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Luminescent mononuclear copper(I) heteroleptic complexes with 6-cyano-2,2'-bipyridine

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ABSTRACT

A new series of luminescent mononuclear Cu(I) heteroleptic complexes of 6-cyano-2,2'-bipyridine (cbpy), [Cu(cbpy)(PPh₃)X] (X = I (1); Br (2)) and [Cu(cbpy)(PPh₃)₂](ClO₄) (3), has been synthesized and characterized. It is revealed that phosphine in place of halide results in lengthening of the Cu – P bond from 2.21 Å of 1 and 2.19 Å of 2 to 2.27 Å of 3. Complexes 1–3 display the weak low-energy absorption bands in the 350– 600 nm region in dichloromethane, which are assigned to the Cu(I) to cbpy metal-to-ligand charge-transfer (MLCT) transitions, probably mixed with some halide-to-ligand charge-transfer (XLCT) character for 1 and 2. Complexes 1–3 show the solid-state emissions at ambient temperature, varying with the second auxiliary ligand coordinated to the {Cu(cbpy)(PPh₃)} motif, which most likely originate from the MLCT excited states, perhaps involving some XLCT character for 1 and 2.

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Copper(I) complexes are of considerable interest with respect to their intriguing photophysical properties and possible utilization in solar energy conversion, luminescence-based sensors, electroluminescence devices, and probes of biological systems [1–5]. The appropriate selection of N-heterocyclic chelating ligands is one key point to tune and improve the luminescence properties of cuprous complexes, which can be highly affected via the steric, electronic, and conformational effects of the coordinated chelates [6–8]. Additionally, the auxiliary ligands such as halide and phosphine also have great effects on the emissive properties of Cu(I) species [9–16].

Recently, we have initiated the investigation of photoactive copper(I) complexes bearing multi-site N-heterocyclic chelate bridging ligands [15–17]. It is shown that both N-heterocyclic chelate and the auxiliary ligand such as halide and phosphine have a significant impact on the construction and luminescence properties of the Cu (I) systems. Moreover, to the best of our knowledge, metal complexes of 6-cyano-2,2'-bipyridine (cbpy) have not been reported hitherto. Herein, we report the syntheses, crystal structures, and photophysical properties of a new series of mononuclear Cu(I) heteroleptic complexes with cbpy, namely, [Cu(cbpy)(PPh_3)X] (X=I (1); Br (2)) and [Cu(cbpy)(PPh_3)_2](ClO_4) (3). It is demonstrated that complexes 1-3 are all emissive in the solid state at ambient temperature, and the second ancillary ligands introduced to the {Cu(cbpy)(PPh_3)} motif exert significant influences on the molecular structures and photophysical properties of the corresponding copper(I) complexes.

Mononuclear Cu(I) halide complexes of cbpy formulated as [Cu (cbpy)(PPh₃)X] (X=I (1); Br (2)) (Scheme 1) were facilely synthesized via treatment of cuprous halide with two equivalents of PPh₃ in dichloromethane, followed by addition of one equivalent of cbpy [18]. In the above reactions, the non-isolated intermediates are actually a binuclear copper(I) compound [(PPh₃)₂Cu(μ -X)₂Cu(PPh₃)] (X=I; Br) as confirmed by X-ray structural analysis [16], which are considerably air-stable and highly soluble in dichloromethane. In addition, monomeric Cu(I) complex possessing PPh₃ and cbpy mixed ligands, [Cu(cbpy)(PPh₃)₂](ClO₄) (**3**) [18], which has no halide ligand, was afforded by treatment of Cu(ClO₄)₂·6H₂O with four equivalents of PPh₃, where PPh₃ served as an unidentate ligand and as a reducing reagent of Cu(II), followed by addition of one equivalent of cbpy.

