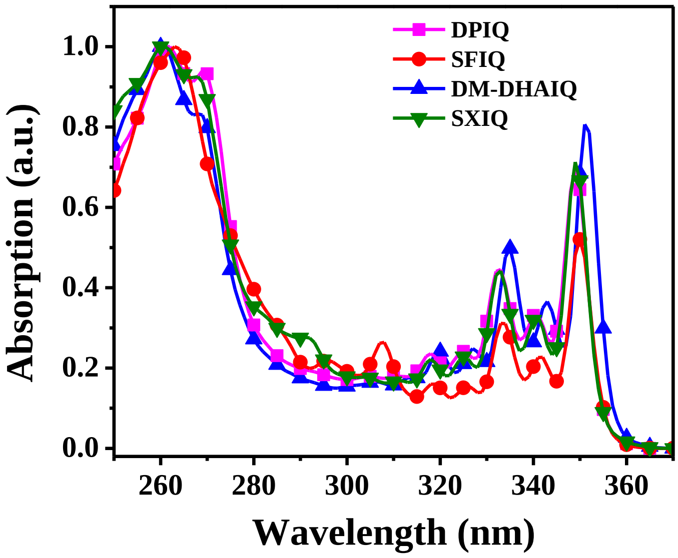


Figure S4. UV-Vis absorption of DPIQ, SFIQ, DM-DHAIQ and SXIQ in PMMA films.

