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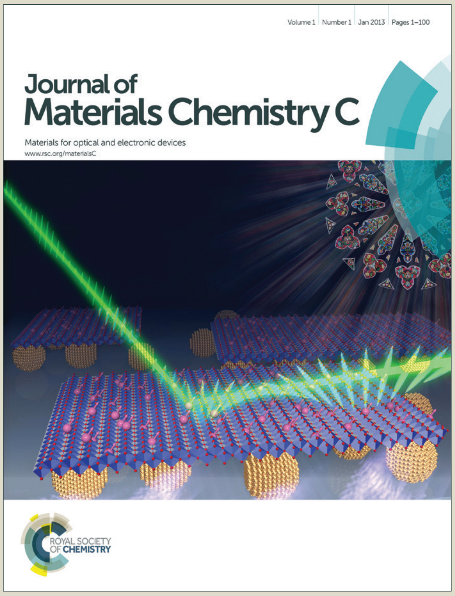

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Highly Efficient, Little Efficiency Roll-Off Orange-Red Electrophosphorescent Devices Over Wide Range of Doping Concentration Based on A Bipolar Transporting Iridium Complex

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A novel phosphorescent emitter BZQPG possessing the bipolar charge transporting ability realizes the most efficient orange-red electroluminescence with the power efficiency of 77.1 lm W^{-1} and external quantum efficiency of 27.3% together with low efficiency roll-off (>50 lm W^{-1} & 26% @ 5000 cd m^{-2}).

Since the initial report by Baldo et al.,[1] phosphorescent organic light-emitting device (PhOLED) becomes a major focus of OLED research nowadays because of its producing theoretically 100% internal quantum efficiency (IQE), which is achieved by harvesting both singlet and triplet excitons through energy transfer from the host to the dopant emitter. Recently, orange or red electrophosphorescent emitters are drawing more and more attention due to their important role in constructing white OLEDs and lots of efficient orange or red PhOLEDs have been achieved.[2] Nevertheless, the EL performances of the true orange-red or red phosphorescent emitters that meet the standard of $\lambda_{max}^{EL} \geq 580$ nm and the Commission Internationale de L'Eclairage ($CIE_{x,y}$) coordinates of $x \geq 0.55$ and $y \leq 0.45$,[3] which are essential for realizing the high-quality white OLEDs with high color rendering index (CRI) values (≥ 80),[4] are still far from satisfactory for their moderate power efficiency (PE, <50 lm/W) and significant efficiency roll-off.

Recently, the emission mechanism of hole and/or electron injection into the highest occupied molecular orbital (HOMO) and/or the lowest unoccupied molecular orbital (LUMO) of the dopant and then direct charge recombination in the dopant, is considered more efficient for the formation of excitons than host-to-dopant energy transfer.[5] Obviously, this direct charge recombination process can eliminate not only the need to overcome the large energy barriers between the charge transport layers and the emitting layer (EML) resulted from the wide HOMO−LUMO gap of the host, but also the energy losses during the exothermic host-dopant energy transfer process, which has led to several very high-performance blue, green and yellow PhOLEDs using simple device configuration and/or easily controlled fabrication processes.[5e,6] Unfortunately, although there are a certain number of papers on the orange-red or red OLEDs that can partly benefited from the direct-charge-recombination mechanism, their EL efficiencies were lower than 15% for the external quantum efficiency (EQE) and 20 lm/W for PE,[7] due to the inherently poor or the unipolar electron- or hole-transporting ability of the corresponding phosphorescent emitters. Therefore, it is still a considerable challenge to further develop new orange-red or red phosphors that can give full play to the advantage of the direct-charge-recombination mechanism, and thus realize the highest possible EL performance.

