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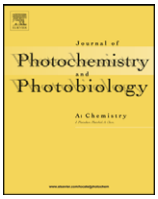

Efficient phosphorescent bis-cyclometallated iridium complex based on triazole-quinoline ligand

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ABSTRACT

Novel phosphorescent bis-cyclometallated iridium(III) complex based on the quinoline and 2-phenyl-1,2,3-triazole moieties fused at the *f* edge of quinoline has been synthesized. The iridium complex exhibits yellow phosphorescence emission of high quantum yield (78% in solution) and microsecond lifetime (2.6 μs), indicating radiative recombination to originate mainly from the triplet metal-to-ligand charge transfer excited state due to strong spin–orbit coupling of the iridium. The revealed properties show a great promise for the new iridium complex to be used in efficient yellow phosphorescent organic light emitting diodes.

1. Introduction

In recent years organic light emitting diodes (OLEDs) have attracted worldwide attention for their high emission efficiency and low operating voltage in flat panel displays [1a]. An important breakthrough in this field of research was development of phosphorescent light emitting diodes (PHOLEDs) based on heavy metal complexes [1b–8]. In particular, iridium complexes have attracted much attention because their emission energy can be finely tuned from blue to red by the replacement of chelating ligands [9,10] or by peripheral ligand functionalization [5,11a, 12] with electron-withdrawing and electron-donating substituents. Furthermore, the use of iridium complexes allows the alteration of the excited state lifetime by a proper mixing of the metal-to-ligand charge transfer (MLCT) excited states [1b] with low-lying triplet ligand centered excited states (LC) [1b]. In contrast to OLEDs made of organic fluorescent materials, in which the efficiency is limited by non-emissive triplet excited states, PHOLEDs internal quantum yield can reach the theoretical value of 100%, since the strong spin–orbit coupling of heavy metal phosphors employed as the electroluminescent materials enables the mixing of singlet–triplet excited states, and in most cases, significantly enhances intersystem crossing to the phosphorescent triplet states [13–16].

Both the electroluminescence (EL) efficiency and the emission wavelength of devices based on iridium complexes are greatly affected by the structure of the organic ligand. It is known that the HOMO of cyclometallated iridium(III) complexes is determined by the 5d orbital of Ir(III) with substantial mixing with the π orbitals of the ligand and the LUMO is related to the π* orbitals of the ligand [17,18]. Thus, by changing the structure of the ligands one can modulate HOMO and LUMO values of Ir(III) complexes in order to tune the emission wavelength. For example, Thompson and co-workers [11a] reported series of neutral iridium(III) complexes exhibiting different emission wavelengths. So far, number of cyclometallated ligands, including 2-arylpyridine [19a–c,5], 2-arylbenzothiazole [11a and 20b], 2-arylbenzooxazole [11a], 2-arylbenzoimidazole [21] and 2-arylisoquinoline [19a], which can readily cyclometallate with iridium trichloride, have been investigated. Recently, Wang and co-workers synthesized a series of new quinoline-based ligands with different substituents and showed that quinoline-based iridium complexes are promising candidates for efficient red emitters [22]. So far the phosphorescent bis-cyclometallated iridium

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complexes based on the novel ligand, comprising of fused quinoline and triazole moieties, have never been reported before.

Here, we report the synthesis of a new cyclometallated ligand with quinoline and 2-phenyl-1,2,3-triazole moieties, which are fused at the *f* edge of quinoline. The corresponding bis-cyclometallated iridium(III) complex was prepared and the optical properties of its dilute solutions in dimethyl sulfoxide (DMSO) were investigated. The properties of the new iridium(III) complex were explored by measuring absorption and photoluminescence (PL) spectra as well as PL quantum yield and the lifetime.

2. Experimental details

2.1. Measurements

The structures of phenyltriazole-quinoline-based ligand and the corresponding bis-cyclometallated iridium(III) complex were identified by elemental analysis, ^{1}H NMR and IR spectroscopies. The ^{1}H NMR spectra were taken on Varian Unity Inova (300 MHz) spectrometer at room temperature. The course of the reactions was monitored by TLC on Alugram SIL G/UV$_{254}$ plates (eluent: acetone–hexane = 7:18) and development with I_2 or UV light. Silica gel (grade 62, 60–200 mesh, 150 Å, Aldrich) was used for column chromatography. Elemental analysis was performed with an Exeter Analytical CE-440 Elemental Analyzer. IR spectroscopy was performed on a PerkinElmer Spectrum BX II FT-IR System, using KBr pellets. UV–vis absorption spectra were recorded on PerkinElmer Lambda 950 spectrophotometer.

