

Mini-Review:**Recent advances in Blue-Emitting Iridium(III) Complexes Featuring 2-(2,4-Difluorophenyl)pyridine as the Cyclometalating Ligand****Nur Khaliesa Zulkarnaen and Noorshida Mohd Ali****Department of Chemistry, Faculty of Science and Mathematics, Universiti Pendidikan Sultan Idris, Tanjong Malim 35900, Perak, Malaysia**** Corresponding author:**

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Abstract: The significance of blue emitters in the realization of cutting-edge organic light-emitting diode (OLED) displays cannot be overstated, leading to considerable efforts aimed at synthesizing intense blue phosphorescent Ir(III) complexes that are stable for use as dopants. Nevertheless, the quest for highly effective blue phosphorescent Ir(III) complexes remains fraught with challenges. Bis(2-(4,6-difluorophenyl)pyridinato-C²,N)(picolinato)iridium(III) (FIrpic), which employs 2-(2,4-difluorophenyl)pyridine (F₂ppy) as the cyclometalating ligand and picolinic acid as the ancillary ligand, serves as a benchmark for blue phosphorescent Ir(III) complexes because of its outstanding performance in OLED devices, yet it still exhibits several limitations. Consequently, the pursuit of more effective blue phosphorescent iridium complexes remains a priority. In recent years, advancements in the field have focused on blue phosphorescent Ir(III) complexes utilizing a variety of cyclometalated ligands in conjunction with bidentate ancillary ligands, leading to extensive investigation. This discussion will highlight and analyze the most recent progress in designing and preparing those complexes, particularly utilizing F₂ppy alongside diverse bidentate ancillary ligands. Future studies are encouraged to focus on structural modification of the F₂ppy cyclometalating ligand, particularly with the combination of suitable ancillary ligands, to improve further color purity of blue-emitting Ir(III) complexes and their photophysical performance.

Keywords: blue phosphorescent; Ir(III) complexes; ancillary ligand; 2-(2,4-difluorophenyl)pyridine; OLED

■ INTRODUCTION

In the previous two decades, the performance of organic light-emitting diodes (OLEDs) in commercial shows has advanced markedly, evolving from first-generation devices to the latest iterations [1]. OLEDs are now positioned to emerge as the leading technology for flat-screen displays, solid-state light sources, and other applications owing to their distinctive and disruptive features, including energy-saving, wide viewing angles, fast response, high contrast, and high color purity [2-3]. OLED devices generate light using purely organic molecules (fluorescent) or organometallic molecules (phosphorescent). Light emission occurs through electron-hole recombination in these devices [4], producing 25%

singlet and 75% triplet excitons [5]. The quantum efficiency of a fluorescent emitter device is constrained to 25% due to charge recombination in the OLED, which generates excited states in a 1:3 ratio of singlet to triplet excitons, resulting in the loss of 75% of the triplet excitons [6]. The second-generation phosphorescent materials are created by incorporating metal atoms like Pt, Ir, and Au into molecules to enhance spin-orbit coupling, facilitated by intersystem crossing, and utilize 75% of excitons in the triplet state [7]. Phosphorescent materials can achieve quantum efficiency close to 100% of the theoretical maximum [8], greatly improving the luminescent efficiency of OLED devices that are extensively used in the industry [9].

The OLED displays and solid-state lighting require red, green, and blue light sources for excellent quality [10]. Leading display manufacturers currently utilize red and green phosphorescent OLEDs (PHOLEDs), which offer device lifetimes of up to 10^5 hours [11]. However, blue PHOLEDs have only recently been upgraded to achieve efficient operation for several hundred hours at commercial luminance, falling short of the device lifetimes required for screens and displays [5]. Blue light is essential for full-color flat-screen displays and solid-state light sources, as it constitutes one of the three primary colors of light [9]. The lighting and display industries are facing critical problems about the short lifetime of blue OLEDs, and the OLED industry is still working to develop efficient and long-lasting blue OLEDs that use phosphorescent and thermally activated delayed fluorescence emitters [2]. Developing a stable, intense blue emitter is vital to increasing the color spectrum and lowering the power consumption of phosphorescent OLEDs [12].