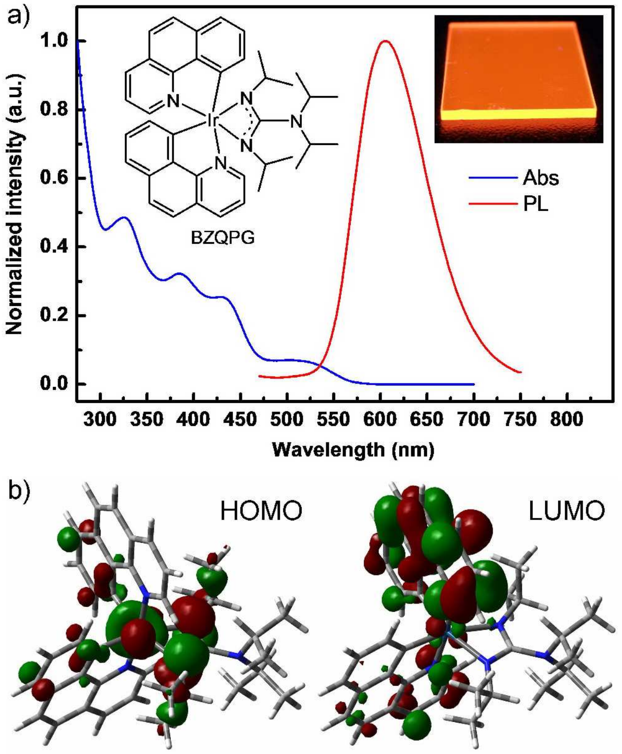

Fig. 1 a) UV-Vis absorption and PL spectra of BZQPG in neat thin film. Inset: the chemical structure and the fluorescence image of BZQPG. b) Contour plots of HOMO and LUMO in BZQPG from DFT calculations.

It is well understood that the charge-transporting properties of the phosphorescent complexes are strongly dependent on the character of their frontier orbitals. In most current mixed-ligand iridium complexes Ir(C^N)$_2$(L^X) (C^N = anionic bidentate cyclometallating ligands and L^X = anionic ancillary ligands), density functional theory (DFT) calculations indicate that the HOMO and LUMO have largely overlapping localized on the heterocyclic rings of the C^N ligands. However, the spatial separation of the HOMO and LUMO distribution as much as possible, is indeed conductive to endow the resulting molecule with the bipolar charge-transporting ability.[7] In this communication, we report a novel orange-red Ir^{III} complex $(bzq)_2Ir(dipig)$ (BZQPG) constructed by a C^N ligand of bis(7,8-benzoquinolinato) (bzq) together with a L^X ligand of *N*,*N*′-diisopropyl-diisopropyl-guanidinate (dipig) (see **Fig. 1a inset**). As we expected, this molecule presents clear separation of the HOMO and LUMO distribution *via* DFT calculations, where the HOMO mainly consists of the dipig ligand (54.6%) and *d*-orbitals of the Ir atom (27.2%), while the LUMO largely located on the bzq ligand (96%) (**Fig. 1b**). This is a rather rare character of frontier orbitals for the orange and/or red phosphorescent complexes. Hence, BZQPG possesses sufficient hole- and electron-transporting abilities for fulfilling the carrier conduction, which can effectively balance the distribution of excitons and broaden the recombination zone in the EMLs and consequently improve the EL efficiency and reduce the efficiency roll-off of the obtained PhOLEDs. Here, series of orange-red PhOLEDs ($\lambda_{max}{}^{EL}$~588-600 nm) that employ the wide-range doping concentrations of 15, 20 and 25 wt%, exhibited similar CIE coordinates (0.57±0.01, 0.41±0.01) together with constantly high peak PE and EQE values (>60 lm W^{-1} & 22%). The optimum device performance is achieved in the OLED with the doping concentration of 20 wt%, which shows very high maximum EQE and PE values of 27.3% and 77.1 lm/W respectively, with the high levels of 26% and 50 lm/W retained at high luminance of 5000 cd m^{-2}. Even when the luminance reaches 50000 cd m^{-2}, the EQE still more than 20%. To the best of our knowledge, these efficiencies are the highest reported for all the orange and red OLEDs.[2,3,8]

The new complex BZQPG was synthesized in high yields following precedents for analogs[6b] and further purified twice through vacuum sublimation. Its chemical structure was confirmed by ^1H NMR spectroscopy, mass spectrometry and elemental analysis. The UV/Vis absorption and photoluminescence (PL) spectra of BZQPG in neat thin film are shown in **Fig. 1a**, which shows strong orange-red PL emission at λ_{max} 605 nm with a quantum yield of 0.26±0.02 in air as the fluorescence image (**Fig. 1a inset**). The corresponding triplet energies (T_1) were estimated to be BZQPG ~2.1 eV (λ=605 nm) from the 0−0 band of the phosphorescence spectrum. The short phosphorescence lifetime of BZQPG in toluene solution is 0.14 μs, which suggests that efficient spin−orbit coupling leads to intersystem crossing from the singlet to triplet states in this complex. In addition, such short lifetime is beneficial for increasing spin-state mixing and reducing the roll-off in EL efficiency.[9]

