

# Photoluminescence and electroluminescence properties of three ternary lutetium complexes

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Three lutetium complexes, Lu(PMIP)<sub>3</sub>(TPPO)<sub>2</sub> (**A**), Lu(PMIP)<sub>3</sub>Bipy (**B**) and Lu(PMIP)<sub>3</sub>Phen (**C**) (where PMIP, TPPO, Bipy and Phen stand for 1-phenyl-3-methyl-4-isobutyl-5-pyrazolone, triphenylphosphin oxide, 2,2'-bipyridine and 1,10-phenanthroline, respectively), were synthesized and complex **A** was characterized by single-crystal X-ray diffraction analysis. Complex **A** was found to crystallize in the *P2<sub>1</sub>/n* (14) space group. The photoluminescence spectra of the complexes reveal that all the complexes have a similar spectrum peaking near 440 nm; however, the intensity of the spectrum of complex **A** is about 100 times higher than that of the other two complexes. When these complexes were utilized to prepare light-emitting devices, that with complex **A** gave a blue light, originating from this complex, with the highest brightness of 119 cd m<sup>-2</sup> at an applied voltage of 19 V, while the same configuration devices using the other two complexes exhibited green light peaking at 512 nm, which arises from exciplexes formed at the interface of TPD and the corresponding lutetium complex. A maximum luminance of 1010 cd m<sup>-2</sup> at 16 V from the exciplex was obtained with the highest power efficiency of 0.13 lm W<sup>-1</sup> at 9 V and a turn-on voltage as low as 3 V for the device with a (ITO)/TPD(10 nm)/B(50 nm)/BCP(20 nm)/AlQ(40 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub>(100 nm)/Ag (100 nm) configuration.

## Introduction

Organic light-emitting devices (OLEDs) are of great interest because of their efficient emission in the visible region and their possible application in the fabrication of flat panel full color displays.<sup>1–6</sup> Rare earth complexes, as organic light-emitting materials, can be classified as either central-ion-emission or ligand-emission complexes. The central-ion-emission complexes, especially europium and terbium β-diketonates, have aroused considerable attention due to their sharp emission band and theoretical 100% quantum efficiency.<sup>7–9</sup> Some rare earth complexes, such as those of Y, La, Gd and Lu with the 4f electronic configuration of their central ions being empty, half-filled or filled, have no emission in the visible light region and therefore belong to the group of ligand-emission complexes, just like the complex AlQ. As electroluminescence materials, these kinds of rare earth complexes have been studied relatively rarely. In our group, devices based on Y(PMIP)<sub>3</sub>Bipy,<sup>10</sup> Gd(PMIP)<sub>3</sub>Bipy<sup>11</sup> and Gd(PMIP)<sub>3</sub>Phen<sup>12</sup> have been fabricated and reported.

In this paper, the synthesis of three ternary lutetium complexes, tris(1-phenyl-3-methyl-4-isobutyl-5-pyrazolone)bis(triphenylphosphin oxide) lutetium, Lu(PMIP)<sub>3</sub>(TPPO)<sub>2</sub> (**A**), tris(1-phenyl-3-methyl-4-isobutyl-5-pyrazolone)-2,2'-bipyridine lutetium, Lu(PMIP)<sub>3</sub>Bipy (**B**), and tris(1-phenyl-3-methyl-4-isobutyl-5-pyrazolone)-1,10-phenanthroline lutetium, Lu(PMIP)<sub>3</sub>Phen (**C**), and a study of their photoluminescence (PL) and electroluminescence (EL) properties is reported.

## Experimental

### Materials

1-Phenyl-3-methyl-4-isobutyl-5-pyrazolone (PMIP) was synthesized in our laboratory.<sup>8</sup> The hole transport material *N,N'*-diphenyl-*N,N'*-bis(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine (TPD, 99%) was purchased from Aldrich and the hole blocking material 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (BCP, 98%) was obtained from Acros; they were used as supplied. Tris(8-hydroxyquinolato) aluminum (AlQ) was synthesized in our lab and sublimed two times before use. Indium tin oxide (ITO) glass substrate with a sheet resistance of 15 Ω cm<sup>-2</sup> was kindly sent by China Southern Glass Holding Co. Ltd. as a gift.

