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# Blue and Green Phosphorescent Liquid-Crystalline Iridium Complexes with High Hole Mobility

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**Abstract:** Blue- and green-emitting cyclometalated liquid-crystalline iridium complexes are realized by using a modular strategy based on strongly mesogenic groups attached to an acetylacetonate ancillary ligand. The cyclometalated ligand dictates the photophysical properties of the materials, which are identical to those of the parent complexes. High hole mobilities, up to  $0.004 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ , were achieved after thermal annealing, while amorphous materials show hole mobilities of only  $\sim 10^{-7}$ – $10^{-6} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ , in common with simple iridium complexes. The design strategy allows for the facile preparation of phosphorescent liquid-crystalline complexes with fine-tuned photophysical properties.

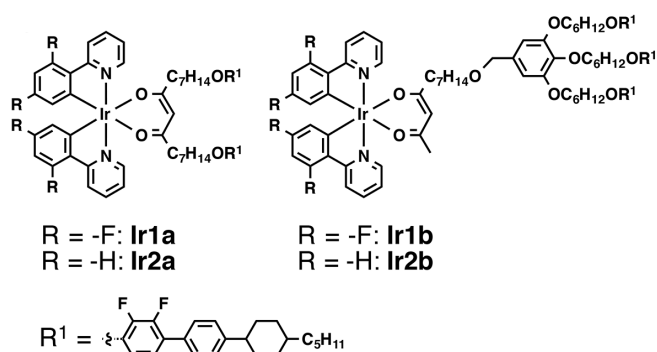
Cyclometalated iridium complex are attracting a lot of interest as phosphorescent emitters because of their generally high luminescence efficiency and the possibility to tune their emission across the entire UV-visible spectrum by simple chemical modifications of the ligands.<sup>[1–2]</sup> In addition to imaging and sensing applications, the possibility to obtain electroluminescent (EL) devices with 100% internal quantum efficiency has been a fascinating driving force for countless studies.<sup>[3]</sup>

In EL devices, these complexes are generally employed as amorphous materials, that is without particular molecular order in the bulk. The provision of molecular order would, however, give appealing properties to these materials. Indeed molecular orientation and ordering in organic semiconductors play very important roles in electrical (e.g. charge mobility) and optical (e.g. out-coupling) properties, vital to improving the performance of devices.<sup>[4–7]</sup> Current research focuses mainly on improving the molecular order via heat treatment of amorphous films.<sup>[8]</sup>

An enticing approach to provide and control molecular order in the bulk is to confer liquid-crystalline properties on the materials. Molecular order in liquid-crystalline phases can greatly improve the charge carrier mobility in organic semiconductor molecules.<sup>[9]</sup> In addition, liquid-crystalline materials have attractive potential use for polarized OLEDs<sup>[9,10]</sup> and as self-healing materials.<sup>[11,12]</sup>

While platinum(II) complexes are favorable for forming liquid crystals owing to their planar geometry,<sup>[13–16]</sup> their emission properties in the solid state are usually poor and strongly red shifted because of significant concentration quenching and formation of Pt...Pt interactions, although there are specific examples where the liquid crystal phase offers control here.<sup>[17]</sup> Due to their octahedral structure, iridium complexes offer advantages in limiting luminescence concentration quenching and developing blue-emitting, phosphorescent materials by avoiding red-shifting metal-metal interactions. However, the octahedral structure is also a hurdle for the formation of liquid crystal mesophases because of the quasi-spherical structure lacking the necessary anisotropic geometry for preferential alignment. As a result, only very few iridium-based luminescent metallomesogens have been reported to date and all emit in the yellow-orange region of the electronic spectrum.<sup>[18–20]</sup>

In this paper a modular approach to the design of mesomorphic phosphorescent iridium complexes is demonstrated, a strategy allowing for the quasi-independent engineering of both the photophysical and aggregation properties of the complexes. To achieve this, we grafted strongly mesogenic groups<sup>[21–25]</sup> onto the non-chromophoric acetylacetonate (acac) ancillary ligand of  $[\text{Ir}(\text{C}^{\wedge}\text{N})_2(\text{acac})]$  complexes. The C<sup>∧</sup>N ligands are the cyclometalated ligands dictating the photophysical properties of the complexes, while the mesogenic units of the modified acac ancillary ligand control the mesomorphic properties of the materials.