X-ray crystallographic studies [19] of the halide complexes 1 and 2 reveal that each four-coordinated Cu(I) atom is bound to two nitrogen atoms of cbpy, one PPh₃, and one halide to generate a distorted tetrahedral configuration because of the small bite angle of cbpy (Fig. 1a and b). The dihedral angles between the N-Cu-N plane and the P-Cu-X plane are around 89.6° and 86.9° for 1 and 2, respectively. The Cu-N (av. 2.1015 Å) and Cu-P (2.2079 Å) distances in 1 are slightly longer than the corresponding bond lengths (2.0945 and 2.1913 Å) of **2**, which are related to the ligand field strengths of the halide ligands $(I^{-} < Br^{-})$ [20]. The Cu-N, Cu-P, and Cu-X distances in 1 and 2 are comparable to those observed in other tetrahedral Cu(I) complexes with halide and PPh₃ mixed ligands [21-26]. The N-Cu-N and P-Cu-X bond angles of 1 (78.39 $(14)^{\circ}$ and $119.79(4)^{\circ}$) are largely identical with the corresponding values of **2** $(78.26(11)^{\circ} \text{ and } 118.87(4)^{\circ})$, while the N-Cu-P and N-Cu-X angles of 1 and 2 have a small variation of 4-8°. It is notable that the cbpy ligands of two adjacent enantiomeric molecules in 1

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Scheme 1. Synthetic route of the Cu(I) cbpy complexes 1-3.

and **2** are essentially parallel and display a favorable pairwise π - π stacking (Fig. 1c and d). The interplanar separations are approximately 3.48 and 3.62 Å for **1** and **2**, respectively, indicating the existence of a weak π - π interaction between two neighboring cbpy ligands [27].

To explore the effect of PPh₃ in place of halide on the metric parameters of molecular structures, X-ray structural determination of PPh₃ substituted **3** was also carried out [19]. As depicted in Fig. 2a, the Cu(I) atom is in a distorted tetrahedral geometry formed by two nitrogen atoms of cbpy and two P donor atoms from PPh₃ ligands. The dihedral angle between the N-Cu-N plane and the P-Cu-P plane is about 81.0° in **3**, which is obviously smaller than those of the halide complexes **1** and **2**, probably owing to replacement of halide via highly encumbered PPh₃. The Cu-N distances (av.

2.1065 Å) of **3** are slightly longer than those observed in **1** and **2**, whereas the Cu–P distances (av. 2.2711 Å) of **3** are much longer than those of **1** (2.2079(12) Å) and **2** (2.1913(12) Å) due to introduction of the second π -acceptor ligand PPh₃. The N–Cu–N bond angle (79.10(19)°) in **3** is comparable to those of **1** and **2**, whereas the P–Cu–P angle (122.26(5)°) of **3** is slightly larger than the P–Cu–X angles of **1** and **2**, perhaps owing to the large steric hindrance between intramolecular two PPh₃ ligands. Different to the stacking manners of two neighboring cbpy ligands in **1** and **2**, the π - π stacking of **3** only occurs between the pyridyl rings of two adjacent cbpy ligands (Fig. 2b). The interplanar distance of **3** is approximately 3.48 Å, suggesting that a weak π - π interaction is likely operating between intermolecular two cbpy ligands.

The UV-vis absorption spectra of cbpy and its complexes 1-3 in dichloromethane at ambient temperature are shown in Fig. 3. The free ligand cbpy shows two absorption bands at $\lambda_{max}\!\approx\!242$ and 282 nm, attributable to the ${}^{1}\pi\pi^{*}$ transitions inside cbpy. The Cu(I) halide complexes 1 and 2 exhibit several strong absorptions $(\epsilon > 10^4 \,\mathrm{M^{-1} \, cm^{-1}})$ in the range of 230–350 nm, most likely coming from cbpy and PPh₃. Further support is given by a close matching of the absorption spectra in high-energy region (\leq 350 nm) of cbpy and its halide complexes 1 and 2. In addition to the high-energy absorptions, complexes 1 and 2 have a comparatively weak low-energy absorption band (ε <750 M⁻¹ cm⁻¹) with a maximum at around 422 and 428 nm, respectively, which is assigned to a metal-to-ligand charge-transfer (MLCT) transition from the d_{π} orbital of the (3d¹⁰) Cu center to the unoccupied π^* orbital of the cbpy ligand, probably mixed with some halide-to-ligand charge-transfer (XLCT) character [1,3,12–15,26]. Accordingly, for complex 3, besides the high-energy absorption bands (\leq 350 nm) from the LC $^{1}\pi$ - π^{*} transitions, a weak low-energy absorption ($\varepsilon \approx$ 2440 M⁻¹ cm⁻¹) centered at 391 nm is displayed and identified as the Cu(I) to cbpy MLCT transition [3,6-11,15,17], which is notably blue-shifted relative to those of 1 and 2.



