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# Phosphorescent cationic iridium(III) complexes with cyclometalating 1H-indazole and 2H-[1,2,3]-triazole ligands

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# ARTICLE INFO

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# 1. Introduction

Phosphorescent Ir(III) complexes are actively developed for next-generation lighting, display, and sensor applications [1–4]. The electronic and photophysical properties of these complexes can be tuned by variation of the organic ligands [1–3]. Pyridine derivatives are commonly used as ligands in Ir(III) coordination chemistry [1–4]. Azoles are versatile and easy-to-make alternatives to pyridines [5,6], and several research groups are developing Ir–azole complexes [7–15]. Here, we report phosphorescent cationic Ir(III) complexes [(C^N)<sub>2</sub>Ir(4,4'-di-tert-butyl-2,2'-dipyridyl)](PF<sub>6</sub>) (**1** and **2**, Scheme 1) with less-common cyclometalating ligands that have 1H-indazole or 2H-[1,2,3]-triazole heterocycles [16,17], and we discuss the influence of aryl ring fusion (**1**) or heteroatom addition (**2**) on the properties of the known analogs **3** (Chart 1) [8].

# 2. Results and discussion

Three ligands, **L1–L3**, were prepared by  $Cu_2O$ -catalyzed C–N coupling of aryl halides with azoles (Scheme 2) [18]. For indazole, only the N1-derivative (**L1**), which is the major product, was isolated. For 1H-[1,2,3]-triazole, the reaction gave N2- and N1-derivatives **L2** and **L3** in equal yields after separation by column chromatography.

# ABSTRACT

Two new cationic iridium(III) complexes with cyclometalating 1-phenylindazole or 2-phenyl-1,2,3-triazole ligands,  $[(C^N)_2 Ir(4,4'-di-tert-butyl-2,2'-dipyridyl)](PF_6)$ , exhibit yellow or green phosphorescence with quantum yields and excited state lifetimes of up to 45% and 840 ns in argon-saturated dichloromethane solution at room temperature.

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The reaction of **L1** or **L2** with  $IrCl_3 \cdot 3H_2O$  in 2-ethoxyethanol/ water gave cyclometalated complexes  $[(C^N)_2 Ir(\mu-Cl)]_2$  (Scheme 1) [19]. However, this reaction failed for **L3** probably because its N3-atom coordinates to Ir(III) to give a mixture of complexes [12,13].

Reaction of  $[(C^N)_2 Ir(\mu-Cl)]_2$  with 4,4'-di-tert-butyl-2,2'-dipyridyl (bpy\*) gave two new cationic Ir(III) complexes **1** and **2** (Scheme 1) as air- and moisture-stable solids that are soluble in polar organic solvents. We chose a bulky neutral bpy\* ligand because it reduces solid state interaction and increases solubility of metal complexes. **1** and **2** were purified by column chromatography and re-crystallization, and were characterized by C, H, N elemental analysis. <sup>1</sup>H and <sup>13</sup>C NMR spectra of **1** and **2** show a single set of signals for the constituent ligands and indicate that the complexes have  $C_2$ -symmetry in solution. Integration of <sup>1</sup>H peaks confirms the 2:1 ratio of the C^N to bpy\*; <sup>19</sup>F NMR confirms the presence of PF<sub>6</sub><sup>-</sup> anion; and ESI\* TOF mass-spectra show the peak of a cation  $[(C^N)_2 Ir(bpy*)]^*$ .

Figs. 1 and 2 display the X-ray structures of **1** and **2**. The Ir(III) ion is in a distorted octahedral  $[(C^N)_2 Ir(N^N)]^+$  coordination environment with the two nitrogen atoms of the C^N ligands in trans-position to each other. The Ir–N bonds to the anionic C^N ligand are shorter than those to the neutral bpy\* by  $\approx 0.1$  Å (Table 1). The ligands are nearly planar: the dihedral angle between their rings is  $3-5^{\circ}$  (bpy\*) or  $8-13^{\circ}$  (C^N) (Table 1). The largest angle is observed for the C^N ligands of **1**: it probably prevents steric clash between the hydrogens of phenyl (2'-H) and indazole (7-H).



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Chart 1. Previously reported Ir(III) complexes [8].



**Scheme 1.** Synthesis of new Ir(III) complexes: (a) cyclometalating ligand, 2-ethoxyethanol/water (3/1), under argon,  $120 \degree$ C; (b) bpy\*, under argon,  $40 \degree$ C, CH<sub>2</sub>Cl<sub>2</sub>/methanol.