**Scheme 1.** Chemical structures of the complexes in this study.

Our design is based on the C<sup>∧</sup>N ligands 2-(2,4-difluorophenyl)pyridine (dFppy, **Ir1**) for blue phosphorescence and 2-phenylpyridine (ppy, **Ir2**) for green phosphorescence. Two acac derivatives (**a** and **b**) are based on the rod-shaped difluorobiphenylcyclohexyl mesogen (Scheme 1).<sup>[21, 22]</sup> Photophysical properties of the complexes in solution are

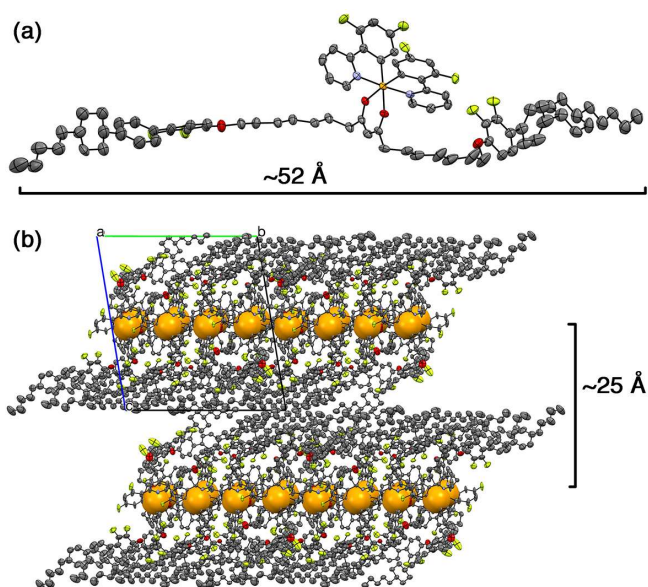
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identical to the parent complexes without mesogenic units<sup>[26]</sup> and limited (less than 20 nm) red-shifted emissions were observed in neat films. The complexes display Smectic A (SmA) liquid-crystalline phase. Importantly, after thermal annealing these materials exhibit the highest hole mobility reported to date for cyclometalated iridium complexes with values up to  $0.004 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ , that is two to three orders of magnitude higher than untreated samples of the same complexes and of non-mesomorphic iridium complexes.<sup>[27]</sup> These findings open a novel strategy for the design of phosphorescent iridium complexes with controlled photophysical properties and molecular order, in particular for high energy emission, which would be of interest for high efficiency OLEDs and polarized electroluminescence.

The ancillary ligands were prepared via a one-pot alkylation between acetylacetone and the mesogenic precursors bearing a bromoalkyl chain using NaH and *n*-BuLi in dry THF with low yield (10-23%).<sup>[28]</sup> The iridium complexes were prepared by reacting the chloro-bridged iridium(III) dimers  $[(C^*N)_4Cl_2Ir_2]$  with the acac ancillary ligands in the presence of tetrabutylammonium hydroxide in a mixture  $\text{CH}_2\text{Cl}_2/\text{MeOH}$  under reflux overnight. See ESI for details.



**Figure 1.** (a) Structure of one of the independent molecule of **Ir1a** (H atoms omitted). (c) Crystal packing of **Ir1a** viewed along the *a* axis, showing the layered structure (iridium ions are represented as a space-filling model).

Single crystals of **Ir1a** were obtained by slow evaporation of a dichloromethane/acetonitrile solution of the complex at room temperature (the structure is shown in Figure 1 and crystallographic parameters are given in ESI). Interestingly, the packing of the complexes results in layers of the iridium core complex alternating with layers of the mesogenic unit (Figure 1b).