Photoluminescence spectra of the bis-cyclometallated iridium(III) complex and the free ligand used in its synthesis were measured using JY HRD 1 double monochromator coupled to Hamamatsu R1463P photomultiplier operating in the photon-counting mode. The PL measurements were carried out at room temperature in dilute DMSO solutions, which were continuously bubbled with high purity helium to remove dissolved oxygen, and thus, avoid quenching of the radiative decay from the triplet states. The use of the solvent with high boiling temperature (189 °C), such as DMSO, was required to prevent the solutions from fast evaporation; therefore, maintaining their concentration during the measurements. For spectral and quantum yield measurements the solutions were diluted to 0.05 OD at the excitation wavelength to avoid reabsorption effects. PL quantum yields of the solutions were estimated by comparing their spectrally integrated emission intensity with that of the standard. A dilute solution of quinine sulfate in 0.1 M H_2SO_4 exhibiting $53 \pm 2.3\%$ quantum yield at the excitation wavelength of 366 nm was used as a standard [23]. Continuous-wave excitation of UV LED (NICHIA NSHU590-B, optical power 1.5 mW) with the peak wavelength of 366 nm was used as an excitation source in PL and PL quantum yield measurements, whereas harmonically modulated excitation of the same LED was employed in PL lifetime measurements.

PL lifetimes of the bis-cyclometallated iridium(III) complex and of its ligand were measured in the frequency domain by comparing the excitation and PL signal phases and modulation amplitudes at frequencies in the range of 0.1–100 MHz. Current of the excitation source was harmonically modulated by high-power signal generator (Aeroflex IFR 2023A). The correspondingly modulated fluorescence response from the compounds under investigation was detected by Hamamatsu H6780-01 photomultiplier. The alternating current component of the signal was measured by radio frequency lock-in amplifier (SR844). Lifetimes (τ) were extracted by fitting the experimentally obtained modulation (m) and phase shift (ϕ) dependences versus frequency (ω) using the following relations [24]:

$$\phi(\omega) = \arctan\left\{\frac{N(\omega)}{D(\omega)}\right\} \qquad m(\omega) = \sqrt{N^2(\omega) + D^2(\omega)} \tag{1}$$

$$N(\omega) = \frac{\sum_i \alpha_i \omega \tau_i^2/(1+\omega^2\tau_i^2)}{\sum_i \alpha_i \tau_i}, \qquad D(\omega) = \frac{\sum_i \alpha_i \tau_i/(1+\omega^2\tau_i^2)}{\sum_i \alpha_i \tau_i} \tag{2}$$

Single ($i = 1$) and double-exponential ($i = 2$) decay models were used to describe experimental frequency responses by the least-squares analysis. Pre-exponential factors (α_i) were used to derive the fractional intensity (f_i) of τ_i by $f_i = \alpha_i\tau_i/\sum_i \alpha_i\tau_i$.

2.2. Materials

All chemicals were purchased from Aldrich and used as received without further purification. The details of the synthesis of the 2-phenyl-2*H*-[1,2,3]triazolo[4,5-*f*]quinoline (**4**) are described in Ref. [25].

The yield of **4** was 40%. ^{1}H NMR (300 MHz, DMSO-d$_6$, δ, ppm): 8.97 (d, *J* = 4.5 Hz, 1H, 1-H of Ht); 8.83 (d, *J* = 8.1 Hz, 1H, 3-H of Ht); 8.27 (d, *J* = 8.0 Hz, 2H, 2,6-H of Ph); 8.12 (d, *J* = 9.4 Hz, 1H, 7-H of Ht); 7.90 (d, *J* = 9.4 Hz, 1H, 6-H of Ht); 7.70 (q, *J* = 4.5 Hz, 2H, 3,5-H of Ph); 7.64 (t, *J* = 8.1 Hz, 1H, 2-H of Ht); 7.53 (t, *J* = 7.5 Hz, 1H, 4-H of Ph). Anal. Calcd for $C_{15}H_{10}N_4$ (%): C 73.16; H 4.09; N 22.75. Found (%): C 73.41; H 3.93; N 22.65.

