Electronic Supporting Information

# Colour tuning by the ring roundabout: [Ir(C^N)<sub>2</sub>(N^N)]<sup>+</sup> emitters with sulfonyl-substituted cyclometallating ligands

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#### General and ligand synthetic details

Microwave reactions were performed using a Biotage Initiator 8 reactor. <sup>1</sup>H and <sup>13</sup>C{<sup>1</sup>H} NMR spectra were recorded on a Bruker Avance III-500 spectrometer at 295 K; chemical shifts are referenced to residual solvent peaks with  $\delta$ (TMS) = 0 ppm. Electrospray ionization (ESI) and LC-ESI mass spectra were recorded on Bruker esquire 3000<sup>plus</sup> spectrometer and a combination of Shimadzu (LC) and Bruker AmaZon X instruments, respectively. FT-IR spectra were recorded on a Perkin Elmer Spectrum Two UATR instrument and absorption spectra on an Agilent 8453 spectrophotometer. Emission spectra in CH<sub>3</sub>CN solution were recorded on a Shimadzu 5301PC spectrofluorophotometer. Photoluminescence quantum yield measurements of solution and powder samples were carried out on a Hamamatsu absolute PL quantum yield spectrometer C11347 Quantaurus QY. Excited-state lifetimes of solution and powder samples as well as emission spectra of powder samples were recorded on a Hamamatsu Compact Fluorescence lifetime spectrometer C11367 Quantaurus Tau. Emission spectra in CH<sub>3</sub>CN solution at 77 K and in thin film were recorded with a Hamamatsu absolute PL quantum yield spectrometer C9920-02. The thin films have the same composition as the active layer in the LEC devices and were prepared from an MeCN solution of each complex (20 mg mL<sup>-1</sup>) mixed with 1-butyl-3methylimidazolium hexafluoridophosphate in 4:1 molar ratio by spin-coating on a quartz plate (1 cm<sup>2</sup>). Measurements in solution at 77K were obtained using a quartz Dewar designed by Hamamatsu adapted to the integrated sphere of the equipment. Electrochemical measurements were performed using cyclic voltammetry and square-wave voltammetry on a CH Instruments 900B potentiostat with glassy carbon working and platinum auxiliary electrodes; a silver wire was used as a pseudo-reference electrode. Dry, purified CH<sub>3</sub>CN was used as solvent and 0.1 M [<sup>n</sup>Bu<sub>4</sub>N][PF<sub>6</sub>] as supporting electrolyte. Ferrocene was used as internal reference and was added at the end of each experiment.

2-(1H-Pyrazol-1-yl)pyridine (pzpy)<sup>1,2</sup> and 2-(3,5-dimethyl-1H-pyrazol-1-yl)pyridine (dmpzpy)<sup>2,3</sup> and H1<sup>4</sup> were prepared using literature methods and <sup>1</sup>H NMR spectroscopic data matched those reported. [Ir(cod)Cl]<sub>2</sub> (cod = cycloocta-1,5-diene) was synthesized as described in the literature.<sup>5</sup> All reagents were used as received. 3-Fluorophenylboronic acid was purchased from Fluorochem and (purity ca. 67% determined by <sup>1</sup>H NMR spectroscopy). Silica was purchased from Fluka (silica gel 60, 0.040–0.063 mm).