**Scheme 2.** Synthesis of the ligands: (a) iodobenzene,  $Cs_2CO_3$ ,  $Cu_2O$  (catalyst), dry DMF, under argon, 120 °C.

The complex **1** contains a  $C_2$ -symmetry axis and the two C^N ligands are equivalent as are the two 4-tert-butylpyridine rings of bpy\*. In **1**, the 'large surface' indazole rings of both C^N ligands in each complex participate in a face-to-face  $\pi$ - $\pi$  stacking with indazole rings of their neighbors at inter-planar distance of 3.344 Å. The net result is the formation of a column of molecules of **1** that are connected through  $\pi$ - $\pi$  interactions. In **2**, only one of the C^N ligands in each complex takes part in a weak  $\pi$ - $\pi$  stacking with a C^N ligand of its neighbor at inter-planar distance of 3.503 Å. The bulky bpy\* in **1** or **2** is not involved in  $\pi$ - $\pi$  stacking. Inter-metallic communication is likely to be negligible because of the long Ir–Ir distances (>8 Å, Table 1). The main structural parameters of **1** and **2** are similar to those of **3H** [8].

Redox potentials of **1** and **2** were measured by cyclic voltammetry relative to  $Fc^*/Fc$  [20] in acetonitrile and DMF (Fig. 3, Table 2, and Figs. S1 and S2, Supporting information). **1** and **2** show two reduction processes: the first one, at -1.8 V, is reversible; the



**Fig. 1.** Structure of **1** (CCDC 863360; 50% probability ellipsoids; H atoms, PF<sub>6</sub> anion, co-crystallized acetone molecule, and the disorder within the tert-butyl group are omitted; ORTEP). Heteroatoms are shown as octant ellipsoids: Ir, black; N, clear.



Fig. 2. Structure of 2 (CCDC 863361; 50% probability ellipsoids; H atoms and  $PF_6$  anion are omitted; ORTEP). Heteroatoms are shown as octant ellipsoids: Ir, black; N, clear.

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Selected	structural	parameters	of Ir(III)	complexes.

Complex	Bond lengths (Å)			Angles (°) <sup>b</sup>		Ir–Ir (Å) <sup>c</sup>
	C^N		bpy*	C^N	bpy*	
	Ir–C	Ir–N	Ir-N <sup>d</sup>			
<b>1</b> <sup>e</sup>	2.011(7)	2.026(6)	2.131(7)	13.00	4.98	8.050(2)
2	2.025(3) 2.042(3)	2.026(2) 2.026(2)	2.125(2) 2.138(2)	8.94 7.88	2.76	8.236(1)
	( )					

<sup>a</sup> Each row corresponds to one ligand in the complex.

<sup>b</sup> The dihedral angle between the constituent rings of the ligands.

<sup>c</sup> The shortest Ir–Ir distance in the structure.

<sup>d</sup> N-atom of bpy\* trans to C-atom of C^N in the same row.

<sup>e</sup> **1** contains a *C*<sub>2</sub>-symmetry axis.

second one, at -2.5 V, is irreversible. Both processes are likely to be bpy\*-centered [21] because their potentials show less than 40 mV variation with the change of the C^N ligand.

The complexes display a quasi-reversible or irreversible oxidation process that probably takes place on Ir–aryl fragment because its potential is sensitive to the nature of the cyclometalating ligand



**Fig. 3.** Cyclic voltammograms of **1** and **2** in DMF (0.1 M NBu<sub>4</sub>PF<sub>6</sub>, 100 mV/s). The potential range is limited to the first reduction and oxidation processes. The unit on the vertical axis is 5 uA. Additional CVs are shown in the Supporting information.

Table 2

Redox properties of Ir(III) complexes.<sup>a</sup>

Complex	Solvent	$E_{1/2}^{ox}(V)$	$E_{1/2}^{\rm red}$ (V)	$\Delta E (V)^{b}$
1 <sup>c</sup> 2	DMF CH₃CN DMF	0.83 (112) <sup>d</sup> 1.23 <sup>e</sup> f	-1.85 (78), -2.54 <sup>e</sup> -1.82 (78), -2.48 <sup>e</sup> -1.81 (88), -2.51 <sup>e</sup>	2.68 3.05
3H <sup>g</sup> 3F <sup>g</sup>	CH₃CN CH₃CN	0.95 1.25	-1.89 -1.83	2.84 3.08

<sup>a</sup> Relative to  $Fc^+/Fc$ . On glassy carbon working electrode, in the presence of 0.1 M (NBu<sub>4</sub>)PF<sub>6</sub>, at scan rate 100 mV/s. Estimated error: ±50 mV. The anodic/cathodic peak separation for the reversible processes is given in brackets (for the standard, Fc<sup>+</sup>/Fc couple, it was 73–83 mV).