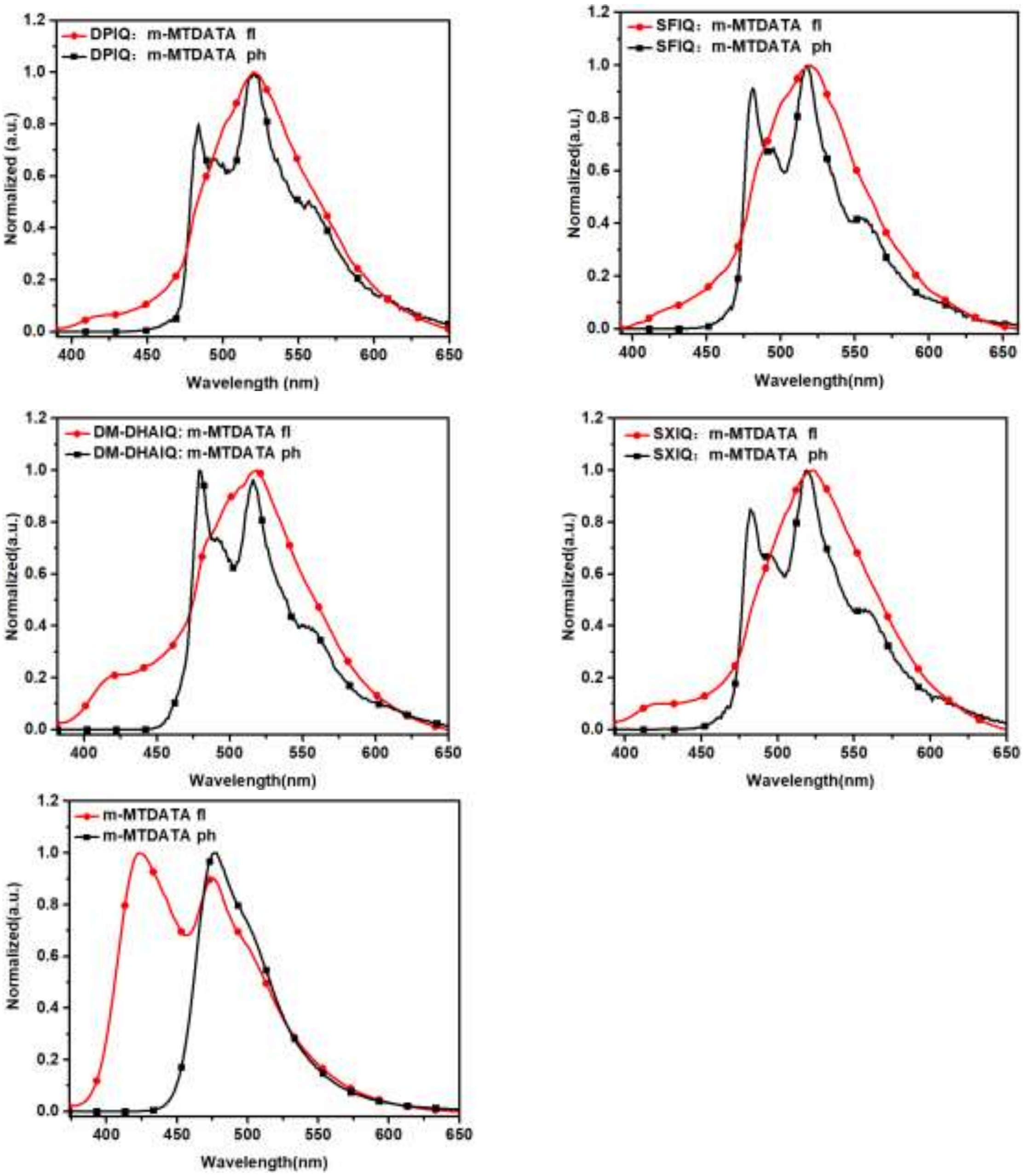


Figure S5. Normalized fluorescence (fl) and delayed fluorescence (at phosphorescence measuring mode, ph, with ~20 ms of delay) spectra of exciplex blends and m-MTDATA at 77 K in PMMA films.