2-(3-Fluorophenyl)pyridine. 3-Fluorophenylboronic acid (67%, 1.67 g, 8.00 mmol), 2-bromopyridine (0.500 mL, 5.22 mmol), PdCl<sub>2</sub> (25.2 mg, 0.142 mmol) and K<sub>2</sub>CO<sub>3</sub> (1.54 g, 11.1 mmol) were suspended in EtOH and  $H_2O$  (1 : 1 vol.). The mixture was heated at reflux overnight, then poured into brine (150 mL) and extracted with *t*-butyl methyl ether ( $3 \times 150$  mL). The combined organic layers were dried over Na<sub>2</sub>SO<sub>4</sub> and the solvent was removed under reduced pressure. The residue was purified by column chromatography (silica, toluene-ethyl acetate 5:1) to yield 2-(3fluorophenyl)pyridine as a colourless oil (764 mg, 4.41 mmol, 84.5%). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$ /ppm 8.66 (dt, J = 4.9, 1.3 Hz, 1H, H<sup>B6</sup>), 7.77–7.70 (overlapping m, 2H, H<sup>A2+A6</sup>), 7.69–7.62 (overlapping m, 2H, H<sup>B3+B4</sup>), 7.41– 7.35 (m, 1H,  $H^{A5}$ ), 7.18 (m, 1H,  $H^{B5}$ ), 7.07 (td, J = 8.4, 2.3 Hz, 1H,  $H^{A4}$ ). <sup>13</sup>C{<sup>1</sup>H} NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$ /ppm 163.3 (d,  $J_{CF}$  = 245.3 Hz, C<sup>A3</sup>), 155.9 (d,  $J_{CF} = 2.7$  Hz,  $C^{B2}$ ), 149.7 ( $C^{B6}$ ), 141.7 (d,  $J_{CF} = 7.6$  Hz,  $C^{A1}$ ), 136.8 ( $C^{B4}$ ), 130.2 (d,  $J_{CF}$  = 8.2 Hz, C<sup>A5</sup>), 122.6 (C<sup>B5</sup>), 122.4 (d,  $J_{CF}$  = 2.8 Hz, C<sup>A6</sup>), 120.5 (C<sup>B3</sup>), 115.7 (d,  $J_{CF}$  = 21.3 Hz, C<sup>A4</sup>), 113.8 (d,  $J_{CF}$  = 22.8 Hz, C<sup>A2</sup>). LC-ESI-MS *m*/*z* 174.0 [*M*+H]<sup>+</sup> (calc. 174.1). Found C 75.95, H 4.87, N 8.25; C<sub>11</sub>H<sub>8</sub>FN requires C 76.29, H 4.66, N 8.09%. Spectroscopic data matched those reported in the literature.<sup>6,7</sup>

2-(3-Methylthiophenyl)pyridine. A microwave vial was charged with 2-(3-fluorophenyl)pyridine (916 mg, 5.29 mmol) and N-methyl-2pyrrolidone (18 mL) and purged with N<sub>2</sub>. Sodium thiomethoxide (1.51 g, 21.5 mmol) was added and the dark pink mixture was heated at 120 °C for 1 h in a microwave reactor. The resulting pale brown mixture was poured into  $H_2O$  (150 mL) and brine (50 mL) and extracted with  $CH_2Cl_2$  (4 × 100 mL). The combined organic layers were washed with  $H_2O$  (4 × 100 mL), dried over Na<sub>2</sub>SO<sub>4</sub> and the solvent was removed under reduced pressure. The residue was purified by column chromatography (silica, toluene toluene-ethyl changing to acetate 1:1)to yield 2-(3methylthiophenyl)pyridine as a colourless liquid (826 mg, 4.10 mmol, 77.6%). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$ /ppm 8.70 (ddd, J = 4.8, 1.8, 1.0 Hz, 1H,