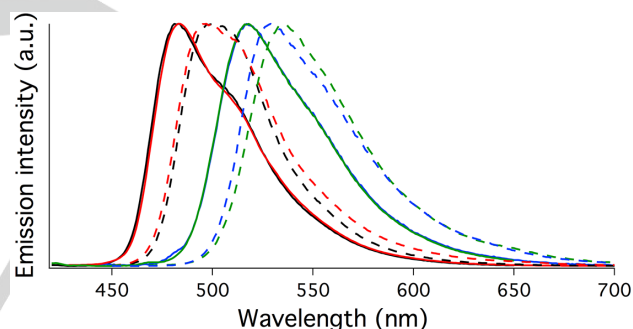
In solution in  $\text{CH}_2\text{Cl}_2$ , the photophysical properties of the complexes are primarily identical to the properties of the parent complexes without mesogenic moieties and data are summarized in Table 1.<sup>[26]</sup> The electronic absorption spectra of all iridium complexes were measured at room temperature

(Figure S1). The absorption bands in the range of 250-320 nm with high molar extinction coefficient ( $\sim 10^5 \text{ M}^{-1} \text{ cm}^{-1}$ ) are assigned to the spin-allowed, ligand-centered  $\pi\text{-}\pi^*$  transitions combined with the absorption features of the mesogenic units. Correspondingly, the iridium complexes bearing three mesogenic units (**Ir1b** and **Ir2b**) display more intense absorption coefficient than the corresponding complexes with only two mesogenic units (**Ir1a** and **Ir2a**).

**Table 1.** Photophysical data of complexes in  $\text{CH}_2\text{Cl}_2$

	Absorption /nm ( $\epsilon/10^3 \text{ M}^{-1} \text{ cm}^{-1}$ ) <sup>[a]</sup>	$\lambda_{em}$ /nm	$\Phi$ <sup>[a]</sup>	$\tau$ / $\mu\text{s}$ <sup>[a]</sup>
<b>Ir1a</b>	257 (103), 329 (12.7), 390 (5.0), 438 (2.2)	482 <sup>[a]</sup> 500 <sup>[b]</sup>	0.64	1.18
<b>Ir2a</b>	259 (100), 338 (10.8), 407 (4.1), 463 (2.7)	517 <sup>[a]</sup> 535 <sup>[b]</sup>	0.51	1.45
<b>Ir1b</b>	256 (131), 330 (12.5), 389 (5.1), 438 (2.2)	482 <sup>[a]</sup> 496 <sup>[b]</sup>	0.65	1.04
<b>Ir2b</b>	261 (126), 340 (11.0), 407 (3.8), 459 (2.5)	517 <sup>[a]</sup> 529 <sup>[b]</sup>	0.45	1.64

[a] At room temperature under argon. [b] Neat film at room temperature.



**Figure 2.** Solution (solid lines) and films (dashed lines) emission spectra of **Ir1a** (black), **Ir1b** (red), **Ir2a** (green), and **Ir2b** (blue).

The emission spectra of the complexes in solution and in neat films are shown in Figure 2 and data are summarized in Table 1. In solution, the dFppy-based complexes present a sky-blue emission at 482 nm, while the ppy-based complexes show a green emission at 517 nm. Being identical to emission from complexes without mesogenic groups, these results demonstrate that the pendent moieties have little effect on the emission property in solution. Using  $[\text{Ir}(\text{dFppy})_2(\text{acac})]$  and  $[\text{Ir}(\text{ppy})_2(\text{acac})]$  as references,<sup>[26]</sup> all complexes possess (within experimental error) photoluminescence quantum yields ( $\Phi$ ) and excited state lifetimes ( $\tau$ ) identical to the acac parent complexes. In solid state, the complexes are luminescent with less than 20 nm red-shift of the emission maxima.

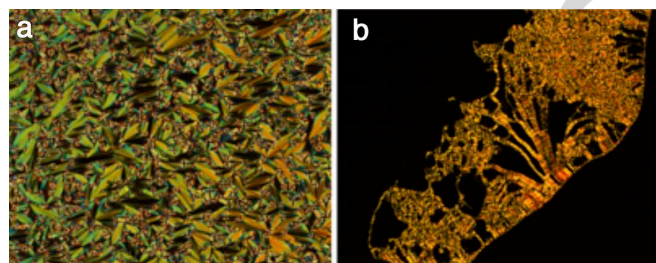
The phase transition behavior of the complexes was initially studied by polarized optical microscopy (POM, Figure 3 and S3) and differential scanning calorimetry (DSC, Figure S2) and data

are given in Table 2. Thus, both complexes with ligand a show a monotropic SmA phase. The two melting points are similar (158 and 163 °C for **Ir1a** and **Ir2a**, respectively), but the mesophase is more stable where the ppy ligand contains only hydrogen, which is to be expected given the great steric requirements of F compared to H. Both **Ir1b** and **Ir2b** also show a SmA phase, but this time it is enantiotropic. Given that the clearing points for the respective pairs of **Ir1** and **Ir2** complexes are broadly similar, then the enantiotropic behavior reflects a less stable crystal phase for **Ir1b** and **Ir2b**, consistent with the unsymmetrical nature of ligand b.