The HOMO level of BZQPG was 4.9 eV based on the oxidation onset of the CV curve (see S-Fig. 3 in *Supporting Information-S.I.*). The bandgap (E_g) of BZQPG was 2.2 eV calculated from the absorption edge of the UV-vis spectrum, and the LUMO level was 2.7 eV by considering the difference between the HOMO and E_g values. As shown in **Fig. 2a**, the HOMO/LUMO levels of BZQPG are well matched with those of common charge transport materials used below: 4,4′-bis(N-(1-naphthyl)-N-phenylamino)biphenyl (NPB) for holes and 1,3,5-tris(N-phenylbenzimidazol-2-yl)benzene (TPBi) for electron. Obviously, because the HOMO level of the fluorescent host material 4,4′-*N*,*N*’-dicarbazolylbiphenyl (CBP) is much deeper than that of BZQPG, the holes can inject predominantly from the HOMO of NPB into the HOMO of BZQPG rather than that of CBP, while the electrons possibly inject through the LUMO of either BZQPG or CBP as a result of no energy barrier to overcome from the LUMO of TPBi to that of BZQPG and/or CBP, although little deeper LUMO (0.3 eV) of CBP than that of BZQPG. On the other hand, BZQPG shows the comparable hole and electron mobilities of 2.9 × 10^{-3} and 2.3 × 10^{-3} cm^2 V^{-1} s^{-1} respectively (see S-Fig. 4 in *S.I.*), which are rather high levels for a phosphorescent emitter, even are similar to or much higher than those of the typical charge transport materials including NPB (3.9 × 10^{-4} cm^2 V^{-1} s^{-1} for hole) and TPBi (9.6 × 10^{-6} cm^2 V^{-1} s^{-1} for electron) measured under the same experimental conditions. Literature values for NPB are ~10^{-4} cm^2 V^{-1} s^{-1} and for TPBi are ~10^{-5} cm^2 V^{-1} s^{-1}.[10,11] Therefore, highly efficient recombination of hole and electron is expected based on charge injection into and balance transport in the EML through the bipolar BZQPG molecules mainly, where the holes and electrons can realize intermolecular hopping smoothly along the respective transporting channels.

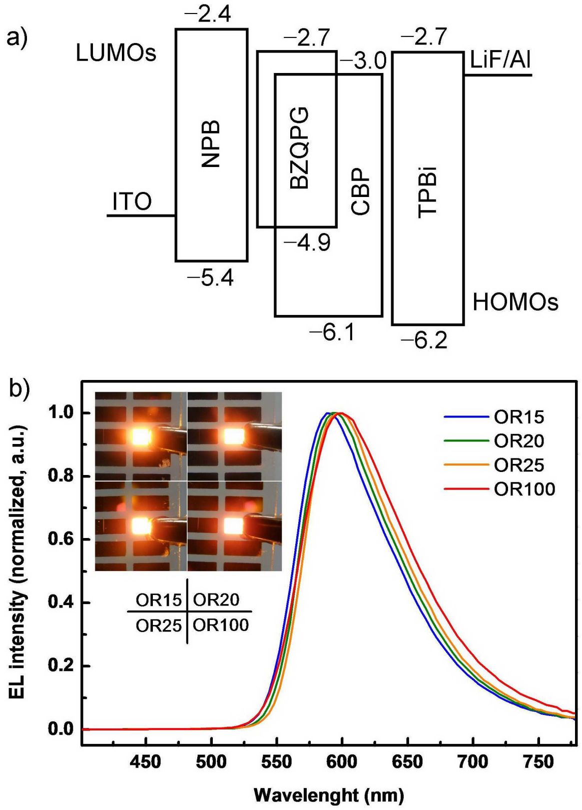

Fig. 2 a) Proposed energy diagram of the materials used in PhOLEDs. b) EL spectra of PhOLEDs at the luminance of 5000 cd m^{-2}. Inset: photograph of PhOLEDs at the luminance of 5000 cd m^{-2}.