### Synthesis

The complexes (molecular structures shown in Fig. 1) were synthesized by the method reported previously.<sup>8</sup> For example: for Lu(PMIP)<sub>3</sub>(TPPO)<sub>2</sub>, to a 50 ml ethanol (95%) solution containing 1 mmol LuCl<sub>3</sub>, 3 mmol PMIP and 2 mmol TPPO, 3 ml of 1.0 mol L<sup>-1</sup> NaOH was added dropwise under stirring and the solution was refluxed for 24 hours. The resulting solution was filtered and a colorless crystal was obtained after 2 days. It was purified by recrystallization from ethanol solution with a yield 85%. M.p.: 158–159 °C. Anal. found (%): C 64.23; H 5.21; N 5.75; calcd (%) for C<sub>78</sub>H<sub>75</sub>N<sub>6</sub>O<sub>8</sub>P<sub>2</sub>Lu: C 63.90; H 5.16; N 5.75. Complexes **B** and **C** were prepared and recrystallized by the same procedure from ethanol–chloroform (2:1

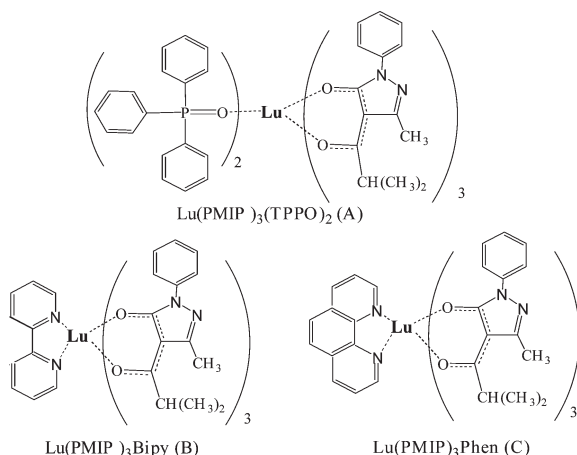


Fig. 1 Chemical structures of the complexes.

V/V) solution with yields higher than 80%. Lu(PMIP)<sub>3</sub>Bipy (m.p.: 254–255 °C): Anal. found (%): C 58.31; H 5.05; N 10.06; calcd (%) for C<sub>52</sub>H<sub>53</sub>N<sub>8</sub>O<sub>6</sub>Lu: C 58.80; H 4.99; N 10.55. Lu(PMIP)<sub>3</sub>Phen (m.p.: 324–326 °C): Anal. found (%): C 59.14; H 4.63; N 9.85; calcd (%) for C<sub>54</sub>H<sub>53</sub>N<sub>8</sub>O<sub>6</sub>Lu: C 59.71; H 4.92; N 10.32.

### Crystallography

A crystal of complex **A** was mounted on glass fiber and transferred to a RIGAKU RAXIS RAPID imaging plate diffractometer with graphite-monochromated Mo K $\alpha$  radiation ( $\lambda = 0.71073$  Å). The structure was solved by Patterson methods (SHELXS-97) and refined by full-matrix least-squares methods using the program SHELXL-97. All non-hydrogen atoms were refined anisotropically. The hydrogen atom positions were calculated in ideal geometries. The final cycle of full-matrix least-squares refinement was based on 10 676 observed reflections [ $I > 2.0\sigma(I)$ ] and 857 variable parameters and converged with  $R_1 = 0.0698$ ,  $wR_2 = 0.0778$ . The maximum and minimum peaks on the final differential Fourier map were 0.863 and  $-0.630$  e Å<sup>-3</sup>.

CCDC reference number 198777. See <http://www.rsc.org/suppdata/nj/b3/b304451c/> for crystallographic data files in CIF or other electronic format.

### Preparation of EL devices

Organic layers were sequentially deposited in one run by high vacuum ( $9 \times 10^{-4}$  Pa) thermal evaporation onto a pre-cleaned ITO glass substrate. A shadow mask with 5 mm diameter openings was used to define the cathode of a 200 nm thick layer of Mg<sub>0.9</sub>Ag<sub>0.1</sub> alloy, with a 100 nm thick Ag cap. The thickness of the deposited layer and the evaporation speed of the individual materials were monitored in vacuum with quartz crystal monitors. The deposition rates were maintained to be 0.1–0.3 nm s<sup>-1</sup> for organic materials and 1.0–1.3 nm s<sup>-1</sup> for the Mg<sub>0.9</sub>:Ag<sub>0.1</sub> alloy.