H<sup>B6</sup>), 7.92 (t, *J* = 1.9 Hz, 1H, H<sup>A2</sup>), 7.79–7.69 (overlapping m, 3H, H<sup>A6+B3+B4</sup>), 7.39 (t, *J* = 7.8 Hz, 1H, H<sup>A5</sup>), 7.31 (ddd, *J* = 7.8, 1.9, 1.1 Hz, 1H, H<sup>A4</sup>), 7.26– 7.23 (m, 1H, H<sup>B5</sup>), 2.56 (s, 3H, H<sup>Me</sup>). <sup>13</sup>C{<sup>1</sup>H} NMR (126 MHz, CDCl<sub>3</sub>) δ/ppm 157.1 (C<sup>B2</sup>), 149.8 (C<sup>B6</sup>), 140.2 (C<sup>A1</sup>), 139.3 (C<sup>A3</sup>), 136.9 (C<sup>B4</sup>), 129.3 (C<sup>A5</sup>), 127.2 (C<sup>A4</sup>), 125.2 (C<sup>A2</sup>), 123.8 (C<sup>A6</sup>), 122.5 (C<sup>B5</sup>), 120.8 (C<sup>B3</sup>), 16.0 (C<sup>Me</sup>). IR (solid,  $\nu/cm^{-1}$ ) 3051 (w), 3006 (w), 2986 (w), 2920 (w), 2857 (w), 1722 (w), 1584 (s), 1560 (s), 1457 (s), 1431 (s), 1400 (m), 1279 (m), 1261 (m), 1153 (m), 1096 (m), 1083 (m), 1066 (m), 1041 (m), 990 (m), 966 (m), 902 (w), 882 (m), 800 (m), 764 (s), 742 (s), 687 (s), 640 (m), 635 (m), 614 (m), 585 (m), 505 (w). LC-ESI-MS *m/z* 202.0 [*M*+H]<sup>+</sup> (calc. 202.1). Found C 71.15, H 5.88, N 6.78; C<sub>12</sub>H<sub>11</sub>NS requires C 71.60, H 5.51, N 6.96%.

2-(3-Methylsulfonylphenyl)pyridine (H2). 2-(3-Methylthiophenyl)pyridine (826 mg, 4.10 mmol) and sodium tungstate dihydrate (677 mg, 2.05 mmol) were suspended in MeOH (25 mL).  $H_2O_2$ (30%, 1.00 mL, 10.0 mmol) was added and the suspension stirred at room temperature overnight. The mixture was poured into a mixture of  $H_2O$ (150 mL) and saturated aqueous Na<sub>2</sub>CO<sub>3</sub> solution (50 mL) and extracted with  $CH_2Cl_2$  (3 × 100 mL). The combined organic layers were dried over Na<sub>2</sub>SO<sub>4</sub> and the solvent was removed under reduced pressure. The residue was purified by column chromatography (silica, CH<sub>2</sub>Cl<sub>2</sub>-1% MeOH changing to CH<sub>2</sub>Cl<sub>2</sub>-2% MeOH) to yield H2 as a white solid (786 mg, 3.37 mmol, 82.1%). M.p. 94.3 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ/ppm 8.74–8.68 (m, 1H,  $H^{B6}$ ), 8.57 (t, I = 1.9 Hz, 1H,  $H^{A2}$ ), 8.30 (m, 1H,  $H^{A4}$ ), 7.98 (ddt, I = 7.7, 2.0, 1.0 Hz, 1H, H<sup>A6</sup>), 7.84–7.75 (overlapping m, 2H, H<sup>B3+B4</sup>), 7.68 (td, *J* = 7.8, 0.9 Hz, 1H, H<sup>A5</sup>), 7.30 (dddd, *J* = 7.1, 4.9, 2.3, 0.8 Hz, 1H, H<sup>B5</sup>), 3.11 (s, 3H,  $H^{Me}$ ). <sup>13</sup>C{<sup>1</sup>H} NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$ /ppm 155.2 (C<sup>B2</sup>), 150.1 (C<sup>B6</sup>), 141.3 (C<sup>A3</sup>), 141.0 (C<sup>A1</sup>), 137.2 (C<sup>B4</sup>), 132.1 (C<sup>A4</sup>), 130.0 (C<sup>A5</sup>), 127.6 (C<sup>A6</sup>), 125.9 (C<sup>A2</sup>), 123.3 (C<sup>B5</sup>), 120.8 (C<sup>B3</sup>), 44.6 (C<sup>Me</sup>). IR (solid, v/cm<sup>-1</sup>) 3080 (w), 2997 (w), 2916 (w), 1586 (m), 1565 (w), 1458 (m), 1444 (m), 1408 (w), 1323 (m), 1297 (s), 1280 (m), 1145 (s), 1093 (m), 1066 (m), 992 (w), 975 (m), 916 (w), 886 (w), 818 (m), 780 (m), 754 (s), 680 (m), 638 (w), 614 (m), 593 (m), 539 (s), 511 (s), 486 (m). LC-ESI-MS m/z 234.0 [M+H]<sup>+</sup> (calc.