To explore the EL performance of BZQPG, four devices were fabricated with the same configuration of [ITO/NPB (40 nm)/emitting layer (30 nm)/TPBI (30 nm)/LiF (0.5 nm)/Al]. In each device, NPB served as the hole-transport layer (HTL) and TPBi as the electron-transport layer (ETL), with ITO (indium-tin oxide) anode and LiF/Al cathode. The devices using doped CBP: BZQPG (15, 20 and 25 wt% BZQPG) films and neat BZQPG film as the EMLs, which are denoted as OR15, OR20, OR25 and OR100, respectively. All the devices showed stable orange-red EL emission within the whole range of the corresponding driving voltages, and no host emission is observed in the EL spectra of all the doped devices. Furthermore, it is desired that the photographs of four devices recorded at a brightness of 5000 cd m^{-2} exhibited the almost same emitting color as shown in **Fig. 2b (inset)**, which are consistant with their nearly overlapping EL curves (**Fig. 2b**) with the $\lambda_{\max}^{\mathrm{EL}}$ 588-600 nm from BZQPG as well as their similar CIE coordinates of (0.57±0.01, 0.41±0.01).

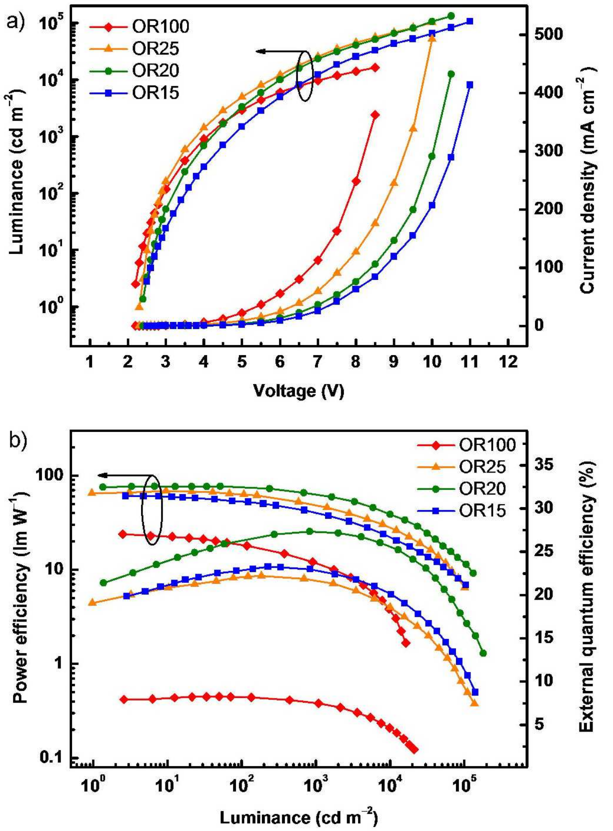

Fig. 3. a) Current density-voltage-luminance (*J-V-L*) curves and b) power efficiency-luminance-external quantum efficiency curves of OR15, OR20, OR25 and OR100.

The current density-voltage-luminance (*J-V-L*) and EL efficiency-luminance (PE/EQE-*L*) characteristics are shown in **Fig. 3a** and **3b**, and the EL data are summarized in **Table 1**. Four devices displayed low driving voltages based on the rapidly increasing *J-V* and *L-V* curves after the onset. The turn-on voltages (V_{on} at 1 cd m^{-2}) of OR15, OR20, OR25 and OR100 are as low as 2.4, 2.4, 2.3 and 2.2 V. The driving voltages at the practical luminance of 1000 cd m^{-2} are 4.5 V, 4.2, and 3.7 V and 4.1 V, respectively. At driving voltages of 6.1, 5.3, 4.8 and 5.7 V for OR15, OR20, OR25 and OR100, very high luminance values of >5000 cd m^{-2} were obtained. The ideal operating voltage with the speedy increase of current and luminance indicates that the hole and electron should inject into the EMLs through the HOMO of BZQPG and the LUMO of BZQPG and/or CBP respectively, due to their cascaded energy level diagram (**Fig. 2a**). Accordingly, the reduced driving voltages with increasing the doping concentration of BZQPG in the devices from OR15 to OR25, are explained by more and more charge injection/transport through the bipolar BZQPG molecules instead of CBP gradually in these EMLs. The increasing BZQPG content can reduce the hopping distance, further enhancing the charge transport followed by exciton formation. This hypothesis is strongly supported by the *J-V* characteristics of the corresponding electron- and hole-only single-carrier devices (see S-Fig. 5 in *S.I.*), where both the hole and electron current are greatly increased when BZQPG is doped into CBP. This further confirms that in the EMLs of CBP-host devices, the BZQPG molecules provide a more favorable charge-transport channel than the CBP molecules, which is due to the narrower energy gap and more balanced charge-transport ability of BZQPG than that of CBP.