Research is currently developing transition metal complexes for light-emitting equipment [13]. Ir(III) complexes are fascinating because they feature short-lived triplet excited states and enhanced photoluminescence quantum yields, essential for OLEDs [14]. Transition-metal complexes such as Ru(II), Os(II), and Re(I), featuring a d^6 configuration, often produce emission linked to the highest occupied molecular orbital (HOMO) of the metal center [15]. On the contrary, the HOMO of numerous Ir(III) complexes also incorporates the π orbitals of their coordinating ligands, resulting in considerably more intricate emissive states [16]. This leads to tunable emission throughout the visible light spectrum, spanning from blue to red [17], making the complexes attractive for many uses, including dopants for OLEDs [18]. Moreover, Ir has a significant spin-orbit coupling [19], therefore, it shows very rapid triplet radiative decay, resulting in high quantum yields for photoluminescence [20]. Furthermore, Ir(III) complexes are recognized as the most favorable since they have excellent thermal and electrochemical stability [21-24]. The innovation of novel phosphorescent Ir(III) complexes with excellent manufacturing capabilities, electronic properties, and high luminescence quantum

efficiency has been the focus of many research activities [25]. Despite significant efforts in the molecular design of blue-emissive Ir(III) complexes, designing and synthesizing deep-blue-emitting cyclometalated Ir(III) complexes remains notably challenging [26].

A primary method for generating blue emission from Ir(III) phosphors involves modifying cyclometalated ligands [27]. Ir phosphors commonly contain phenylpyridine (ppy) derivatives as cyclometalating ligand frameworks [27-29]. The energy difference between the HOMO and the lowest unoccupied molecular orbital (LUMO) can be significantly increased by adding electron-withdrawing groups (EWGs) to the phenyl moiety or incorporating electron-donating groups (EDGs) into the pyridine moiety [30-31]. This approach entails introducing EWGs like F atoms, trifluoromethyl, carbonyl, cyano, and sulfonyl groups into the phenyl group of 2-phenylpyridine, or replacing CH units in the phenyl ring with sp^2 -hybridized nitrogen atoms [27,32-33].

A well-known example of this strategy is the widely used blue phosphorescent material bis(2-(4,6-difluorophenyl)pyridinato- C^2,N)(picolinato)iridium(III), or FIrpic (complex **1** in Fig. 1) [30,34-36], emitting at a peak wavelength of around 470 nm with an emission quantum yield of about 80% [37]. As demonstrated by FIrpic, more than one fluorine atom must typically be incorporated into the phenyl ring in order to achieve blue emission [27,38]. While FIrpic is recognized as a standard blue phosphor for high-performance PHOLEDs, it has certain limitations frequently observed in many blue phosphorescent emitters [39].

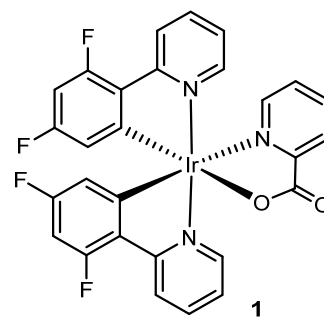
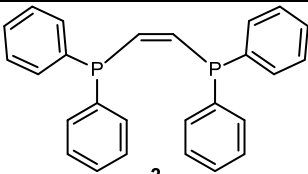
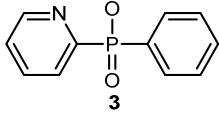
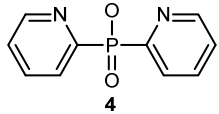
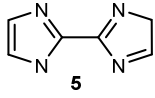
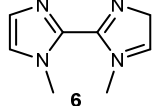
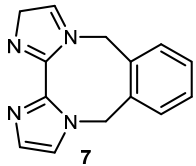
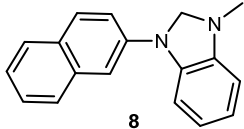