234.1). Found C 61.53, H 4.98, N 6.22; C<sub>12</sub>H<sub>11</sub>NO<sub>2</sub>S requires C 61.78, H 4.75, N 6.00%.

# Infrared spectroscopic data for iridium(III) complexes [Ir(2)<sub>2</sub>Cl]<sub>2</sub>

IR (solid, v/cm<sup>-1</sup>) 2922 (w), 1609 (m), 1577 (m), 1480 (m), 1423 (m), 1401 (w), 1298 (s), 1224 (w), 1146 (s), 1096 (m), 1068 (m), 1054 (m), 1032 (m), 959 (s), 887 (w), 827 (m), 784 (m), 761 (s), 728 (m), 703 (m), 639 (w), 593 (s), 558 (s), 522 (s), 486 (m), 463 (m).

## [Ir(1)<sub>2</sub>(pzpy)][PF<sub>6</sub>]

IR (solid, v/cm<sup>-1</sup>) 3121 (w), 1724 (w), 1611 (m), 1575 (w), 1524 (w), 1488 (m), 1477 (m), 1450 (w), 1430 (w), 1409 (w), 1375 (w), 1355 (w), 1309 (m), 1292 (m), 1143 (s), 1092 (m), 1063 (m), 1047 (w), 959 (m), 915 (w), 878 (w), 833 (s), 806 (m), 780 (m), 753 (s), 726 (m), 718 (m), 701 (m), 667 (m), 652 (m), 599 (m), 557 (s), 546 (s), 526 (m), 488 (m), 481 (m).

#### [Ir(1)<sub>2</sub>(dmpzpy)][PF<sub>6</sub>]

IR (solid, v/cm<sup>-1</sup>) 2924 (w), 1610 (m), 1578 (w), 1564 (w), 1475 (m), 1453 (m), 1431 (w), 1414 (w), 1392 (w), 1373 (m), 1320 (m), 1295 (m), 1268 (m), 1148 (s), 1094 (m), 1062 (w), 1038 (w), 994 (w), 957 (m), 874 (w), 836 (s), 805 (m), 782 (m), 756 (s), 749 (s), 701 (m), 666 (m), 652 (m), 593 (m), 557 (m), 547 (s), 523 (m), 481 (m).

#### [Ir(2)<sub>2</sub>(pzpy)][PF<sub>6</sub>]

IR (solid, v/cm<sup>-1</sup>) 3646 (w), 3133 (w), 1612 (m), 1578 (w), 1524 (w), 1481 (m), 1451 (w), 1409 (w), 1355 (w), 1299 (m), 1224 (w), 1165 (w), 1145 (s), 1111 (w), 1096 (w), 1067 (w), 1056 (m), 1033 (m), 960 (m), 837 (s), 781 (s), 758 (s), 716 (w), 703 (w), 639 (w), 593 (m), 556 (s), 524 (s), 478 (m).

## [Ir(2)<sub>2</sub>(dmpzpy)][PF<sub>6</sub>]

IR (solid, v/cm<sup>-1</sup>) 3086 (w), 2923 (w), 1609 (w), 1579 (w), 1538 (w), 1479 (m), 1451 (w), 1423 (w), 1396 (w), 1372 (w), 1323 (w), 1297 (s), 1285 (m), 1274 (w), 1227 (w), 1169 (w), 1144 (s), 1094 (m), 1077 (w), 1063 (w), 1055 (w), 1031 (w), 999 (w), 981 (w), 964 (m), 954 (m), 898 (w), 880 (w), 833 (s), 823 (s), 795 (m), 783 (m), 761 (s), 741 (m), 701 (m), 651 (w), 639 (w), 595 (m), 556 (s), 523 (m), 486 (m), 457 (w).