Table 1. EL data of devices OR15, OR20, OR25 and OR100.

Device	OR15	OR20	OR25	OR100
$V_{\text{turn-on}}$ (V)^a	2.5	2.4	2.3	2.2
$L_{\max}$ /cd m^{-2}	105300	132940	101500	16350
(V at $L_{\max}$)	(11)	(10.5)	(10)	(8.5)
PE^b /lm W^{-1}	60.5, 40.6, 27.8	77.1, 63.2, 50.2	68.8, 49.1, 34.3	23.8, 11.6, 6.3
EQE^b /%	23.3, 22.6, 20.8	27.3, 27.2, 26.3	22.2, 21.6, 20.1	8.3, 7.3, 5.5
EL $\lambda_{\max}$^{c} /nm	588	592	596	600
CIE $(x,y)^c$	0.56, 0.42	0.57, 0.42	0.58, 0.41	0.58, 0.40

^{a}Recorded at 1 cd m^{-2}. ^{b}In the order of maximum, then values at 1000 and 5000 cd m^{-2}. ^{c}Measured at 5000 cd m^{-2}.

The device OR100 based on the neat BZQPG EML showed the highly efficient orange-red EL emission with the maximum PE of 23.8 lm W^{-1} and EQE of 8.3%, which is a strong evidence for that the BZQPG complex is able to realize the electrophosphorescence through the direct exciton formation, because it is the only possible mechanism occured in the neat EML without a host. This indicates the exceptional multifunctional character of this phosphor, including the balanced charge transport property, high quantum yield and short excited state lifetime in the solid state. Furthemore, desired EL efficiencies have been achieved for the doped devices OR15, OR20 and OR25 as shown in **Fig. 3b** and **Table 1**. Three devices above achieve maximum PE and EQE values of more than 60 lm W^{-1} and 22% respectively by employing such wide doping concentration as 15, 20 and 25 wt%. To the best of our knowledge, these EL efficiencies exceed any previously reported values for the OLEDs with the true orange-red CIE coordinates (x≥0.55 and y≤0.45).[3] Among them, OR20 adopting the optimized concentration of 20 wt% reaches the very high EL efficiencies of 77.1 lm W^{-1} and 27.3%, Moreover, the efficiency roll-off of them is low upon increasing the luminance and current density. The PE and EQE values at a practical luminance of 1000 cd m^{-2} maintained the very high levels of 63.2 lm W^{-1} and 27.2%, respectively. What is more, >95% of the maximum EQE is maintained up to the extremely high luminance of 5000 cd m^{-2}, and the EQE values remain >20% over the very wide luminance range of 1–50000 cd m^{-2}, corresponding to wide current density ranges of ca. 0.002–100 mA cm^{-2}. Obviously, These ultra-high efficiencies and remarkably low roll-off are considerably better than all the OLEDs emitting the yellow-orange, orange and orange-red colors ever reported.[2,3,8]

These desirable EL data demonstrate that TTA processes, especially at high luminance levels, have been greatly reduced. This results from the balanced charge fluxes and broader recombination zones within the EMLs of OR15, OR20 and OR25, where the content of BZQPG molecules is high enough to perform the main function on hole/electron transport due to the bipolar charge transporting ability, and then emit predominantly through direct exciton recombination. It can be seen from **Fig. 3b** that OR25 showed slightly lower EQE and higher roll-off than OR20. This implies slightly more TTA quenching in OR25 than in OR20, which is supported by the lower luminance of OR25 than of OR20 at a constant current density (see S-Fig. 6 in *S.I.*). In OR20, the optimal content of BZQPG (20 wt%) achieved a balance between the enhancing exciton density and the gradual emerging TTA within the EML along with the increasing BZQPG concentrations from 15 to 25 wt%, and even 100 wt% in OR100. That is to say, the higher content of BZQPG in OR25 and OR100, results in excess triplet formation directly on the emitting molecules, leading to increased TTA and thus decreased luminance and EQE values. Nevertheless, maintaining such high and stable EL performance within a certain doping concentration range as exhibited in OR15, OR20 and OR25, is also a rare advantage of BZQPG as a novel orange-red phosphorescent emitter, indicating an easily controlled process for fabricating highly efficient PhOLEDs.