### Apparatus

FT-IR spectra were taken on a Nicolet MAGNA-IR spectrometer. The photoluminescence and electroluminescence spectra were measured on a Hitachi F-4500 fluorescence spectrophotometer. UV-visible absorption spectra were recorded using a Shimadzu 3100 UV-VIS-NIR spectrophotometer. The brightness was determined by a ST-86LA spot photometer and a close-up lens with a focal spot of 5 mm in diameter. The layer thickness was measured by a Dektak sur-

face profile measuring system and an IL-1000 quartz crystal monitor. Synchrotron radiation photon-electron spectroscopy (SRPES) was detected by a photon-electron spectrometer (*vs* Scientific Instruments, Ltd.) through synchrotron radiation under ultrahigh vacuum ( $3\text{--}4 \times 10^{-8}$  Pa). The photon-electron spectrum was recorded under 32.7 eV synchrotron radiation light.

## Results and discussion

### Crystallography

The crystallographic data for complex **A** are listed in Table 1. An ORTEP diagram for the common repeating unit is shown in Fig. 2. The lutetium metal ion is octa-coordinated and the geometry is a distorted bicapped trigonal prism. The PMIP oxygen atoms [O(2), O(5), O(4), O(1)] and two TPPO oxygen atoms [O(7), O(8)] form the trigonal prism. Another two oxygen atoms [O(3) and O(6)] cap the two quadrilateral faces O(1)–O(4)–O(8)–O(2) and O(7)–O(4)–O(8)–O(5). The average Lu–O distance is 2.304 Å [2.211(2)–2.453(3) Å], which is little smaller than the sum of radii of Lu<sup>3+</sup> (0.977 Å, eight coordinated) and O<sup>2-</sup> (1.42 Å).<sup>13</sup>

### Film formation properties

All three complexes can be evaporated to form homogeneous ultrathin films. As an example, FT-IR spectra of complex **A** as an 80 nm thick film evaporated on Si substrate and in the solid state are shown in Fig. 3. It is clear that the two spectra are identical with each other, indicating the complex is not decomposed during evaporation. Complexes **B** and **C** show the same behavior.

### UV-Vis spectra

UV-Vis spectra of the three complexes, the neutral ligands and NaPMIP are shown in Fig. 4(a). The absorbance of TPPO is mainly located in the higher energy range (less than 240 nm), while the absorbances of Bipy and Phen are in a much lower energy range, from 300 to 215 nm. However, in the ground state, the UV-Vis spectra of all the three complexes are just the sum of those of PMIP<sup>-</sup> and the corresponding neutral ligand, no obvious change is observed.

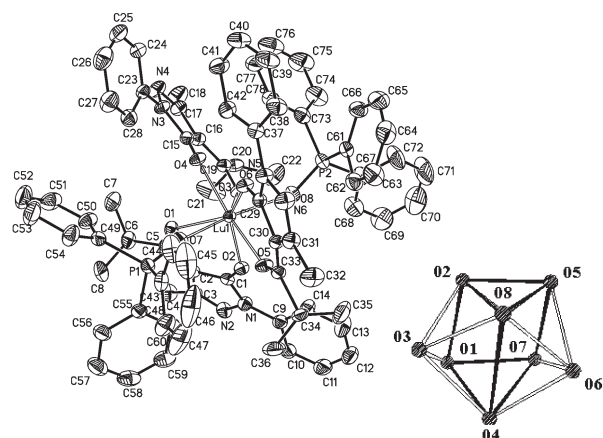
### PL properties

Normalized PL spectra (excitation and emission) of the complexes are shown in Fig. 4(b). All three complexes show similar emission spectra peaking near 440 nm; however, their intensity

Table 1 Crystallographic data for Lu(PMIP)<sub>3</sub>(TPPO)<sub>2</sub>

Empirical formula	C <sub>78</sub> H <sub>75</sub> N <sub>6</sub> O <sub>8</sub> P <sub>2</sub> Lu
FW	1461.35
Crystal system	Monoclinic
Space group	<i>P</i> 2 <sub>1</sub> / <i>n</i> (14)
<i>a</i> /Å	13.2633(6)
<i>b</i> /Å	23.0476(13)
<i>c</i> /Å	22.9725(6)
Angle/deg	91.390(14)
<i>U</i> /Å <sup>3</sup>	7020.3(5)
<i>Z</i>	4
<i>D</i> <sub>calcd</sub> /g·cm <sup>-3</sup>	1.383
<i>T</i> /K	293(2)
$\lambda$ /Å	0.71073
$\mu$ /mm <sup>-1</sup>	1.512
<i>R</i> <sub>1</sub> <sup>a</sup> [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )]	0.0698
<i>wR</i> <sub>2</sub> <sup>a</sup> [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )]	0.0778

$$^a R_1 = \sum \|F_o| - |F_c|\| / \sum |F_o|, wR_2 = \{\sum [w(F_o^2 - F_c^2)] / \sum [w(F_o^2)]\}^{1/2}$$