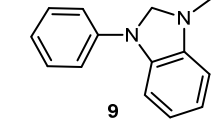
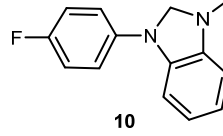
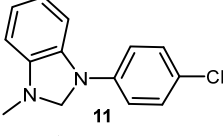
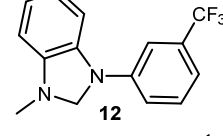
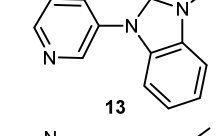
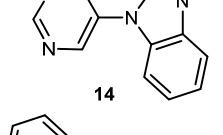
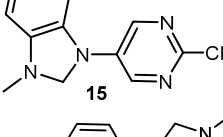
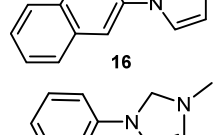
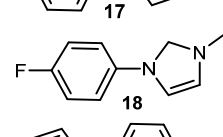
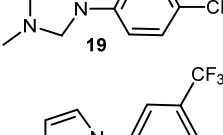
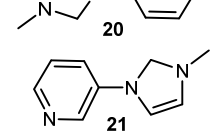

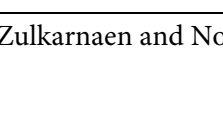
Fig 1. Structure of the first iridium(III) complexes with F_2 ppy as the cyclometalating ligand (complex **1**)

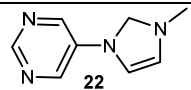
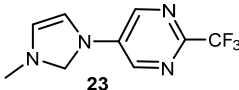
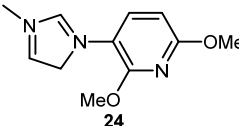
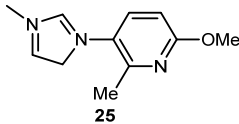
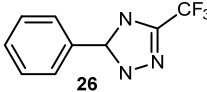
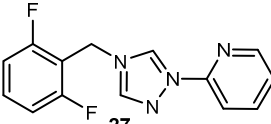
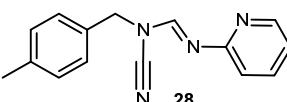
The ancillary ligands involved are vital for modifying the light emission energy of Ir(III) phosphors [40]. When effectively paired with a well-designed cyclometalated ligand, it can result in phosphors that deliver blue emission, a narrow full width at half maximum, and enhanced photoluminescence quantum efficiency. The selection of the appropriate ancillary ligand is crucial for achieving blue emission, as it increases the energy gap of Ir(III) phosphors [27]. The photophysical characteristics of Ir(III) complexes with bidentate ligands are mainly governed by the structures of the ligands. With a rational approach to structural design, these complexes can display emission colors spanning the ultraviolet to near-infrared range [30]. Blue-emitting

Ir(III) complexes can be developed by incorporating stronger σ -donating ancillary ligands, effectively widening the HOMO-LUMO gap [41] and enhancing blue emission. Therefore, this review aims to provide a thorough summary of the most recent advancements in the design and synthesis of blue phosphorescent Ir(III) complexes, using 2-(2,4-difluorophenyl)pyridine (F_2ppy) as the cyclometalated ligand in conjunction with various bidentate ancillary ligands. Table 1 summarizes the photophysical data of reported Ir(III) complexes featuring the F_2ppy cyclometalating ligand and various types of ancillary ligands that exhibit blue emission, reported between 2012 and 2023.

Table 1. Photophysical data of reported Ir(III) complexes featuring the F_2ppy cyclometalating ligand and various types of ancillary ligands

Structure of ancillary ligand	Type of ancillary ligand	λ_{max} (nm)	Φ_{PL}	Lifetime, τ (μs)	Ref.
	Phosphine	471	0.18	9.20	[14]
	Phosphine	471	0.35	1.44	[38]
	Phosphine	470	0.25	1.34	[38]
	Imidazole	464, 490	0.20	1.559	[42]
	Imidazole	451, 484	0.02	0.091	[42]
	Imidazole	457, 487	0.68	3.84	[42]
	N-heterocyclic carbenes	485	0.06	1.90	[41]