<sup>b</sup>  $\Delta E = E_{1/2}^{ox} - E_{1/2}^{red}$ 

<sup>c</sup> 1 has low solubility in CH<sub>3</sub>CN (CV not recorded).

<sup>d</sup> Quasi-reversible process; the anodic/cathodic peak separation is given in brackets.

<sup>e</sup> Irreversible process; the oxidation or reduction peak potential is reported.

 $^{\rm f}$  Oxidation was outside of the electrochemical window of DMF (>1.0 V relative to Fc\*/Fc).

<sup>g</sup> Data from the literature [8].

and increases by 400 mV in the order 1 < 2 (Table 2). The variation of oxidation potentials in 1-3 shows that the C^N ligands become less electron-rich and more electron-withdrawing in the order L1 < 1-phenylpyrazole [8] < L2  $\approx$  1-(2',4'-difluorophenyl)pyrazole [8] (Table 2).

Electrochemical gap,  $\Delta E = E_{1/2}^{\text{ox}} - E_{1/2}^{\text{red}}$ , in **1** and **2** is 2.68 and 3.05 V, respectively. It is of note that the same effect on the redox properties of **3H** [8] is achieved by replacing either pyrazole with 2H-[1,2,3]-triazole (**2**) or phenyl with 2',4'-difluorophenyl (**3F**, [8]): in both cases, the changes in potentials are almost identical and  $\Delta E$  increases by  $\approx 0.2$  V (Table 2).



**Fig. 4.** Absorption spectra of **1**  $(7.26 \times 10^{-5} \text{ M})$  and **2**  $(8.46 \times 10^{-5} \text{ M})$  in CH<sub>2</sub>Cl<sub>2</sub>. Additional absorption spectra are shown in the Supporting information.

#### Table 3

Optical absorption of Ir(III) complexes.<sup>a</sup>

Complex	$\lambda_{\rm abs}/\rm{nm}~(\epsilon/10^3~M^{-1}~cm^{-1})$	$\lambda_{onset}/nm^{b}$
1	271 (46), 297 (35), 309 (36), 365 (9.6, sh), 429 (0.7, sh)	530
2	261 (41), 296 (24), 306 (22, sh), 346 (6.8, sh)	490

<sup>a</sup> In CH<sub>2</sub>Cl<sub>2</sub>, at room temperature, in the range 250–600 nm. Estimated errors:  $\pm 2$  nm for  $\lambda_{abs}$ ;  $\pm 5$  nm for  $\lambda_{onser}$ ;  $\pm 5\%$  for  $\varepsilon$ .

<sup>b</sup> Defined as a wavelength above which  $\varepsilon < 0.01\% \varepsilon_{max}$ .



**Fig. 5.** Corrected and normalized emission spectra of **1** and **2** at  $10^{-5}$  M in argonsaturated dichloromethane at room temperature ( $\lambda_{exc} = 360$  nm;  $\Delta \lambda_{em} = 1$  nm).

#### Table 4

Photophysical properties of Ir(III) complexes.<sup>a</sup>

Complex	$\lambda_{\rm em}/\rm nm$	$\Phi$ /%	τ/ns	$\tau_{rad}/\mu s$
1	575	24	640	2.7
2	520	45	840	1.9

<sup>a</sup> In argon-saturated CH<sub>2</sub>Cl<sub>2</sub> at 10<sup>-5</sup> M at room temperature. Estimated errors: ±2 nm for  $\lambda_{em}$ ; ±15% for  $\Phi$ ; ±20 ns for  $\tau$ .

**1** and **2** are pale yellow solids; in solution they exhibit a broad band in the visible range at  $\lambda > 400$  nm with molar absorption coefficients ( $\varepsilon$ ) of <10<sup>3</sup> M<sup>-1</sup> cm<sup>-1</sup> (Fig. 4, Table 3, and Figs. S3 and S4, Supporting information). The absorption onset of **1** and **2** is observed at 530 and 490 nm, respectively (Table 3): the lower  $\lambda_{onset}$  corresponds to the higher value of the electrochemical gap (Table 2). We assign the lowest energy transition in **1** and **2** to (Ir-phe-nyl)-to-bpy\* charge transfer, and we explain its weak intensity by the poor overlap of the participating molecular orbitals [8].

The intense near-UV bands of **1** and **2** at  $\lambda < 400$  nm with  $\varepsilon = (7-46) \times 10^3 \text{ M}^{-1} \text{ cm}^{-1}$  likely originate from Ir-to-bpy\* charge-transfer and ligand  $\pi$ - $\pi$ \* transitions (Table 3; Figs. S3 and S4, Supporting information).