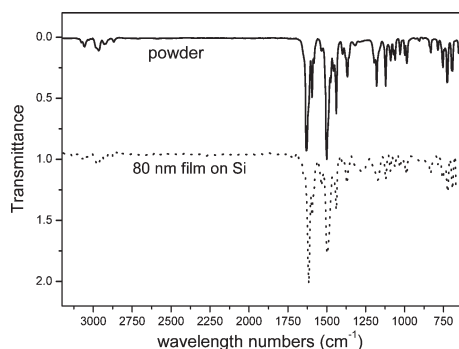


**Fig. 2** ORTEP diagram of  $\text{Lu}(\text{PMIP})_3(\text{TPPO})_2$  with the thermal ellipsoids drawn at the 30% probability level and the H atoms removed for clarity.

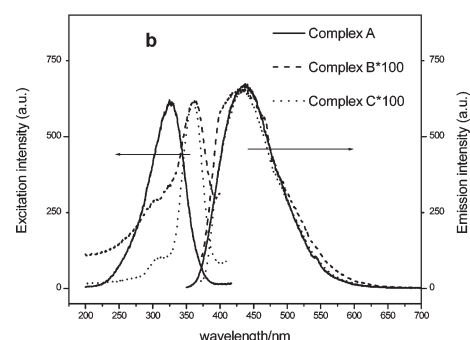
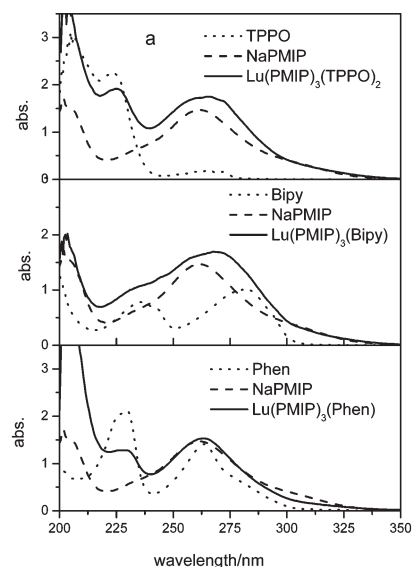
is quite different: that of complex **A** is about 100 times higher than that of complexes **B** and **C**. Lutetium complexes are ligand-emission materials, whose fluorescence originates from the anionic ligand (in the three complexes, it is  $\text{PMIP}^{-1}$ ). From the UV-vis spectra in Fig. 4(a), it is clear that the absorption of TPPO is located in the higher energy range of less than 240 nm while that of  $\text{PMIP}^{-1}$ , Bipy and Phen are in the same range from 300 to 215 nm, as has been mentioned above; this indicates that the singlet energy level of TPPO is higher than those of the other three. In other words, the energy absorbed by TPPO can be transferred to  $\text{PMIP}^{-1}$  and strengthen its emission, while the energy absorbed by Bipy and Phen cannot transfer to  $\text{PMIP}^{-1}$  efficiently, which leads to different PL intensities for the three complexes.

### HOMO and LUMO energy levels

The ultraviolet photoelectron spectrum (UPS) was measured from an evaporated ultrathin film (on ITO substrate) of each complex under ultrahigh vacuum conditions using synchrotron radiation as the energy source ( $h\nu$ , in this work it was 32.7 eV), from which the first ionization energy ( $I_p$ ) could be obtained according to the equation reported by Tang *et al.*:<sup>14</sup>  $I_p = h\nu - \phi$ , where  $\phi$  stands for the width of the UPS spectrum. The energy of the highest occupied molecular orbital (HOMO) of each complex is equal to its  $I_p$  value. The  $\phi$  values obtained were 26.15, 26.43 and 26.10 eV for complexes **A**, **B** and **C**, respectively, so the HOMO level values for complexes **A**, **B** and **C** are 6.55, 6.27 and 6.60 eV, respectively. The energy of the lowest unoccupied molecular orbital (LUMO) of the complex is estimated from its HOMO level and the energy gap between the HOMO and LUMO, which can be obtained by