Structure of ancillary ligand	Type of ancillary ligand	λ_{\max} (nm)	Φ_{PL}	Lifetime, τ (μs)	Ref.
 9	N-heterocyclic carbenes	483	0.14	2.10	[41]
 10	N-heterocyclic carbenes	485	0.26	1.70	[41]
 11	N-heterocyclic carbenes	483	0.65	1.80	[41]
 12	N-heterocyclic carbenes	470	0.37	1.90	[41]
 13	N-heterocyclic carbenes	473	0.73	1.80	[41]
 14	N-heterocyclic carbenes	465	0.30	1.80	[41]
 15	N-heterocyclic carbenes	469	0.57	1.90	[41]
 16	N-heterocyclic carbenes	465	0.51	1.90	[41]
 17	N-heterocyclic carbenes	469	0.69	1.80	[41]
 18	N-heterocyclic carbenes	474	0.63	1.90	[41]
 19	N-heterocyclic carbenes	473	0.61	1.80	[41]
 20	N-heterocyclic carbenes	471	0.28	1.90	[41]
 21	N-heterocyclic carbenes	471	0.33	1.70	[41]

Structure of ancillary ligand	Type of ancillary ligand	λ_{\max} (nm)	Φ_{PL}	Lifetime, τ (μs)	Ref.
	N-heterocyclic carbenes	470	0.11	1.70	[41]
	N-heterocyclic carbenes	455	0.32	1.90	[41]
	N-heterocyclic carbenes	467	0.66	0.45	[43]
	N-heterocyclic carbenes	464	0.31	0.38	[43]
	N-heterocyclic carbenes	457, 481	0.201	2.48	[44]
	N-heterocyclic carbenes	461	-	-	[48]
	Pyridine-formimidamide	462, 487	0.02951	0.37	[50]

■ INFLUENCE OF ANCILLARY LIGANDS ON LIGHT EMISSION CHARACTERISTICS

Phosphine

Incorporation of strong σ -donating ligands as ancillary ligands can elevate and isolate the non-radiative $d-d^*$ excited state from the emitting triplet states, including the triplet metal-to-ligand charge transfer ($^3\text{MLCT}$) [14]. This enhancement leads to improved stability and efficiency of emission [41]. For example, phosphine-based ligands are known for their strong σ -donating properties. In 2016, Rota Martir and his team [14] synthesized thirteen cationic Ir(III) complexes featuring bisphosphine ancillary ligands represented by the formula $[\text{Ir}(\text{C}^{\wedge}\text{N})_2(\text{P}^{\wedge}\text{P})]\text{PF}_6$. One of these complexes (complex **2** in Fig. 2) includes 2-(2,4-difluorophenyl)pyridine as the primary ligand ($\text{C}^{\wedge}\text{N}$) and a biphosphine ($\text{P}^{\wedge}\text{P}$) ancillary ligand. This complex exhibited green-blue phosphorescence with an emission peak at 471 nm. Introducing F atoms into the $\text{C}^{\wedge}\text{N}$ ligand stabilizes the frontier molecular orbitals, resulting in a blue emission shift, as demonstrated by complex **2**, while

also enhancing the photoluminescence quantum yield and prolonging emission lifetimes ($\Phi_{\text{PL}} = 18\%$).

Furthermore, Wu and his team [38], in 2016, synthesized novel pyridinylphosphinate ancillary ligands, and the addition of the phosphoryl ($\text{P}=\text{O}$) group was intended to enhance the coordination environment of the ancillary ligand, thereby inducing a hypsochromic (blue) shift. Consequently, they developed two innovative ancillary ligands: phenyl(pyridin-2-yl)phosphinate (ppp) and dipyrinylphosphinate (dpp) for the fabrication of highly effective blue phosphorescent Ir(III) complexes, specifically $(\text{dfppy})_2\text{Ir}(\text{ppp})$ (complex **3**) and $(\text{dfppy})_2\text{Ir}(\text{dpp})$ (complex **4**) as shown in Fig. 2, where dfppy denotes 2-(2,4-difluorophenyl)pyridine. Both complexes displayed strong blue phosphorescence: complex **3** showed a maximum emission at 471 nm with a noticeable shoulder at 497 nm, while complex **4** emitted at 470 nm with a weaker signal at 496 nm. The phosphorescence quantum yields were determined to be 0.35 for complex **3** and 0.25 for complex **4**. Furthermore, the lifetimes of