**Table 2.** Phase Transitions<sup>[a]</sup> and hole mobilities of the iridium complexes

	Phases, transition temperature	$\mu_h / \text{cm}^2 \text{V}^{-1} \text{s}^{-1}$	
		As cast	Annealed
<b>Ir1a</b>	Cr 158 Iso (SmA - Iso 124)	$1.2 \times 10^{-6}$	$4.5 \times 10^{-4}$
<b>Ir2a</b>	Cr 163 Iso (SmA - Iso 151)	$2.1 \times 10^{-7}$	$1.5 \times 10^{-4}$
<b>Ir1b</b>	Cr 104 SmA 130 Iso	$5.6 \times 10^{-6}$	0.003
<b>Ir2b</b>	Cr 126 SmA 153 Iso	$3.9 \times 10^{-4}$	0.004

[a] Scan rate for all runs was 10 °C min<sup>-1</sup>. Phase nomenclature: Cr = crystal, SmA = smectic A mesophase, Iso = isotropic liquid.



**Figure 3.** Photomicrographs of the complexes in the SmA phase upon cooling from isotropic state: (a) **Ir1a** at 120 °C, (b) **Ir2b** at 145 °C.

Temperature-dependent, small-angle X-ray scattering (SAXS) was carried out to confirm the smectic phase observed by POM, and the corresponding data are listed in Table S1. Besides the interlayer distance, the four complexes exhibit identical SAXS patterns in the mesophase (Figure S4-S7) and so **Ir1a** was selected as a representative example to illustrate the layer structure in detail. Thus, at 30 °C, there is an intense reflection at 3.4° and several reflection peaks in the region  $2\theta = 6-18^\circ$ , indicating long-range order characteristic of a crystalline phase (Figure S4).

On cooling from the isotropic liquid, the XRD pattern (Figure S4) shows one sharp and intense reflection centered at  $2\theta = 2.34^\circ$  (37.8 Å) and a weak reflection at  $2\theta = 4.67^\circ$  (18.9 Å). The ratio of their reciprocal *d*-spacings is 1:2 and they are indexed as the (001) and (002) reflections of a smectic phase. This *d*-

spacing is much smaller than the calculated molecular length of ~52 Å obtained from the X-ray crystal structure (Figure 1b), indicating significant interdigitation. The broad reflection at  $2\theta \sim 10^\circ$  corresponds to a poorly correlated separation of electron-rich (and therefore strongly diffracting) Ir atoms centered at about 9 Å (in the crystal structure, distances between neighboring iridium ions vary between ~6.8 and ~14.8 Å). Additionally, a very broad and weak reflection peak at about 18.5° (4.8 Å) is observed, assigned to the molten alkoxy chains in their liquid-like conformation. **Ir1a** and **Ir2a** are monotropic liquid crystals: upon further cooling the complexes crystallise and on reheating the crystal phase, they display only the Cr-Iso transition.

The hole mobilities of the complexes were estimated at room-temperature (as in a device) using the space charge limited current (SCLC) method. The hole-only device were fabricated with the architecture ITO/PEDOTS:PSS (40 nm)/iridium complexes/MoO<sub>3</sub> (10 nm)/Ag (100 nm). For devices without any treatment, the pristine films of iridium complexes display hole mobilities in the range of 10<sup>-7</sup>-10<sup>-4</sup> cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>, which is similar to the hole mobility of their corresponding parent iridium complex, such as [Ir(ppy)<sub>2</sub>(acac)].<sup>[27]</sup> After thermal treatment of the films at 150 °C for 30 min, the hole mobilities at room-temperature increased by up to three orders of magnitude. **Ir1b** and **Ir2b** show hole mobilities as high as 0.003 and 0.004 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>, respectively, which are the highest reported hole mobilities for iridium complex (Table 2, Figure S9).