Device data

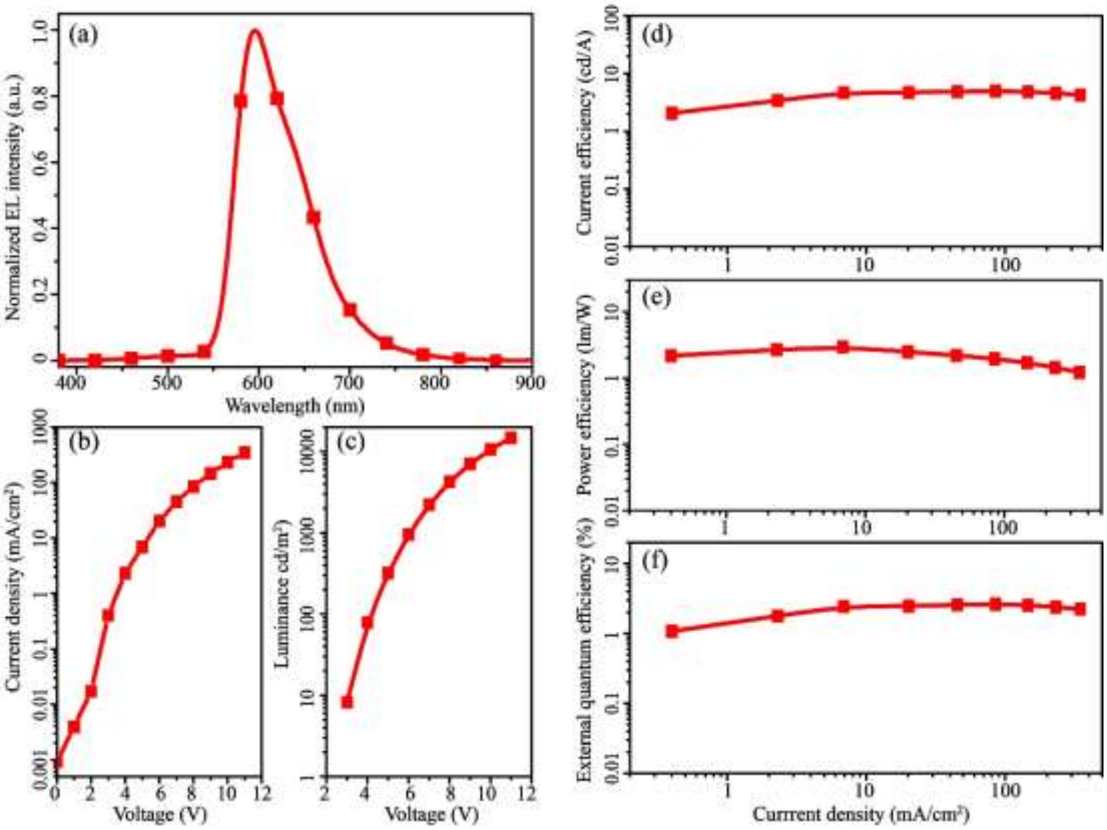


Figure S6. (a) EL spectrum of devices; (b) Current density-Voltage curve; (c) Luminance-Voltage curve; (d) Current efficiency and (e) Power efficiency and (f) EQE versus Current density curves. **Device structure:** ITO/PEDOT: PSS (60 nm)/m-MTDATA: Ir(PQ)$_2$acac (95:5, 60 nm)/TmPyPB (60 nm)/Liq (1 nm)/Al (100 nm).

Table S5. Comparison of the EL Performances of PhOLEDs doped with Ir(PQ)$_2$acac.

Host	Device Type	EQE(max)	Roll off	Ref.
m-MTDATA: DM-DHAIQ	solution	14.1 %	-	This work
m-MTDATA: SFIQ	solution	13.4 %	10.1 %	This work
m-MTDATA: DPIQ	solution	13.1 %	11.6 %	This work
m-MTDATA: SXIQ	solution	10.6 %	9.2 %	This work
SMeFCz	Solution	8.2 %	-	[3]
CzDBF	Solution	8.8 %	-	[4]

m-MTDATA+ TPBI	Solution	9.9 %	-	[5]
2-TNATA + TPBI	Solution	10.2 %	-	[5]
m-CzTP	vacuum	17.2 %	11 %	[6]
BZC-Q	vacuum	12.7 %	-	[7]
BZC-mPQ	vacuum	10.5 %	-	[7]
BZC-pPQ	vacuum	11.5 %	-	[7]
CC-MP2	vacuum	12.2 %	24 %	[8]
CC-MP4	vacuum	15.4 %	25 %	[8]
CC-MP6	vacuum	13.2 %	5 %	[8]
CCMP8	vacuum	15.5 %	28 %	[8]
DSTPA	vacuum	16.4 %	1.2%	[9]
SPPO21	vacuum	20 %	-	[10]
OPH-1C	vacuum	10.5%	-	[11]
2SFOPO	vacuum	14%	-	[12]
DCDPA	vacuum	19.2%	11 %	[13]
mCP	vacuum	18.5%	39 %	[13]