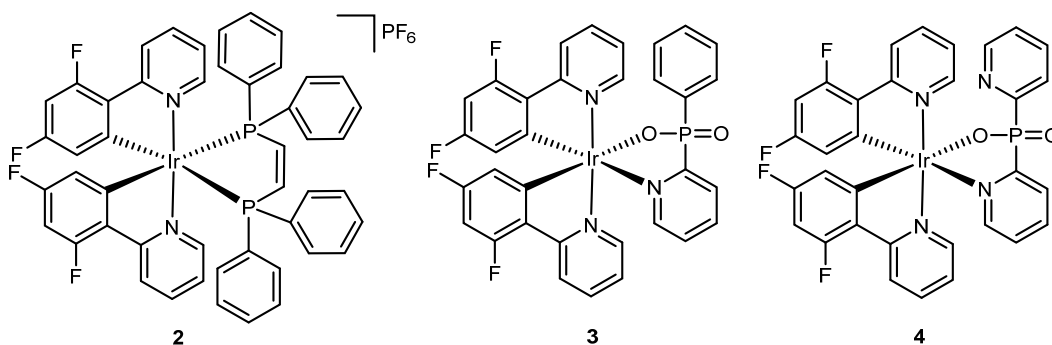


Fig 2. Structure of cyclometalated F₂ppy iridium(III) complexes with phosphine-based ancillary ligands (complexes 2–4)

the two novel complexes were measured in the range of microseconds, specifically 1.44 μ s for complex 3 and 1.34 μ s for complex 4. These findings indicate that Ir(III) complexes incorporating pyridinylphosphinate ancillary ligands exhibit considerable potential as blue phosphorescent materials.

Imidazole

Imidazole-based ligands have also been identified as promising candidates for electron-rich diimine frameworks, enabling the development of intense blue emitters. In 2014, Henwood and colleagues [42] employed a strategy to improve complex rigidity by tethering biimidazole, which enhanced photoluminescent quantum yields. They synthesized three cationic Ir(III) complexes, [Ir(dFppy)₂(N[^]N)]PF₆, featuring biimidazole-type N[^]N ancillary ligands. Complex 5 utilized the parent biimidazole, biim, while complex 6 featured a dimethylated variant, dMebiim, and complex 7 included an *ortho*-xylyl-tethered biimidazole, *o*-Xylbiim, as shown in Fig. 3. All synthesized complexes demonstrated deep

blue emissions, with their peak maximums recorded at 464 nm for complex 5 and at 457 nm for both complexes 6 and 7 at ambient temperature. At reduced temperatures, complex 5 exhibited maximum emission peaks at 453 nm, complex 6 at 451 nm, and complex 7 at 450 nm. The complexes reported in this study are recognized in literature as among the most intense deep-blue cationic iridium emitters, exhibiting a maximum emission wavelength of less than 470 nm. Additionally, the low-temperature emission lifetimes (τ_e) for the three complexes were comparable, ranging from 3.6 to 4.0 μ s. At 298 K, although τ_e for complexes 5 and 7 remained in the microsecond range, complex 6 experienced a significant reduction to 90 ns. The Φ_{PL} for complex 5 was determined to be 0.20, which drastically decreased to 0.02 for complex 6, whereas complex 7 exhibited a Φ_{PL} of 0.68. This strategy enhances the photoluminescence quantum yield by limiting the conformational flexibility of the bidentate chelate, thereby increasing molecular rigidity and resulting in an elevated Φ_{PL} in strongly blue-emitting cationic Ir(III) complexes.