2.3. Tetrakis(2-phenyl-2H-[1,2,3]triazolo[4,5-f]quinoline-N,C$^{2'}$)(μ-dichloro)diiridium

(**5**)

0.4 g (1.34 mmol) of iridium trichloride hydrate and 0.845 g (3.43 mmol) of 2-phenyl-2*H*-[1,2,3]triazolo[4,5-*f*]quinoline (**4**) were dissolved in the mixture of 2-ethoxyethanol (10 ml) and water (5 ml). The mixture was refluxed under argon atmosphere for 24 h, and cooled down to the room temperature. Obtained orange crystals were filtered, dried under vacuum, and used in the next step without additional purification. Yield of **5** was 80% (0.77 g).

2.4. Iridium(III)-bis(2-phenyl-2H-[1,2,3]triazolo[4,5-f]quinolinato-N,C$^{2'}$)acetylacetonate

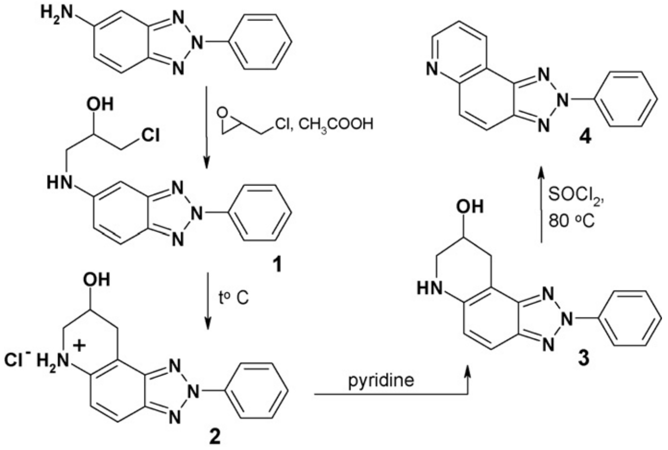
(**6**)

0.77 g (0.536 mmol) of chloro-bridged dimer **5** was mixed with 2,4-pentanedione 0.161 g (1.6 mmol) and sodium carbonate (0.568 g, 5.36 mmol) in 8 ml of degassed 2-ethoxyethanol. The mixture was refluxed under argon atmosphere for 3 h. After termination of the reaction (TLC control) obtained solution was treated with chloroform and washed with distilled water several times. The organic layer was dried over anhydrous $MgSO_4$, filtered and solvents were evaporated. The obtained residue was purified by column chromatography using acetone:*n*-hexane 5:20 as an eluent. Yield of **6** was 44% (0.37 g).

^{1}H NMR (300 MHz, CDCl$_3$, δ, ppm): 9.10–9.02 (m, 4H, 1, 3-H of Ht); 8.06 (d, *J* = 9.4 Hz, 2H, 7-H of Ht); 8.01–7.95 (m, 2H, 2-H of Ph); 7.90 (d, *J* = 9.4 Hz, 2H, 6-H of Ht); 7.69 (q, *J* = 4.6 Hz, 2H, 2-H of Ht); 7.05–6.96 (m, 2H, 3-H of Ph); 6.76–6.67 (m, 2H, 4-H of Ph); 6.23–6.16 (m, 2H, 5-H of Ph); 5.35 (s, 1H, CH of pentadione); 1.88 (s, 6H, CH$_3$). Elemental analysis. Calcd for $C_{35}H_{25}IrN_8O_2$ (%): C 53.77; H 3.22; N 14.33. Found (%): C 53.61; H 3.28; N 14.42.

3. Results and discussion

The synthesis route to the ligand **4** comprising of quinoline ring fused with 2-phenyl-1,2,3-triazole at the *f* edge is shown in



Scheme 1. Synthesis route to the phenyltriazole-quinoline-based ligand **4**.