Fig. 1. ORTEP drawings (a and b) and pairwise stacking views (c and d) of **1** (top) and **2** (bottom). Hydrogen atoms are omitted for clarity. Selected bond distances (Å) and angles (°): for **1**, Cu1 – N1 2.090(3), Cu1 – N2 2.113(3), Cu1 – P1 2.2079(12), Cu1 – I1 2.5828(6), N1 – Cu1 – N2 78.39(14), N1 – Cu1 – P1 117.06(10), N1 – Cu1 – I1 109.12(9), N2 – Cu1 – P1 116.87(10), N2 – Cu1 – I1 108.21(10), P1 – Cu1 – I1 119.79(4); for **2**, Cu1 – N1 2.082(3), Cu1 – N2 2.107(3), Cu1 – P1 2.1913(12), Cu1 – Br1 2.4228(9), N1 – Cu1 – N2 78.26(11), N1 – Cu1 – P1 123.12(8), N1 – Cu1 – Br1 105.69(8), N2 – Cu1 – P1 122.25(8), N2 – Cu1 – Br1 100.31(8), P1 – Cu1 – Br1 118.87(4).



Fig. 2. ORTEP drawing and pairwise stacking view of the cation of **3**. Hydrogen atoms are omitted for clarity. Selected bond distances (Å) and angles (°): Cu1–N1 2.111(4), Cu1–N2 2.102(4), Cu1–P1 2.2683(13), Cu1–P2 2.2739(13), N1–Cu1–N2 79.10 (19), N1–Cu1–P1 114.33(12), N1–Cu1–P2 104.30(12), N2–Cu1–P1 108.23(12), N2–Cu1–P2 120.29(11), P1–Cu1–P2 122.26(5).

The blue-shift can be rationalized in views of the lowering of the HOMO energy upon replacement of the halide of **1** and **2** with a good π -acceptor ligand PPh₃, thus effectively increasing the HOMO-LUMO energy gap.

The solid-state emissions of cbpy and its complexes **1–3** are investigated at ambient temperature (Fig. 4). The free ligand cbpy emits a purple color with λ_{max} = 366 nm in the solid state, attributed to the fluorescent emission from ligand-centered $\pi \rightarrow \pi^*$ transition. The halide



Fig. 3. UV-vis absorption spectra of cbpy (\Box) , **1** (\bullet), **2** (\blacktriangle), and **3** (\checkmark) in diluted dichloromethane solution at ambient temperature.



Fig. 4. Emission spectra of 1 (\blacktriangle), 2 (\bullet), and 3 (\blacksquare) in the solid state at ambient temperature.

complexes 1 and 2 show a broad solid-state emission peaking at 594 and 613 nm at ambient temperature, respectively. With reference to previous work [1,3,12–14,21,26], the highest occupied molecular orbitals (HOMO) of the halide complexes **1** and **2** are believed to most likely spread over the Cu(I) ion and the halide, probably including some contributions from the phosphine, while their lowest unoccupied molecular orbitals (LUMO) are thought to be basically localized on the cbpy ligand. Hence, the emissive excited states of the halide complexes 1 and 2 are perhaps best regarded as the metal-to-ligand charge-transfer (MLCT) excited states with some mixing of the halide-to-ligand chargetransfer (XLCT) character. The solid-state emission (λ_{max} 594 nm) of 2 exhibits a small blue-shifting of approximately 19 nm compared with 1 (λ_{max} 613 nm). Such a blue-shifting can be rationalized via the replacement of the iodide by a weaker electron-donating bromide, lowering the HOMO energy level, less influencing the LUMO energy, and thus resulting in an increase of the HOMO-LUMO energy gap. Complex 3, which has no halide ligand, exhibits a broad solid-state emission maximum at 563 nm, displaying a blue-shift of ca. 50 nm with respect to that of 1, which may be due to the substitution of the iodide via a good π -acceptor ligand PPh₃, decreasing the HOMO level, and hence leading to a lager HOMO-LUMO gap. In addition, the mode of the π - π stacking between intermolecular cbpy ligands (Figs. 1c and 2b) perhaps plays a positive role in increasing the blue-shift of the emission of **3** relative to **1**. For **3**, the LUMO is still believed to be mainly localized on cbpy, such that the introduction of the second PPh₃ would be anticipated to have little effect on the LUMO energy. On the other hand, its HOMO is largely centered on the Cu(I) center, probably involving some contributions from PPh₃. Thus the emissive excited state of 3 is reasonably assigned to an MLCT excited state [3,6-11,17].