**Fig. 3** FT-IR spectra of complex **A** and its 80 nm-evaporated film on a Si substrate.

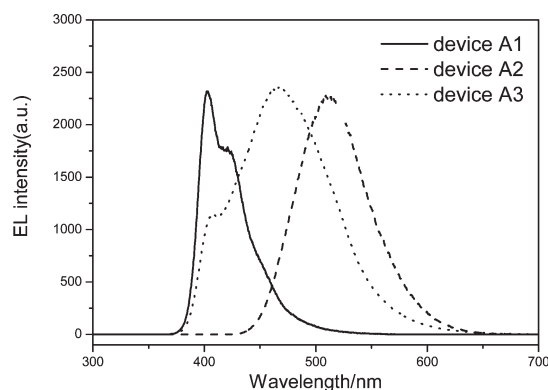


**Fig. 4** (a) UV-vis absorbance spectra of the complexes measured from their  $\text{CHCl}_3$  solutions. (b) Normalized photoluminescence spectra (excitation and emission) of the complexes **A** (—), **B** (---) and **C** (···).

referring to the absorption spectrum of each complex; these values are 3.35, 3.31 and 3.35 eV for complexes **A**, **B** and **C**, respectively. Thus, the LUMO values are 3.20, 2.96 and 3.25 eV for complexes **A**, **B** and **C**, respectively.

### EL of complex A

The electroluminescence spectra of the devices based on complex **A** are shown in Fig. 5. For a double layer device, A1, with



**Fig. 5** EL spectra of devices using complex **A** as the emitting material. Device A1: ITO/TPD(40 nm)/A(40 nm)/ $\text{Mg}_{0.9}\text{Ag}_{0.1}$ ; device A2: ITO/TPD(40 nm)/A(40 nm)/AlQ(40 nm)/ $\text{Mg}_{0.9}\text{Ag}_{0.1}$ ; device A3: ITO/TPD(20 nm)/A(40 nm)/BCP(20 nm)/AlQ(40 nm)/ $\text{Mg}_{0.9}\text{Ag}_{0.1}$ .

the configuration ITO/TPD(40 nm)/A(40 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub>, the blue EL emission peaking at 403 nm was from TPD; no emission from complex A was detected, which reveals that complex A has good electron-transport characteristics. When AIQ was used as the electron injection and transport material to form device A2 with the configuration ITO/TPD(40 nm)/A(40 nm)/AIQ(40 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub>, the emission (Fig. 5) peaking at 513 nm was from AIQ, indicating that this complex has good hole-transport properties as well. When a hole-blocking layer (BCP) was introduced between AIQ and complex A in device A3, ITO/TPD(20 nm)/A(40 nm)/BCP(20 nm)/AIQ(40 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub>, a blue light (Fig. 5) was observed and the maximum luminance of this device was 119 cd m<sup>-2</sup> at 19 V. The emission peaking at 465 nm was mainly from complex A, although it is a little red-shifted compared to its PL, with a shoulder from TPD. Fig. 6 depicts the energy level diagram complete with the Fermi level positions for the indium tin oxide (ITO) anode and Mg<sub>0.9</sub>Ag<sub>0.1</sub> cathode. Other energy levels shown in Fig. 6 are gathered from the literature.<sup>15–17</sup> The EL spectrum can be explained as follows: the electron injection barrier from complex A to TPD (0.7 eV) and the hole injection barrier from TPD to complex A (0.7 eV) are equal; at the same time, as complex A has very good electron-transport characteristics, for device A1 the recombination occurs in the TPD layer and blue light from TPD is observed. For device A2, although the hole injection barrier (0.7 eV) from TPD into the complex layer is much higher than electron injection from AIQ to the complex layer (−0.2 eV), the holes and electrons recombine in the AIQ layer, caused by the high hole-transport rate in TPD and the low electron-transport rate in AIQ.<sup>18</sup> When a BCP layer was inserted between complex A and AIQ, the holes are prevented from entering the ALQ layer, so device A3 emits light mainly from complex A.