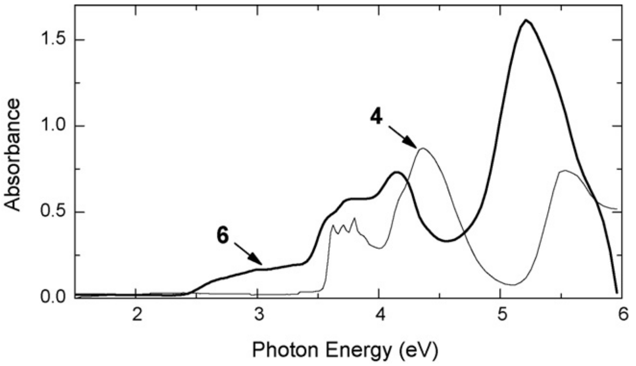


Fig. 1. UV–vis absorption spectra of bis-cyclometallated iridium complex **6** (thick line) and its free phenyltriazole-quinoline-based ligand **4** (thin line) in DMSO (10^{-4} M).

Scheme 1. The starting 5-amino-2-phenyl-[1,2,3]benzotriazole was alkylated with epichlorohydrin in the presence of catalyst acetic acid. The resulting 5-(3-chloro-2-hydroxypropylamino)-2-phenyl-2*H*-[1,2,3]benzotriazole (**1**) was then refluxed in chlorobenzene and undergone cyclization. Isolated intermediate hydrochloride **2** was treated with pyridine, and obtained 2-phenyl-6,7,8,9-tetrahydro-2*H*-[1,2,3]triazolo[4,5-*f*]quinolin-8-ol (**3**) was refluxed in the thionyl chloride to obtain the goal product **4** in 40% yield.

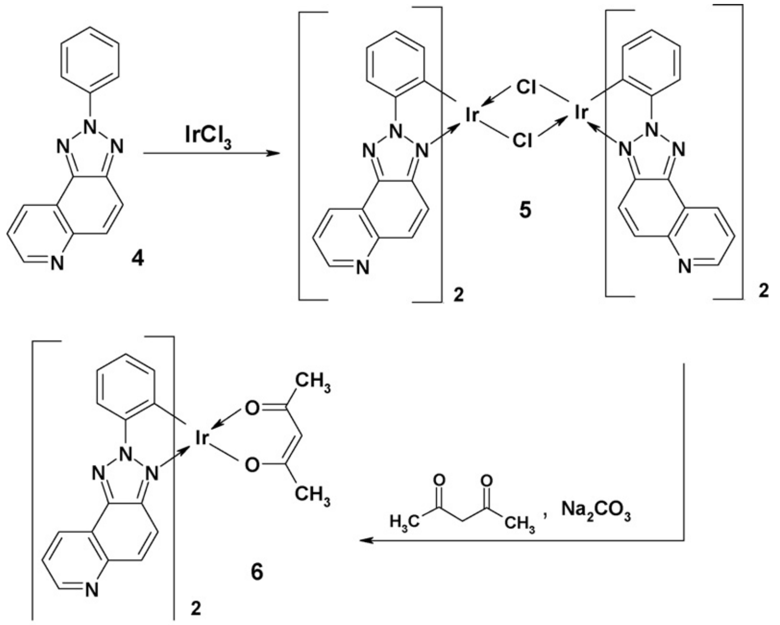
The synthesis of the bis-cyclometallated iridium complex **6** was carried out in two steps (Scheme 2). First, $IrCl_3 \cdot nH_2O$ reacted with an excess of ligand **4** to form a chloro-bridged dimer **5**. Then dimer **5** was easily converted to the desired product by replacing the bridging chloride with bidentate β-diketonate ligand (acetylacetone). All compounds were purified by column chromatography. The structures and purity of the synthesized compounds were confirmed by ^{1}H NMR and elemental analysis.

Absorption spectra of the DMSO solutions of bis-cyclometallated iridium complex **6** and the free ligand composed of 2-phenyl-1,2,3-triazole fused with quinoline ring **4** are shown in Fig. 1. By comparing the features in the absorption spectra, we attribute the bands above 3.5 eV in bis-cyclometallated iridium complex to the spin-allowed π–π^* transition of the bis-cyclometallated ligand.