In summary, a new series of three luminescent mononuclear copper (I) heteroleptic complexes with 6-cyano-2,2'-bipyridine (cbpy) has been synthesized and characterized. It is demonstrated that the second auxiliary ligand introduced, such as halide and phosphine, has a considerable effect on both the molecular structures and the photophysical properties of Cu(I) cbpy complexes. Further investigation on mononuclear copper(I) heteroleptic complexes with 6-monosubstituted 2,2'-bipyridine is underway.

Acknowledgments

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Appendix A. Supplementary material

CCDC 831454, 831453, and 831452 contain the supplementary crystallographic data for **1–3**, respectively. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Supplementary data to this article can be found online at doi:10. 1016/j.inoche.2011.09.040.

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- [19] Crystal data for **1** (CCDC 831454): $C_{29}H_{22}$ CulN₃P, *M*=633.91, triclinic, *P* 1, *a*=9.5392(9) Å, *b*=9.7743(9) Å, *c*=15.5917(15) Å, *α*=89.2440(10)°, *β*=83.7480(10)°, *γ*=71.5920(10)°, *V*=1370.8(2) Å³, *Z*=2, *T*=296(2) K, *μ*(Mo Kα) = 2.003 mm⁻¹, *D_c*=1.536 g cm⁻¹, *λ*=0.71073 Å, 2θ=50°. The structure, refined on *F*², converged for 4456 unique reflections (*R*_{int}=0.0239) and 3342 observed reflections with *I*>2*σ*(*I*) to give *R*₁=0.0336 and *wR*₂=0.0769 and a good-of-fit=0.990. Crystal data for **2** (CCDC 831453): *C*₂₉H₂₂BrCuN₃P, *M*=586.92, triclinic, *P* 1, *a*=8.670(4) Å, *b*=9.511(4) Å, *c*=17.055(7) Å, *α*=89.631(4)°, *β*=87.973(5)°, *γ*=67.419(5)°, *V*=1297.8(9) Å³, *Z*=2, *T*=296 (2) K, *μ*(Mo Kα) = 2.465 mm⁻¹, *D_c*=1.502 g cm⁻¹, *λ*=0.71073 Å, 2θ=49.98°. The structure, refined on *F*², converged for 4180 unique reflections (*R*_{int}=0.0211) and 3271 observed reflections with *I*>2*σ*(*I*) to give *R*₁=0.0355 and *wR*₂=0.0924 and a good-of-fit=1.035. Crystal data for **3** (CCDC 831452): C-48H₃₉Cl₃CuN₃O₄P₂. *M*=953.65, monoclinic, *P*2₁/c, *a*=15.8564(12) Å, *b*=14.7892(11) Å, *c*=20.8431(15) Å, *β*=111.1230(10)°, *V*=4559.4(6) Å³, *Z*=4, *T*=296(2) K, *μ*(Mo Kα)=0.772 mm⁻¹, *D_c*=1.389 g cm⁻¹, *λ*=0.71073 Å, 2θ=52.2°. The structure, refined on *F*², converged for 8933 unique reflections (*R*_{int}=0.0475) and 5586 observed reflections with *I*>2*σ*(*I*) to give *R*₁=0.0675 and *wR*₂=0.1948 and a good-of-fit=1.031.
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