^{1}H NMR and ^{13}C NMR spectra

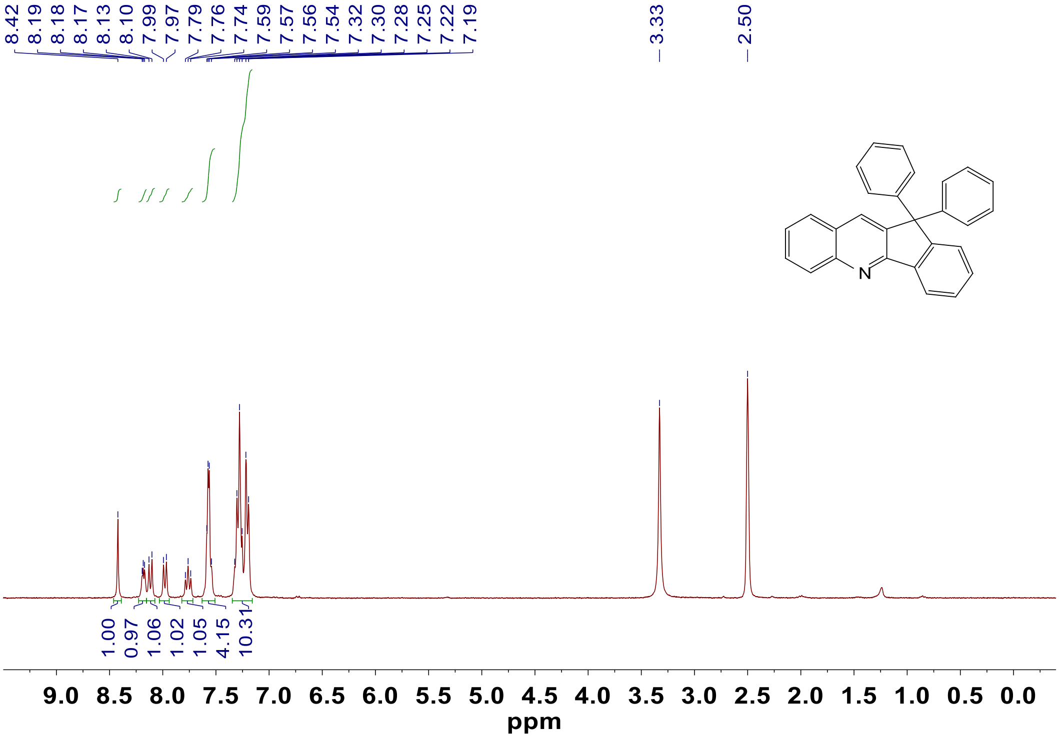


Fig S7.^{1}H NMR of DPIQ

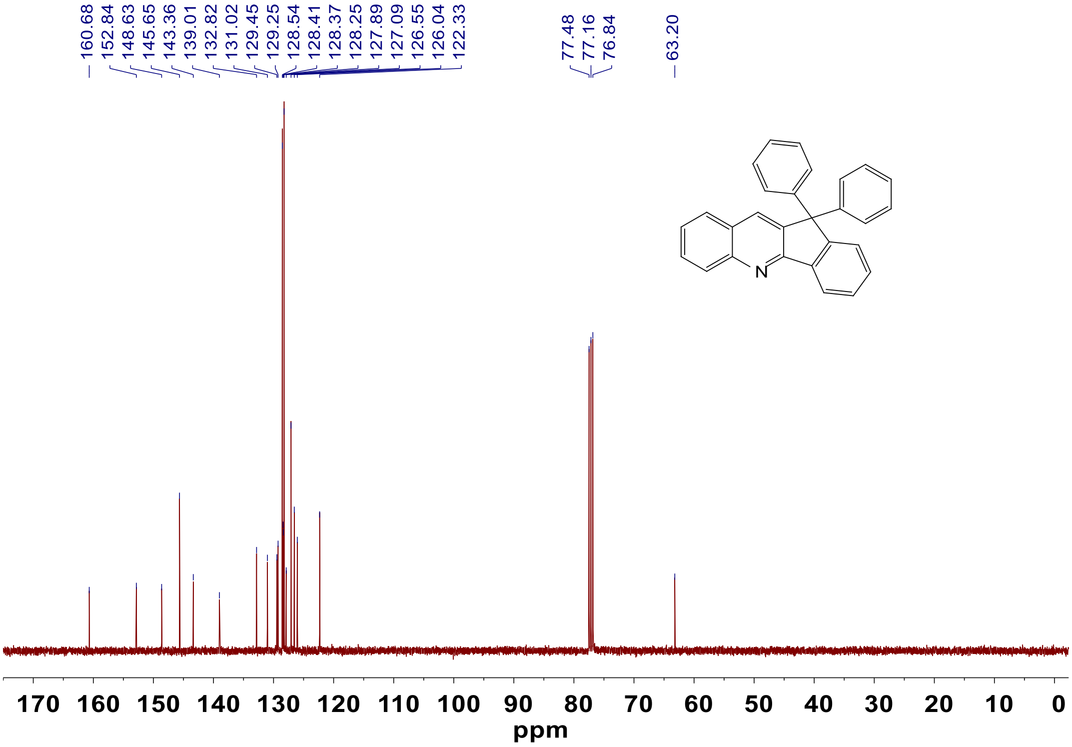