**1** and **2** are phosphorescent at ambient conditions and exhibit broad emission band in  $10^{-5}$  M deoxygenated (argon-saturated) dichloromethane solutions at room temperature (Fig. 5): the phosphorescence color is yellow (**1**) or pale green (**2**). The excitation spectra of **1** and **2** match the absorption spectra (Fig. 55, Supporting information). The luminescence decays are single exponential functions suggesting the presence of one emissive center in solution (Fig. S6, Supporting information). The emission maxima ( $\lambda_{\rm em}$ ), luminescence quantum yields ( $\Phi$ ), observed excited-state lifetimes ( $\tau$ ), and calculated radiative lifetimes of the excited states ( $\tau_{\rm rad} = \tau/\Phi$ ) of **1** and **2** are reported in Table 4.

With respect to **3H** ( $\lambda_{em}$  = 538 nm in CH<sub>2</sub>Cl<sub>2</sub>, [8]), aryl ring fusion in **1** red-shifts the phosphorescence spectrum, while heteroatom addition in **2** blue-shifts it (Table 4). The quantum efficiencies and lifetimes of **1** and **2** are comparable to those of the reported analogs [8,15]. The broad phosphorescence spectra

and relatively short radiative lifetimes suggest that **1** and **2** emit from a charge-transfer excited state, probably, of (Ir–phenyl)-to-bpy\* nature [8].

In conclusion, cyclometalating ligands with 1H-indazole or 2H-[1,2,3]-triazole heterocycles provide Ir(III) complexes that are brightly-phosphorescent in solutions. The large electrochemical gap and blue-shift of phosphorescence spectrum of **2** with respect to **3H** [8] suggest that 2H-[1,2,3]-triazole can be used as a building block to develop high-energy emitting Ir(III) complexes.

# 3. Experimental

The following data are provided in the Supporting information: general methods, equipment, and chemicals used; synthesis and characterization of **L1–L3** and  $[(C^N)_2 Ir(\mu-CI)]_2$ ; <sup>1</sup>H, <sup>13</sup>C, and <sup>19</sup>F NMR spectra; experimental details for the X-ray, electrochemical, and spectroscopic measurements; crystallographic data (Table S1); cyclic voltammograms (Figs. S1 and S2); electronic absorption spectra (Table S2 and Figs. S3 and S4); phosphorescence excitation spectra (Fig. S5); and luminescence decays (Fig. S6).

Purification, crystal growth, and handling of all compounds were carried out under air. All products were stored in the dark. Chemicals from commercial suppliers were used without purification. Chromatography was performed on a column with an i.d. of 30 mm on silica gel 60 (Fluka, Nr 60752). The progress of reactions and the elution of products were followed on TLC plates (silica gel 60  $F_{254}$  on aluminum sheets, Merck).

# 3.1. Synthesis of the complexes

The structures of **1** and **2** are shown in Scheme 1. The reactions were performed under argon and in the absence of light. The solvents were de-oxygenated by bubbling with Ar, but they were not dried.  $[(C^N)_2 Ir(\mu-CI)]_2$  was dissolved in a mixture of  $CH_2 CI_2$ (40 mL) and CH<sub>3</sub>OH (5 mL) at RT, and 4,4'-di-tert-butyl-2,2'-dipyridyl (bpy\*; excess, Aldrich) was added. The reaction mixture was stirred at 40 °C overnight to give yellow solution. It was evaporated to dryness. The impurities were removed by chromatography (silica, 15 g) by eluting with 2% (1) or 3-5% (2) CH<sub>3</sub>OH in CH<sub>2</sub>Cl<sub>2</sub>. The product was recovered as yellow fraction by eluting with 4-6% (1) or 5–7% (2) CH<sub>3</sub>OH in CH<sub>2</sub>Cl<sub>2</sub>. The fractions containing the product were evaporated. Dry residue was dissolved in CH<sub>3</sub>OH (5 mL) and added drop-wise to a stirred aqueous solution of KPF<sub>6</sub> (670 mg in 30 mL of water, 3.64 mmol, excess, Alfa Aesar). The resulting suspension was stirred for 10 min and filtered. The complex was washed with water and either ether (1) or hexane (2). This workup gave the pure 1. In contrast, 2 had to be additionally re-crystallized by pouring its solution in  $CH_2Cl_2$  (3.5 mL) to ether (25 mL): the precipitate was filtered and washed with ether. 1 and 2 are air- and moisture-stable solids. Further details are provided below.