An enhancement of absorbance in iridium complex below 3.5 eV, as compared with that of the free ligand, can be attributed to the transitions to singlet and triplet MLCT states, in analogy to those ascribed in similar quinoline ligand-based iridium complexes $(PPQ)_2Ir(acac)$, $(BPPQ)_2Ir(acac)$, etc. [11a,22]. The transition to the triplet MLCT is partly spin-allowed due to strong spin–orbit coupling of the 5d electrons of iridium [11a]. An admixture of $^3(\pi$–$\pi^*)$ ligand states to this long-wavelength absorption is also probable [11b]. Further evidence for significant mixing of the singlet and triplet excited states followed by enormous enhancement of emission efficiency from the triplet MLCT state is provided in PL spectra measured at room temperature.

Solid lines in Fig. 2 show PL spectra of helium degassed DMSO solutions of bis-cyclometallated iridium complex **6** and phenyltriazole-quinoline-based ligand **4** used for its synthesis. For comparison, the spectrum of air saturated solution of the iridium complex is also displayed by a dashed line.

PL spectrum of the free phenyltriazole-quinoline-based ligand **4** exhibits one broad band peaked at 2.85 eV due to emission



Scheme 2. Synthesis of the bis-cyclometallated iridium complex **6**.

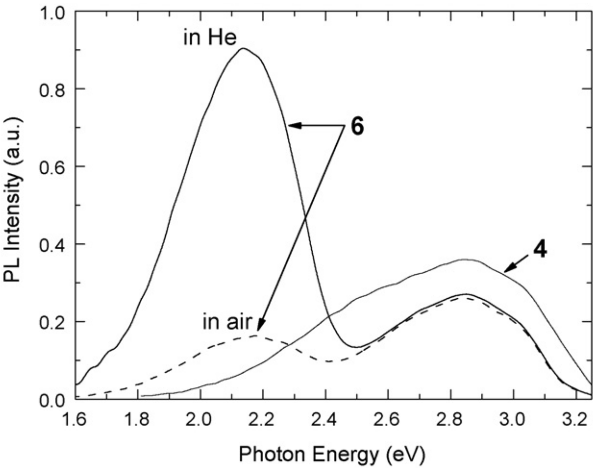


Fig. 2. Room temperature PL spectra of DMSO solutions (10^{-4} M) of bis-cyclometallated iridium complex **6** and its phenyltriazole-quinoline-based ligand **4**. Solid line, helium degassed solution; dashed line, air saturated solution.

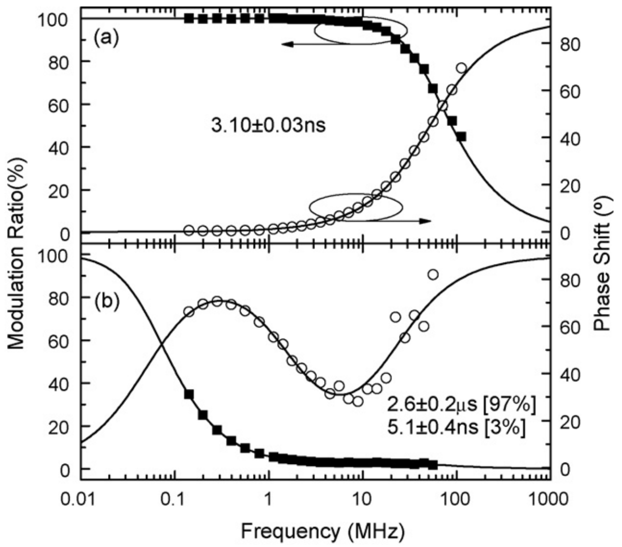


Fig. 3. PL phase shift (open points) and modulation ratio (solid points) as a function of modulation frequency in helium degassed DMSO solutions of phenyltriazole-quinoline-based ligand **4** (a) and bis-cyclometallated iridium complex **6** (b). Lines correspond to single (a) and double-exponential (b) decay modeling of the data with the characteristic lifetimes τ (indicated).