In summary, we report a modular strategy for the design of liquid-crystalline phosphorescent iridium complexes, based on the attachment to a non-chromophoric ancillary ligand, acetylacetonate, of a unit able to promote mesomorphism. As such, the core of the complex can be modified to change the photophysical properties and the mesogenic groups can be adapted to alter the molecular organization in the bulk; thus the four complexes display SmA mesophase. As such, blue and green phosphorescent liquid-crystalline iridium complexes have been obtained for the first time.

Importantly, these iridium metallomesogens exhibited hole mobilities up to 0.004 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> after thermal annealing, which are the highest values reported for iridium complexes. This result is very promising for using these materials for phosphorescent OLEDs<sup>[29]</sup> and as donor materials for organic solar cells.<sup>[30-33]</sup>

## Acknowledgements

We thank the European Union (MC-IIF-329199), the National Natural Science Foundation of China (21202139) and the University of York for financial support and EPSRC for funds to purchase the SAXS instrument.

**Keywords:** Iridium • Phosphorescence • Liquid-crystal • Hole mobility • Smectic

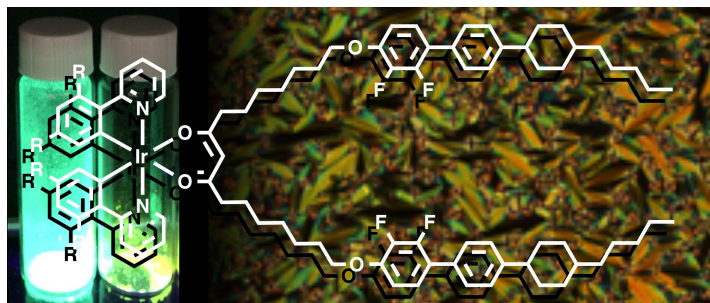
**X-ray data for Ir1a:** C<sub>85</sub>H<sub>95</sub>F<sub>8</sub>IrN<sub>2</sub>O<sub>4</sub>, *M<sub>r</sub>* = 1552.82, 0.2633 × 0.0656 × 0.0219 mm<sup>3</sup>, triclinic, P-1, *a* = 25.2543(4), *b* =

25.9749(4),  $c = 26.1335(5)$ ,  $\alpha = 75.4021(16)$ ,  $\beta = 75.9839(16)$ ,  $\gamma = 62.4919(17)$ ,  $V = 14555.2(5)$  Å<sup>3</sup>,  $Z = 8$ ,  $\rho_{\text{calcd}} = 1.417$  g cm<sup>-3</sup>,  $\mu = 4.158$  mm<sup>-1</sup>, CuK $\alpha$  ( $\lambda = 1.54184$ ),  $T = 99.97(16)$  K,  $2\theta_{\text{max}} = 133.198^\circ$ , 81484 reflections collected, 50657 independent reflections ( $R_{\text{int}} = 0.0370$ ), 4956 parameters,  $R_1$  [ $I > 2\sigma(I)$ ] = 0.0437,  $wR_2$  (all data) = 0.1156, goodness-of-fit on  $F^2 = 1.086$ ,  $\Delta F_{\text{max}}$  ( $\Delta F_{\text{min}}$ ) = 1.43 (-2.01) e Å<sup>-3</sup>. CCDC 1418808 contains the supplementary crystallographic data for this paper. These data are provided free of charge by The Cambridge Crystallographic Data Centre.

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Layout 2:

## COMMUNICATION



Yafei Wang, Christopher P. Cabry, ManJun Xiao, Louise Male, Stephen J. Cowling, Duncan W. Bruce,\* Junwei Shi, Weiguo Zhu and Etienne Baranoff\*

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**Blue and Green Phosphorescent Liquid-Crystalline Iridium Complexes with High Hole Mobility**

A simple modular design was used to impart liquid crystalline properties to iridium complexes without modification of their photophysical properties. Annealed films of the liquid crystalline complexes display hole mobilities up to three orders of magnitude higher than parent iridium complex without mesogenic groups and are the highest values reported for iridium complexes.

**Keywords:** Iridium, Phosphorescence, Liquid crystals, Hole mobility, Smectic