**1**: [(**L1**)<sub>2</sub>Ir(μ-Cl)]<sub>2</sub> (100 mg, 0.081 mmol) and bpy<sup>\*</sup> (55 mg, 0.20 mmol) gave pale yellow solid that emits yellow phosphorescence in solution and as a powder: 107 mg (0.108 mmol, 67%). *Anal.* Calc. for C<sub>44</sub>H<sub>42</sub>F<sub>6</sub>IrN<sub>6</sub>P (MW 992.03): C, 53.27; H, 4.27; N, 8.47. Found: C, 53.37; H, 4.55; N, 8.45%. <sup>1</sup>H NMR (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>):  $\delta$  = 8.34 (d, *J* = 1.6 Hz, 2H), 8.28 (d, *J* = 8.8 Hz, 2H), 8.13 (d, *J* = 5.6 Hz, 2H), 7.92 (dd, *J* = 8.0, 0.8 Hz, 2H), 7.77 (d, *J* = 8.0 Hz, 2H), 7.70–7.64 (m, 2H), 7.53–7.47 (m, 4H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.24–7.17 (m, 2H), 6.83 (td, *J* = 7.6, 0.8 Hz, 2H), 6.32 (dd, *J* = 7.6, 1.2 Hz, 2H), 1.46 (s, 18H, tert-butyl) ppm. <sup>13</sup>C NMR (100 MHz, CD<sub>2</sub>Cl<sub>2</sub>): all of the expected signals in aromatic (18C) and aliphatic (2C) regions were observed,  $\delta$  = 164.55, 156.04, 150.57, 144.62, 138.03, 134.36, 132.66, 131.79, 130.07, 125.40, 125.37, 124.72, 123.64, 123.08, 121.78, 120.94, 111.70, 110.58, 35.68 [*C*(CH<sub>3</sub>)<sub>3</sub>],

30.01 (CH<sub>3</sub>) ppm. <sup>19</sup>F NMR (376 MHz, CD<sub>2</sub>Cl<sub>2</sub>):  $\delta$  = -73.43 (d,  $J_{P-F}$  = 710 Hz, PF<sub>6</sub>) ppm. ESI<sup>+</sup> TOF MS: m/z 847.3 ({M-PF<sub>6</sub>}<sup>+</sup>, 100%).

**2**:  $[(L2)_2 Ir(\mu-Cl)]_2$  (100 mg, 0.097 mmol) and bpy\* (65 mg, 0.24 mmol) gave pale yellow-green solid: 89 mg (0.10 mmol, 51%). It emits pale green phosphorescence in solution; the emission in the powder is very weak. *Anal.* Calc. for  $C_{34}H_{36}F_6 IrN_8P$  (MW 893.88): C, 45.68; H, 4.06; N, 12.54. Found: C, 45.70; H, 4.38; N, 12.35%. <sup>1</sup>H NMR (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>):  $\delta = 8.31$  (s, 2H), 8.06 (d, *J* = 6.0 Hz, 2H), 8.00 (s, 2H), 7.79 (dd, *J* = 8.0, 1.2 Hz, 2H), 7.52 (dd, *J* = 6.0, 1.6 Hz, 2H), 7.23 (s, 2H), 7.20 (dd, *J* = 8.0, 1.2 Hz, 2H), 7.05 (td, *J* = 7.2, 1.2 Hz, 2H), 6.34 (dd, *J* = 7.6, 0.8 Hz, 2H), 1.46 (s, 18H, tert-butyl) ppm. <sup>13</sup>C NMR (100 MHz, CD<sub>2</sub>Cl<sub>2</sub>): all of the expected signals in aromatic (13C) and aliphatic (2C) regions were observed,  $\delta = 165.05$ , 156.14, 150.99, 141.22, 136.12, 133.42, 132.34, 129.07, 128.90, 125.58, 124.12, 121.15, 114.83, 35.72 [C(CH<sub>3</sub>)<sub>3</sub>], 29.95 (CH<sub>3</sub>) ppm. <sup>19</sup>F NMR (376 MHz, CD<sub>2</sub>Cl<sub>2</sub>):  $\delta = -73.30$  (d, *J*<sub>P-F</sub> = 710 Hz, PF<sub>6</sub>) ppm. ESI<sup>+</sup> TOF MS: *m/z* 749.3 ({M-PF<sub>6</sub>}<sup>+</sup>, 100%).

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# Appendix A. Supplementary material

CCDC 863360 and 863361 contain the supplementary crystallographic data for **1** and **2**, respectively. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data\_request/cif. Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.ica.2012.03.008.

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