Fig S8. ^{13}C NMR of DPIQ

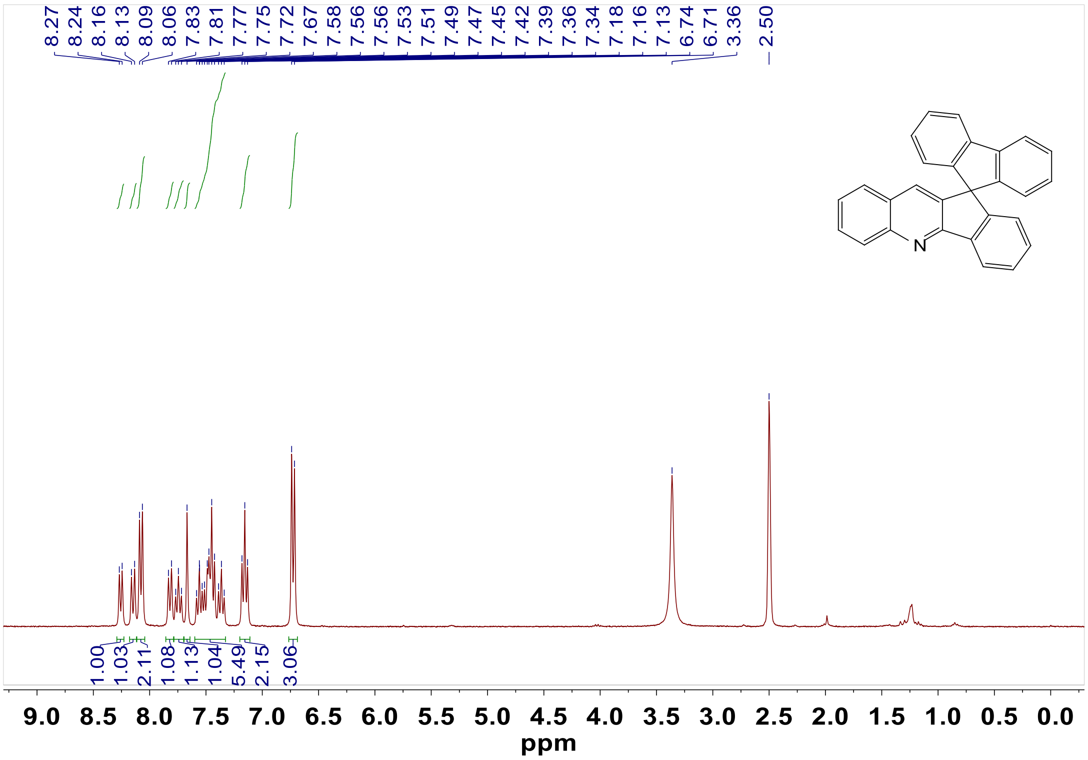


Fig S9. ^{1}H NMR of SFIQ