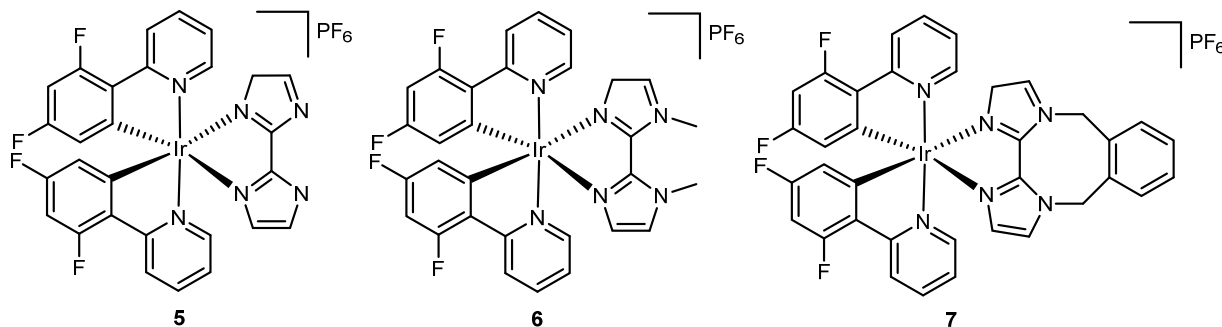


Fig 3. Structure of cyclometalated F₂ppy iridium(III) complexes with imidazole-based ancillary ligands (complexes 5–7)

N-Heterocyclic Carbenes

In 2015, Li and co-workers [41] employed monoanionic *N*-heterocyclic carbene (NHC) ancillary ligands to develop sixteen neutral Ir(III) phosphorescent complexes emitting blue to greenish-blue light, (F₂ppy)₂Ir(NHC). These complexes exhibited characteristic phosphorescence between 455 and 485 nm, with photoluminescence quantum efficiencies as high as 0.73. Wu and his team [38] altered the phenyl groups in the NHCs by adding EWGs like –CF₃, or –F, or substituting them with *N*-heteroaromatic rings such as pyridine or pyrimidine. These changes effectively increased the energy separation between the HOMO and LUMO, causing a shift in emission toward the blue region. Complex **20** demonstrated the strongest photoluminescent intensity and efficiency compared to other evaluated complexes under identical conditions. These findings strongly suggest the reported iridium phosphors (complexes **8–23** in Fig. 4) are well-suited for use in OLEDs, with complex **20** emerging as a particularly promising emitter.

Following the achievements of Li et al. [41], in 2016, Aghazada and his research team [43] synthesized an innovative series of neutral Iridium(III) bis(2-(2',4'-difluorophenyl)pyridine)(1-(2'-aryl)-3-methylimidazol-2-ylidene) complexes as part of their study. This investigation aimed to gain insight into how NHC ancillary ligands featuring cyclometalating moieties with electron-donating properties influence the system. They systematically replaced the cyclometalating aryl group on the NHC ligand, moving from 2,4-dimethoxyphenyl to 4-methoxy-2-methyl-3-pyridine, which led to shifts of the Ir(III) complexes' energy levels and creation of new blue-emitting materials with improved HOMO and triplet level energies. Furthermore, to assess the impact of the π -donor strength of the NHC ligands, they selected imidazole and 1,2,4-triazole for their study. Remarkably, only those complexes that included an NHC ligand with imidazole and a nitrogen atom in the cyclometalating aryl group exhibited blue light emission. Complexes **24** and **25** (Fig. 4) showed maximum emission wavelengths at 467 and 464 nm, respectively, with complex **25** exhibiting the most notable, blue-shifted emission. Interestingly, despite

the HOMO and LUMO levels being identical for both complexes, the emission energies differed slightly, with complex **24** possessing a more stabilized E(T₁) energy level. Among the synthesized complexes, complex **24** recorded the highest photoluminescence quantum yield (PLQY) at 0.66, whereas complex **25** presented a lower PLQY of 0.31.

Additionally, the functionalized 1,2,4-triazole acts as a proficient ligand for facilitating blue emission in Ir(III) complexes [44–46]. A study carried out in 2020 by Bain and colleagues [47] incorporated 1,2,4-triazole, leading to the successful creation of a blue-light-emitting Ir(III) complex. A sky-blue-emitting complex, identified as (F₂,4ppy)₂Ir(tfmptz) (complex **26** in Fig. 4), was synthesized using 2-(3-(trifluoromethyl)-1*H*-1,2,4-triazol-5-yl)pyridine as the ancillary and 2-(2,4-difluorophenyl)pyridine (F₂ppy) as the primary ligands. This complex exhibited distinct emission maxima at 457 and 481 nm with a notably high PLQY (39.61%) and a short excited-state lifetime (0.53 μ s). This contributed to the complex's outstanding performance, as evidenced by an external quantum efficiency of 21.23% and sky-blue emission with CIE coordinates of (0.15,0.26). The study further demonstrated that this ancillary ligand, with its stronger ligand field strength, effectively lowers the HOMO energy levels and increases the energy band gap.