### EL of complex B

The EL spectra of the devices using complex B as emitter are shown in Fig. 7. Device B1 is a double layer device with the same configuration (ITO/TPD(40 nm)/B(40 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub>) as device A1, however, its EL spectrum is quite different; in spite of the minor emission from TPD peaking at 403 nm, a new emission peaking at 512 nm appeared and no emission from complex B was detected. This new emission is from the exciplex formed between TPD and complex B. It is known that the formation of an exciplex is a result of the charge transfer between a donor and an acceptor at their interface in the excited state, which often occurs between planar molecules. Neutral ligand Bipy becomes a rigid plane after it is coordinated to Lu<sup>3+</sup> and allows complex B to easily form an exciplex, which is proven by the PL of their 1 : 1 mixture (Fig. 7, inset), in which a new emission having the same feature as the EL emission peaking at 512 nm is observed. On the other hand,

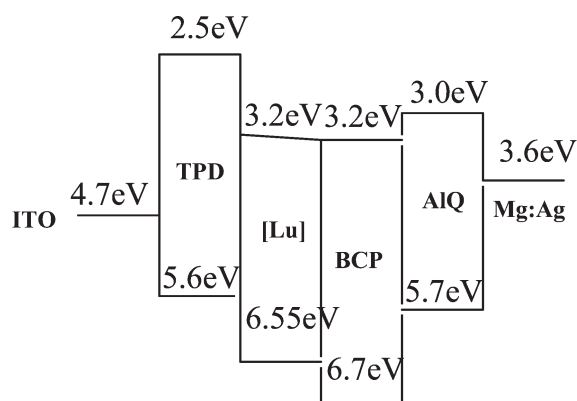


Fig. 6 The energy diagram of the materials used in device A3.

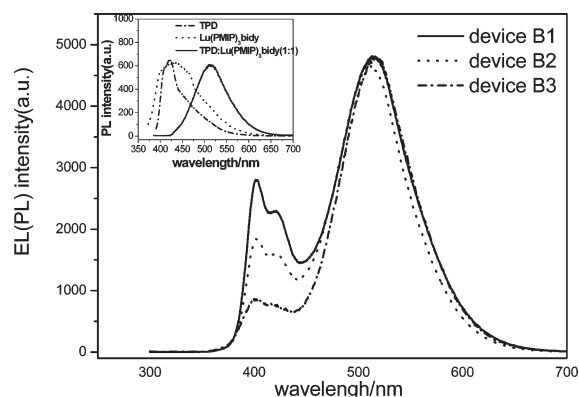


Fig. 7 The normalized EL spectra of devices using complex B as emitter. Device B1: ITO/TPD(40 nm)/B(40 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub>; device B2: ITO/TPD(20 nm)/B(60 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub>; device B3: ITO/TPD(10 nm)/B(50 nm)/BCP(20 nm)/AIQ(40 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub>. Insert: PL spectra of TPD, complex B and their 1:1 mixture by coating them on quartz slides from their corresponding CHCl<sub>3</sub> solutions, the excitation wavelengths are 380, 360 and 380 nm, respectively.

the UV-vis spectrum of this mixture is just the sum of its components, indicating that the new species is not formed in the ground state. Our previous work<sup>19</sup> has proven that the exciplex is formed between the excited state of TPD and the ground state of the complex in the interface. When a positive voltage was applied to the ITO electrode, a maximum luminance of 160 cd m<sup>-2</sup> at 19 V from device B1 was observed. When the thickness of TPD was decreased while the device kept a fixed total thickness, the relative emission intensity from TPD decreased and, at the same time, the light emitted changed from blue green to pure green as the thickness of TPD changed from 40 to 5 nm (Fig. 7, device B2).

In order to gain higher performance from the exciplex, device B3 with the configuration ITO/TPD(10 nm)/B(50 nm)/BCP(20 nm)/AIQ(40 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub> was fabricated. As complex B has good hole-transport features and the HOMO and LUMO energy levels of complex B (6.3 and 3.0 eV, respectively) are similar to those of complex A, it is necessary to introduce BCP as the hole-blocking layer in order to obtain emission from the exciplex. A green light (Fig. 7) with a maximum brightness as high as 1010 cd m<sup>-2</sup> at 16 V and a maximum luminous efficiency of 0.13 lm W<sup>-1</sup> at 9 V was achieved with a turn-on voltage of 3 V from this device. Such an improvement of the luminance and the low turn-on voltage is the contribution of the hole-transport properties of the complex and employment of the electron injection layer of AIQ. It is well-known that AIQ can carry a higher current density,<sup>11</sup> which balances carrier injection in the interface of TPD and the complex.