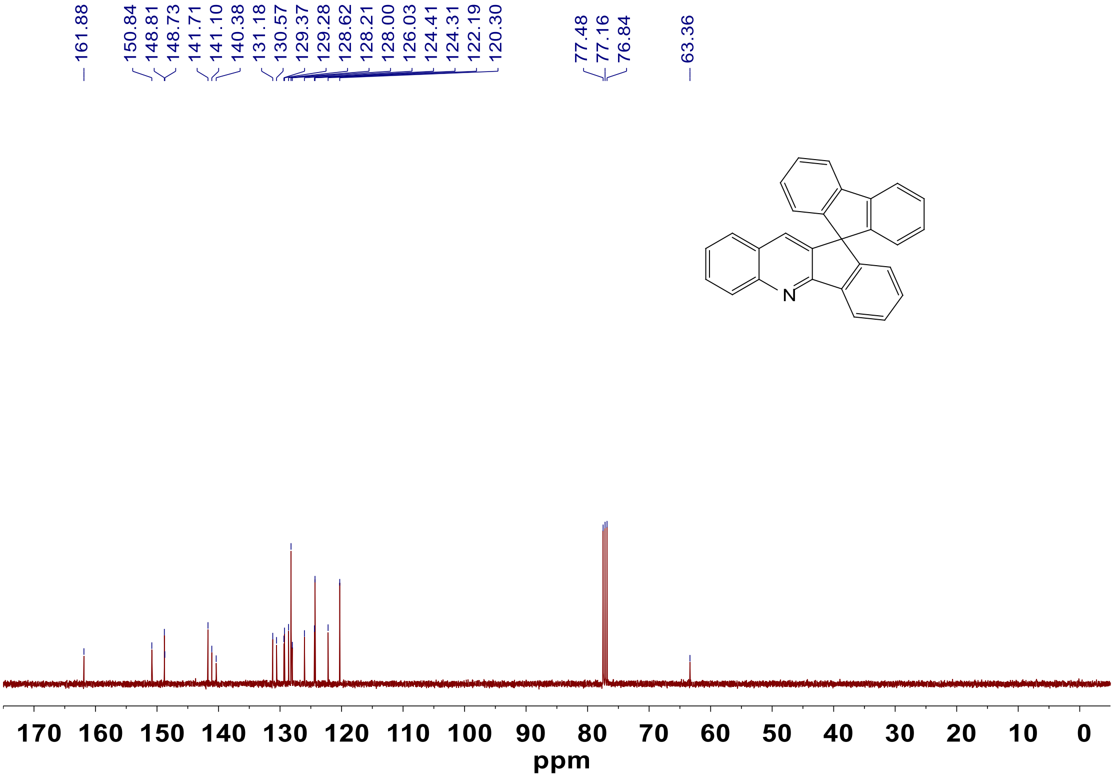


Fig S10. ^{13}C NMR of SFIQ

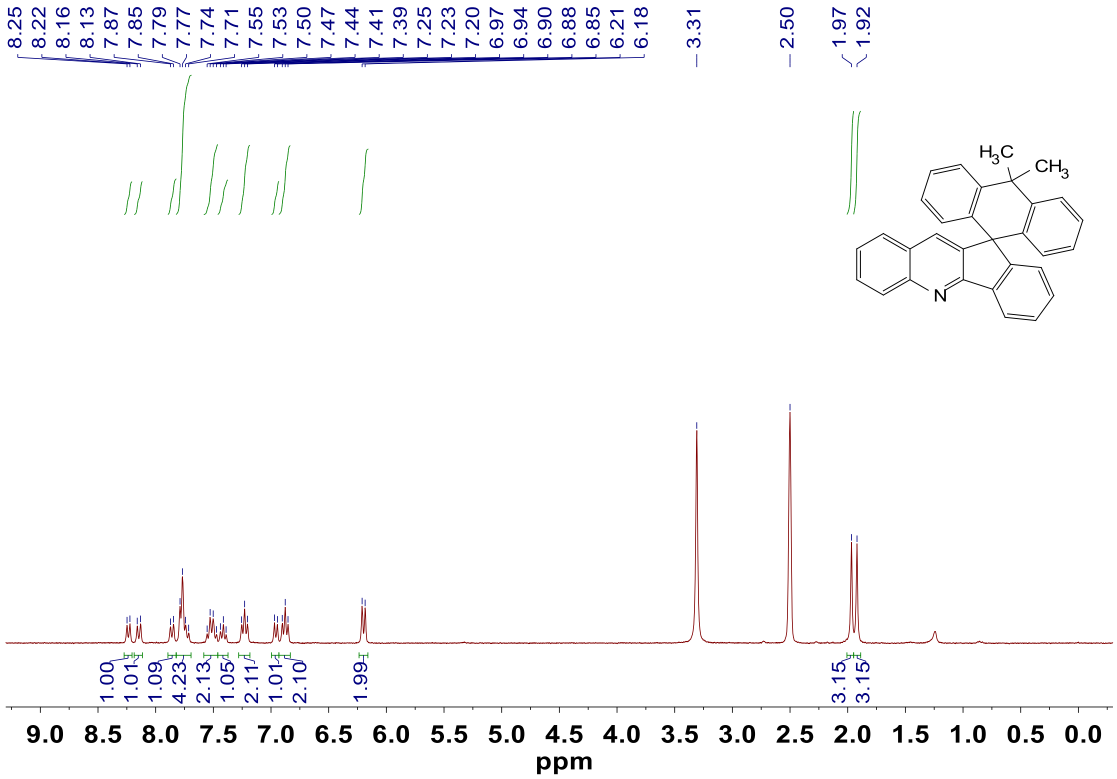


Fig S11.^{1}H NMR of DM-DHAIQ