In 2021, our research group also explored the potential of 1*H*-1,2,4-triazole-based NHCs as ancillary ligands. These ligands were modified explicitly with a 2,6-difluorobenzyl substituent to develop the Ir(III) complex, referred to as [Ir(2,4-F₂ppy)₂(F₂bpyta)]PF₆ (complex **27** in Fig. 4) [48]. The incorporation of a pyridyltriazole-based ancillary ligand was strategically aimed at increasing the energy of the LUMO, thereby enhancing the luminescent characteristics of the resulting complexes [26,48–49]. The steady-state photoluminescence spectrum of [Ir(2,4-F₂ppy)₂(F₂bpyta)]PF₆ exhibited green-blue emission, featuring a peak wavelength at 461 nm. Notably, their findings demonstrated triazole-based heteroleptic Ir(III) complexes effectively raise the energy of the LUMO by widening the energy gap, thereby resulting in an elevated T₁ energy level [47]. Furthermore, this research emphasizes

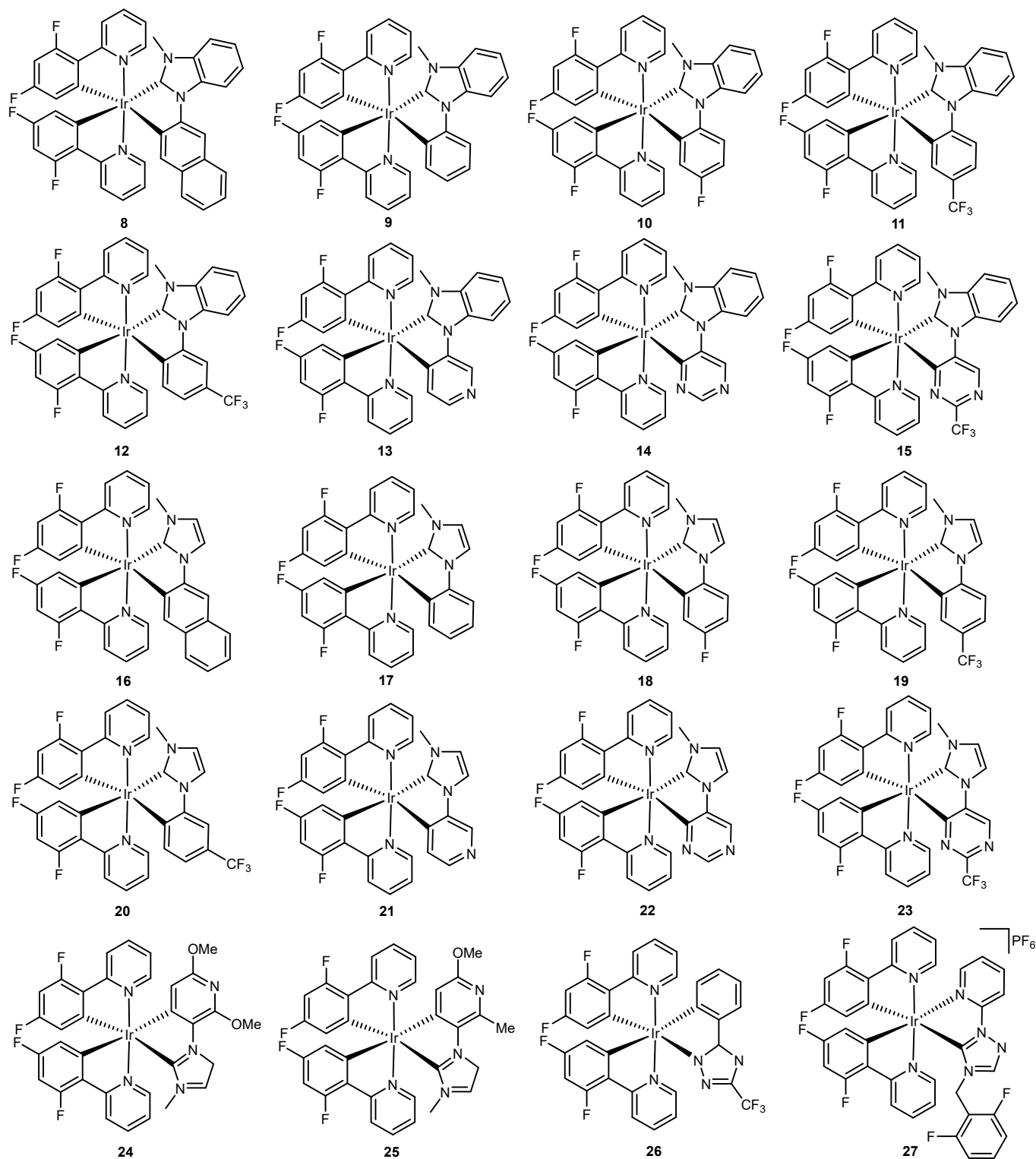


Fig 4. Structure of cyclometalated F₂ppy iridium(III) complexes with *N*-heterocyclic carbene-based ancillary ligands (complexes 8–27)

the potential of modifying 1,2,4-triazolepyridyl-based ligands as an effective approach to tuning the electronic

characteristics of Ir(III) complexes for the development of blue-emitting phosphorescent materials.

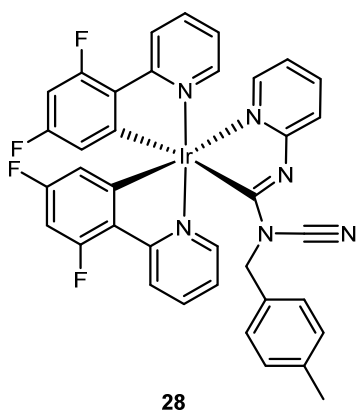


Fig 5. Structure of cyclometalated F₂ppy iridium(III) complex with pyridine-formimidamide-based ancillary ligands (complex **28**)

Pyridine-Formimidamide

Subsequently, in 2023, our research team unveiled a new neutral Ir(III) complex, referred to as [Ir(2,4-F₂ppy)₂(mbpyta)] (complex **28** in Fig. 5) [50]. This complex similarly utilized 1*H*-1,2,4-triazole-type NHCs but featured a 4-methylbenzyl substituent. Notably, infrared spectroscopy revealed a significant absorption band at 2220 cm⁻¹, attributed to the nitrile