#### **Time-Dependent DFT calculations**

**Table S1.** Lowest triplet excited states calculated at the TD-DFT B3LYP/(6-31G\*\*+LANL2DZ) level for complexes  $[Ir(C^N)_2(N^N)]^+$  (C<sup>N</sup> = [1]<sup>-</sup> and [2]<sup>-</sup>, N^N = pzpy and dmpzpy) and  $[Ir(ppy)_2(pzpy)]^+$  in acetonitrile solution. Vertical excitation energies (*E*), dominant monoexcitations with contributions (within parentheses) greater than 18%, nature of the electronic transition and description of the excited state are summarized.

Complex	State	<i>E</i> (eV)	Monoexcitations	Nature	Description
[Ir(ppy) <sub>2</sub> (pzpy)] <sup>+</sup>	T <sub>1</sub>	2.766	$\mathrm{H} \rightarrow \mathrm{L+1}~(65)$	$d_{\pi}(Ir) + \pi_{main} \rightarrow \pi^*_{main}$	<sup>3</sup> LC/ <sup>3</sup> MLCT
	T <sub>2</sub>	2.812	H → L+2 (53) H-1→ L+1 (24)	$\begin{array}{c} d_{\pi}(Ir) + \pi_{main} \rightarrow \\ \pi^{*}_{main} \\ d_{\pi}(Ir) + \pi_{main} \rightarrow \\ \pi^{*}_{main} \end{array}$	<sup>3</sup> LC/ <sup>3</sup> MLCT
	<b>T</b> <sub>3</sub>	2.938	H→ L (97)	$d_{\pi}(Ir)$ + $\pi_{main} \rightarrow \pi^*_{anc}$	<sup>3</sup> MLCT/ <sup>3</sup> LLCT
[lr( <b>1</b> ) <sub>2</sub> (pzpy)] <sup>+</sup>	T <sub>1</sub>	2.716	$H \rightarrow L(69)$	$d_{\pi}(Ir) + \pi_{main} \rightarrow \pi^{*}_{main}$	<sup>3</sup> LC/ <sup>3</sup> MLCT
	<b>T</b> <sub>2</sub>	2.772	H→ L+2 (58)	$\begin{array}{c} d_{\pi}(Ir) \text{+} \pi_{main} \rightarrow \\ \pi^{*}_{main} \end{array}$	<sup>3</sup> LC/ <sup>3</sup> MLCT
	$T_3$	3.193	$H \rightarrow L+1$ (29)	$d_{\pi}(Ir)$ + $\pi_{main} \rightarrow \pi^*_{anc}$	<sup>3</sup> MLCT/ <sup>3</sup> LLCT
$[Ir(1)_2(dmpzpy)]^+$	T <sub>1</sub>	2.712	$H \rightarrow L(69)$	$d_{\pi}(Ir)$ + $\pi_{main} \rightarrow \pi^{*}_{main}$	<sup>3</sup> LC/ <sup>3</sup> MLCT
	T <sub>2</sub>	2.770	$\mathrm{H} \rightarrow \mathrm{L+1}~(53)$	$d_{\pi}(Ir) + \pi_{main} \rightarrow \pi^{*}_{main}$	<sup>3</sup> LC/ <sup>3</sup> MLCT
	<b>T</b> <sub>5</sub>	3.294	$\mathrm{H} \rightarrow \mathrm{L+2} \ (80)$	$d_{\pi}(Ir)$ + $\pi_{main} \rightarrow \pi^*_{anc}$	<sup>3</sup> MLCT/ <sup>3</sup> LLCT
[Ir( <b>2</b> ) <sub>2</sub> (pzpy)] <sup>+</sup>	T <sub>1</sub>	2.879	$\mathrm{H} \rightarrow \mathrm{L+1}~(52)$	$d_{\pi}(Ir)$ + $\pi_{main} \rightarrow \pi^{*}_{main}$	<sup>3</sup> LC/ <sup>3</sup> MLCT
	<b>T</b> <sub>2</sub>	2.918	H→ L+2 (44)	$\begin{array}{c} d_{\pi}(Ir) \text{+} \pi_{main} \rightarrow \\ \pi^{*}_{main} \end{array}$	<sup>3</sup> LC/ <sup>3</sup> MLCT
	$T_3$	3.283	$H \rightarrow L (82)$	$d_{\pi}(Ir)$ + $\pi_{main} \rightarrow \pi^*_{anc}$	<sup>3</sup> MLCT/ <sup>3</sup> LLCT
[Ir( <b>2</b> ) <sub>2</sub> (dmpzpy)] <sup>+</sup>	T <sub>1</sub>	2.876	$H \rightarrow L (53)$	$d_{\pi}(Ir) + \pi_{main} \rightarrow \pi^*_{main}$	<sup>3</sup> LC/ <sup>3</sup> MLCT
	<b>T</b> <sub>2</sub>	2.914	$\mathrm{H} \rightarrow \mathrm{L+2} \ (40)$	$d_{\pi}(Ir) + \pi_{main} \rightarrow \pi^*_{main}$	<sup>3</sup> LC/ <sup>3</sup> MLCT
	<b>T</b> <sub>3</sub>	3.341	$\mathrm{H}\rightarrow\mathrm{L+1}\ (50)$	$d_{\pi}(Ir)$ + $\pi_{main} \rightarrow \pi^*_{anc}$	<sup>3</sup> MLCT/ <sup>3</sup> LLCT