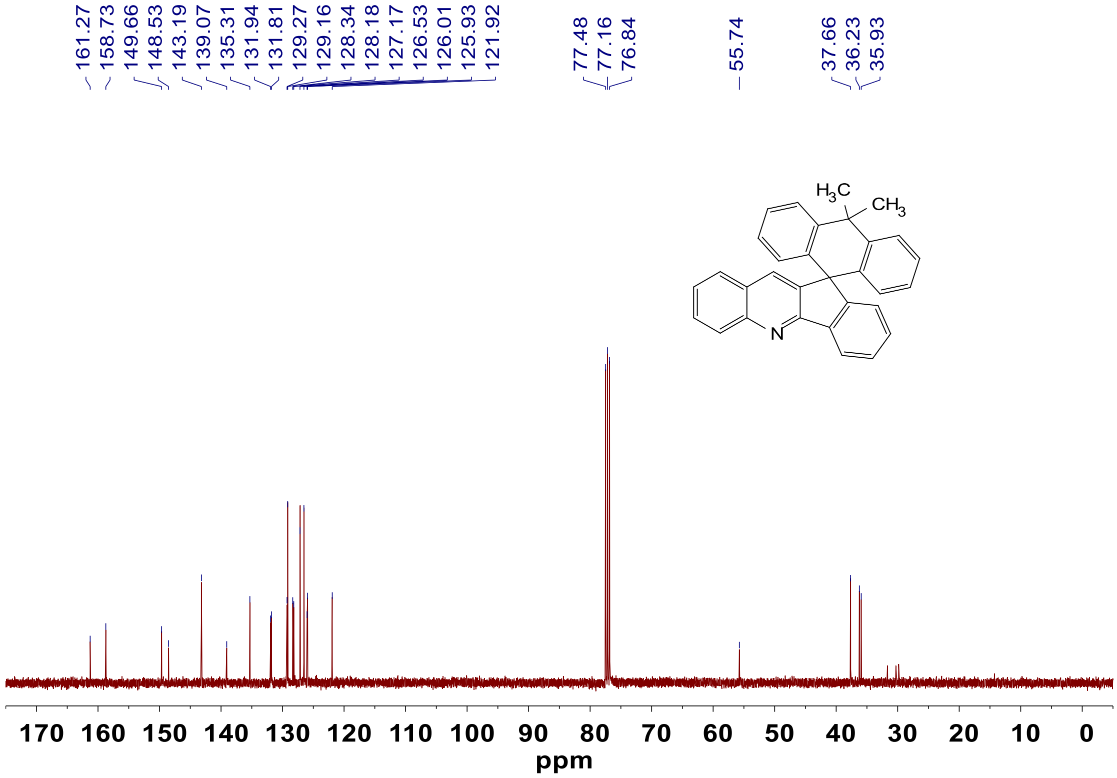


Fig S12. ^{13}C NMR of DM-DHAIQ

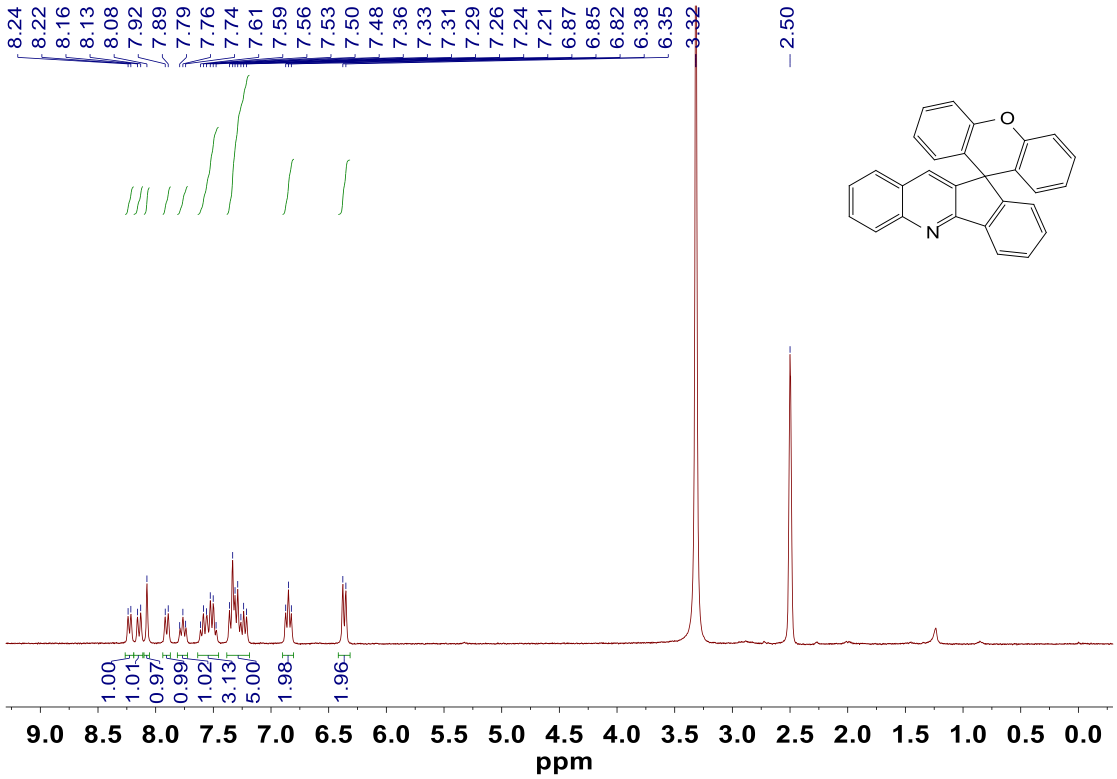


Fig S13.^{1}H NMR of SXIQ