from the singlet excited state with the quantum yield of $39 \pm 2\%$, whereas the spectrum of iridium complex contains analogous ligand-based band and an additional lower-energy band at 2.13 eV. This lower-energy band is attributed to radiative recombination (phosphorescence) from triplet MLCT excited state, since it is present only in the PL spectrum of heavy metal complex. However, small admixture of ligand-based emission to phosphorescence from triplet MLCT excited state is possible, because of the small overlap of these two bands. Presence of the ligand-based emission band in the PL spectrum of iridium complex is caused by the direct excitation of lowest excited states of the ligand by UV-LED ($\sim$3.4 eV). Preliminary PL data on the spin-casted films of bis-cyclometallated iridium complexes show no traces of the ligand-based emission indicating very efficient excitation energy transfer to triplet MLCT states.

Helium degassing of the solution results in sixfold boost in phosphorescence intensity of bis-cyclometallated iridium complex **6** (see Fig. 2), indicating significant reduction of molecular oxygen, which is known to effectively quench the emission from excited triplet states [26]. The phosphorescence band is broad and without vibronic modes supporting the lowest excited triplet state of the complex to originate mainly due to triplet MLCT excited state [11a,19a,22]. PL quantum yield of our bis-cyclometallated iridium complex possessing phenyltriazole-quinoline-based ligand is estimated to be of $78 \pm 3\%$, which is four times higher than that reported for the similar bis-cyclometallated iridium complexes based on quinoline ligands: 2,4-diphenylquinoline, 2-(4-methoxyphenyl)-4-phenylquinoline, *N*,*N*-diphenyl-*N*-[4-(4-phenylquinol-2-yl)phenyl]amine, 2-(4-fluorophenyl)-4-phenylquinoline and 2-(diphen-4-yl)-4-phenylquinoline ligands [22]. The emission of the iridium complex displayed Commission Internationale d'Eclairage (CIE) chromaticity coordinates of (0.47, 0.51), which refer to the yellow part of the CIE diagram.

The emission lifetimes of the two PL bands, i.e. ligand-based band and the band related to triplet MLCT excited state, shown in Fig. 2, was determined by measuring frequency dependences of the phase shift (open points) and modulation ratio (solid points) at the band peaks. Measured frequency responses for the free phenyltriazole-quinoline-based ligand **4** and bis-cyclometallated iridium complex **6** are depicted in Fig. 3(a) and (b), respectively. The lifetimes were extracted by fitting the experimental data with the formulae (1) and (2). The free ligand-based emission features single-exponential decay with nanosecond lifetime ($\tau = 3.1$ ns), whereas the decay of the triplet MLCT excited state is double-exponential with dominant characteristic τ of 2.6 μs and the weaker component of τ of the order of 5 ns. The estimated microsecond lifetime in bis-cyclometallated iridium complex verifies our assumption on the origin of the lower-energy emission band to arise from the triplet MLCT excited state. Much shorter lifetime of the weaker component (with fractional intensity of only 3%) clearly implies the small contribution from the ligand-based singlet states, which decay on the nanosecond time scale. We stress that the phase shift and modulation ratio yield independent values of τ, which upon coincidence proves the reliability of the lifetime measurements.

Estimated microsecond emission lifetime (2.6 μs) of the triplet MLCT excited state in our bis-cyclometallated iridium complex is consistent with the lifetime (1–14 μs) reported for the family of $C\hat{N}_2$Ir(acac) complexes [11a], which, however, exhibit maximal quantum yield of only 60%.

4. Conclusions

High efficiency phosphorescent bis-cyclometallated iridium(III) complex based on novel triazole-quinoline ligand has been synthesized and optically characterized. The new complex is shown to exhibit an enhanced long-wavelength absorption, below the $^3(\pi-\pi^*)$ transitions, and microsecond lifetime (2.6 μs) of phosphorescence readily quenched by oxygen. The observed features support the dominant MLCT nature of the excited triplet states involved in the transition, which is known to be partly allowed due to strong spin–orbit coupling of the 5d electrons of iridium. The revealed properties along with the high phosphorescence quantum yield (78%) advise the new bis-cyclometallated iridium(III) complex, based on the novel triazole-quinoline ligand, as an efficient yellow triplet-emitter for OLEDs.

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