#### **Electroluminescent devices**



**Fig. S1** Luminance (solid blue line) and average voltage (open red squares) versus time for ITO/PEDOT:PSS/[Ir()<sub>2</sub>(pzpy)][PF<sub>6</sub>]:[Bmim][PF<sub>6</sub>] 4:1/Al under a constant pulsed current (1000 Hz, 50% duty cycle and block wave) of 100 A m<sup>-2</sup> (average current density). [Bmim][PF<sub>6</sub>] = 1-butyl-3-methylimidazolium hexafluorophosphate.



**Fig. S2** Luminance (solid blue line) and average voltage (open red squares) versus time for ITO/PEDOT:PSS/[Ir(1)<sub>2</sub>(dmpzpy)][PF<sub>6</sub>]:[Bmim][PF<sub>6</sub>] 4:1/Al under a constant pulsed current (1000 Hz, 50% duty cycle and block wave) of 100 A m<sup>-2</sup> (average current density). [Bmim][PF<sub>6</sub>] = 1-butyl-3-methylimidazolium hexafluorophosphate.



**Fig. S3** Luminance (solid blue line) and average voltage (open red squares) versus time for ITO/PEDOT:PSS/[Ir(**2**)<sub>2</sub>(pzpy)][PF<sub>6</sub>]:[Bmim][PF<sub>6</sub>] 4:1/Al under a constant pulsed current (1000 Hz, 50% duty cycle and block wave) of 100 A m<sup>-2</sup> (average current density). [Bmim][PF<sub>6</sub>] = 1-butyl-3-methylimidazolium hexafluorophosphate.



**Fig. S4** Luminance (solid blue line) and average voltage (open red squares) versus time for ITO/PEDOT:PSS/[Ir(2)<sub>2</sub>(dmpzpy)][PF<sub>6</sub>]:[Bmim][PF<sub>6</sub>] 4:1/Al under a constant pulsed current (1000 Hz, 50% duty cycle and block wave) of 100 A m<sup>-2</sup> (average current density). [Bmim][PF<sub>6</sub>] = 1-butyl-3-methylimidazolium hexafluorophosphate.

#### Photoluminescent spectra in thin film



**Fig. S5** Photoluminescent spectra of complexes  $[Ir(C^N)(N^N)][PF_6]$  (C<sup>N</sup>=[**1**]<sup>-</sup> and [**2**]<sup>-</sup>, N^N = pzpy and dmpzpy) in thin film (complex:[Bmim][PF<sub>6</sub>] 4:1 on quartz plate). [Bmim][PF<sub>6</sub>] = 1-butyl-3-methylimidazolium hexafluoridophosphate.

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