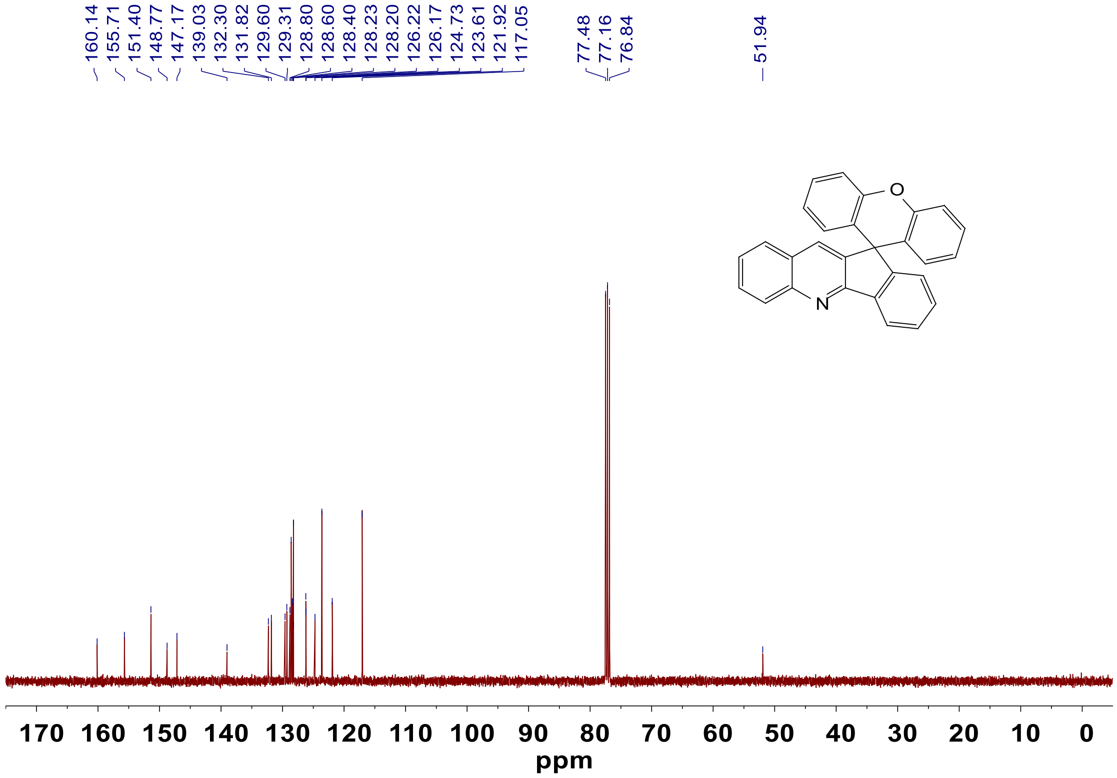


Fig S14. ^{13}C NMR of SXIQ

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