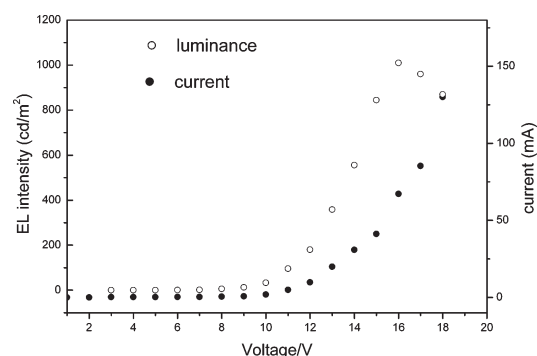


Fig. 8 The current density–voltage and luminance–voltage characteristics of device B2: (ITO)/TPD(10 nm)/B(50 nm)/BCP(20 nm)/AIQ(40 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub>.



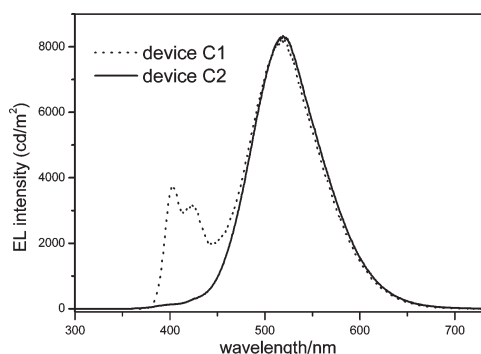
The current density–voltage and luminance–voltage characteristics of device B3 are shown in Fig. 8.

### EL of complex C

Complex **C** shows the same properties as complex **B**, that is, it easily forms an exciplex with TPD emission peaking at 512 nm. The EL spectra shown in Fig. 9 indicates this clearly. A double layer device C1 with the configuration ITO/TPD(15 nm)/C(95 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub> achieved a highest brightness of 475 cd m<sup>−2</sup> at 17 V, which is greatly enhanced compared to the double layer device B1, ITO/TPD(40 nm)/B(40 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub>, indicating that carrier injection is well-balanced in this device. In order to eliminate the emission from TPD, in device C2 with the configuration ITO/TPD(5 nm)/C(40 nm)/BCP(20 nm)/AlQ(40 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub> the thickness of TPD was reduced to 5 nm; as expected, no emission at 403 nm from TPD was detected (Fig. 9, solid line), however, the performance of device C2 decreased to 776 cd m<sup>−2</sup> at 17 V, caused by the thinned TPD layer, which reduced hole injection.

### Conclusions

Three ternary lutetium complexes, Lu(PMIP)<sub>3</sub>(TPPO)<sub>2</sub> (**A**), Lu(PMIP)<sub>3</sub>Bipy (**B**) and Lu(PMIP)<sub>3</sub>Phen (**C**), were synthesized and their photoluminescence and electroluminescence properties investigated. Complex **A** was characterized by single-crystal X-ray diffraction. Experimental results revealed that different structures not only affect the complexes' PL features, but also change their EL properties. The PL intensity of complex **A** is about 100 times higher than that of complexes **B** and **C**, but they have similar spectral character. Blue light originating from complex **A**, with a luminance of 119 cd m<sup>−2</sup>, was obtained by constructing a configured device. Although the PL intensity of complexes **B** and **C** was weaker than that of complex **A**, they displayed a better EL performance because



**Fig. 9** The normalized EL spectra of devices using complex **C** as emitter. Device C1: ITO/TPD(15 nm)/C(95 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub>; device C2: ITO/TPD(5 nm)/C(40 nm)/BCP(20 nm)/AlQ(40 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub>.

of formation of the exciplex. A device with the configuration ITO/TPD(10 nm)/B(50 nm)/BCP(20 nm)/AlQ(40 nm)/Mg<sub>0.9</sub>Ag<sub>0.1</sub> presented a maximum luminance of 1010 cd m<sup>−2</sup> at 16 V with the highest power efficiency, 0.13 lm W<sup>−1</sup> at 9 V, and a turn-on voltage as low as 3 V; this is the highest brightness obtained from an exciplex so far. Therefore, the three lutetium complexes all have potential applications as blue or green light-emitting materials. Our results indicate not only that complexes with a high PL intensity can be used as emitters in OLEDs, but also that those showing weak or no PL have potential applications if they can form exciplexes with a high EL efficiency.

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