group, which suggested the formation of a pyridine-formimidamide ancillary ligand through the splitting of two nitrogen atoms within the 1,2,4-triazole ring. The crystal structure analysis confirmed that the Ir(III) complex exhibited a distorted octahedral coordination geometry in the solid state. Furthermore, at room temperature, this complex emitted green-blue light (λ_{em} = 464 nm), with a PLQY (Φ_{PL}) of 0.37. This investigation highlighted the potential of using a pyridine-formimidamide-based ligand as an ancillary ligand to modify the electronic characteristics of the Ir(III) complex, thereby enabling the design of an innovative blue-emitting phosphorescent complex.

CONCLUSION

This mini-review outlines the recent advancements in blue phosphorescent Ir(III) complexes that utilize 2-(2,4-difluorophenyl)pyridine as a cyclometalating ligand, alongside various bidentate ancillary ligands. The emission characteristics of these Ir(III) complexes can be finely tuned by altering the ancillary ligands, with changes in substituents also influencing the photophysical

characteristics of the complexes. A range of strategically designed ancillary ligands has been coordinated with the Ir center to develop an effective blue emitter. Future research needs to direct its attention towards the advancement of innovative design strategies and the molecular design of ancillary ligands, which could significantly increase their potential as blue phosphorescent materials, thereby improving the efficacy of blue Ir(III) phosphor.

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CONFLICT OF INTEREST

The authors have no conflict of interest.

AUTHOR CONTRIBUTIONS

Nur Khaliesa Zulkarnaen and Noorshida Mohd Ali wrote and revised the manuscript. All authors agreed to the final version of this manuscript.

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