In summary, we have successfully prepared a bipolar orange-red Ir^{III} complex (bzq)$_2$Ir(dipig) by incorporating a well-chosen guanidinate group (dipig) as an ancillary ligand with a phosporescent "(bzq)$_2$Ir" species. The newly developed phosphorescent emitter exhibited similar CIE coordinates (0.57±0.01, 0.41±0.01) and constantly high peak PE and EQE values (>60 lm W^{-1} & 22%) in series of PhOLEDs by adopting the wide-range doping concentrations of 15, 20 and 25 wt% respectively. Among them, the highest PE and EQE values of 77.1 lm W^{-1} and 27.3% were realized respectively. To the best of our knowledge, these EL efficiencies exceed any previously reported values for orange-red OLEDs. It has been demonstrated that such excellent EL performance is attributed to the superior hole-/electron-transporting ability of BZQPG, which can ensure the balanced charge fluxes with the exciton recombination regions are broadened as wide as the whole EML, and in turn significantly decreasing the probability of TTA. Obviously, developing the bipolar phosphorescent emitters is really an effective strategy for realizing the efficient PhOLEDs through direct charge recombination. Meanwhile, the long lifetime of these devices is worth expecting because of their many advantages such as low driving voltages, high efficiency and little efficiency roll-off by improving operating conditions.

Notes and references

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† Electronic Supplementary Information (ESI) available: [photophysical property of compound PPI under same condition]. See DOI: 10.1039/b000000x/

‡ Footnotes should appear here. These might include comments relevant to but not central to the matter under discussion, limited experimental and spectral data, and crystallographic data.

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Graphic Abstract

Highly Efficient, Little Efficiency Roll-Off Orange-Red Electrophosphorescent Devices Over Wide Range of Doping Concentration Based on A Bipolar Transporting Iridium Complex

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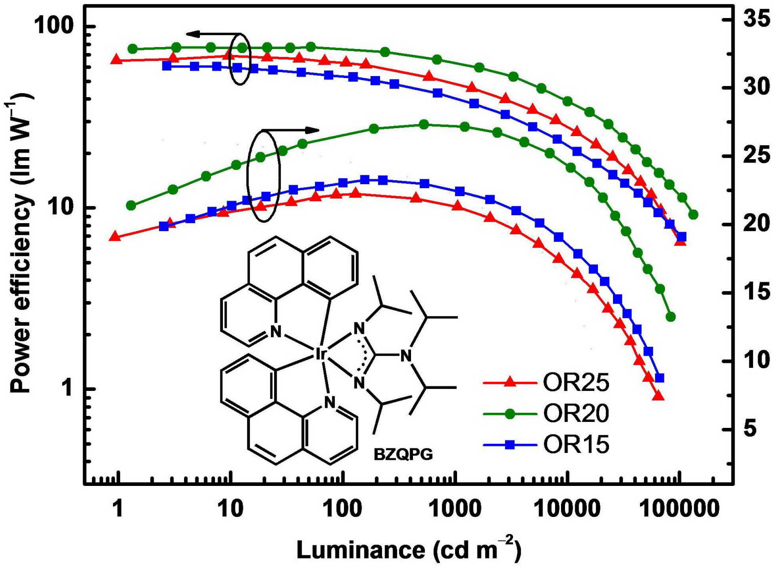

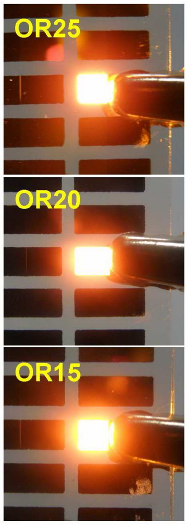

A novel orange-red emitting iridium complex BZQPG possessing sufficient and balanced hole- and electron-transporting abilities has been used to fabricate series of highly efficient PhOLEDs OR15, OR20 and OR25, where this complex is doped in a common host CBP with the wide-range doping concentrations of 15, 20 and 25 wt% respectively as the EMLs. They exhibited stable emitting color with similar CIE coordinates (0.57±0.01, 0.41±0.01) and constantly high peak PE and EQE values of >60 lm W^{-1} and 22% respectively. The highest EL efficiencies of 77.1 lm W^{-1} and 27.3% together with low efficiency roll-off (>60 lm W^{-1} / 27% @ 1000 cd m^{-2} and >50 lm W^{-1} / 26% @ 5000 cd m^{-2}) were realized in OR20 with the optimized doping concentration of 20 wt%, which exceed any previously reported values for the orange-red OLEDs.