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Synthesis, crystallography, phosphorescence of platinum complexes coordinated with 2-phenylpyridine and a series of β-diketones

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ABSTRACT

Metallocyclic platinum(II) complexes coordinating with 2-phenylpyridine (ppy) and a series of β-diketone ancillary *O^O* ligands, (ppy)Pt(acac), (ppy)Pt(ba), (ppy)Pt(dbm), and (ppy)Pt(tta) (acac = acetylacetone, ba = benzoylacetone, dbm = dibenzoylmethane, tta = thenoyltrifluoroacetone) were synthesized. The crystal structure, absorption, emission, quantum yield and phosphorescence life time were characterized. As the conjugative π system of the *O^O* ligand increases in the order acac < ba < dbm, or there is a group $-CF_3$to attract the electron density of the tta ligand, the quantum yield decreases in the order (ppy)Pt(acac) > (ppy)Pt(ba) > (ppy)Pt(dbm) > (ppy)Pt(tta) due to an energy back-flow from ppy to the *O^O* ligand, a trend also in contrast to the phosphorescence emission spectra and time decay (biexponentially, ~0.7–13 μs).

1. Introduction

Luminescent square planar platinum complexes have been extensively used in DNA probing, photosensitizers and optoelectronic devices [1–4]. Recently, researches for organic light-emitting electrophosphorescent display (OLED) have attracted much attention because of their high internal efficiency using all of the electrogenerated singlet and triplet excitons [5–9]. In order to understand the electron transfer processes in the excited state and apply this knowledge to practical applications, numerous emitting phosphors have been synthesized. Structurally, these emitting materials usually incorporate a heavy metal iron (Pt, Ir, Os, Ru, Rh, Re) to induce spin flips so that both singlet and triplet states can be used electronically. Porphines [5,9–13], phenylpyridines [13–16], thienylpyridines [17], and N^N ligands (bipyridines, isoquinolines [18–23]), are previously used as the ligands to synthesize phosphorescent dyes. Besides the C^N and N^N ligands, Pt(II) can also coordinate with an ancillary bidentate O^O ligand, e.g. β-diketones [13,15,24], which also take part in the photophysical processes and greatly affect the physical properties. These metallocyclic complexes are known to be strongly emissive and have long luminescent lifetimes. Reports on the structured emission pattern and long luminescent lifetime indicate that the emission arises from an excited state that is predominantly a ^{13}LC (ligand-centered) state, mixed with MLCT (metal to ligand charge transfer) character by strong spin–orbit-coupling [25,26]. However, the role of the ancillary β-diketones in the photophysics of the platinum complexes is still not much clear.

In this paper, we synthesized Pt(II) complexes using 2-phenylpyridine and four different β-diketones: benzoylacetone (ba), dibenzoylmethane (dbm), acetylacetone (acac) and thenoyltrifluoroacetone (tta), in order to examine their photophysical properties.

2. Experimental

2.1. Materials and reagents

2-Phenylpyridine (ppy), dibenzoylmethane (dbm) and thenoyltrifluoroacetone (tta) were purchased from Acros Chemicals Inc. The platinum μ-dichloro-bridge dimer (ppy)Pt(μ-Cl)$_2$Pt(ppy) was prepared according to the published procedures [24,25]. The other reagents are of analytical grade and were used as received. All solvents were purified and distilled by standard procedures before use.

2.2. Synthesis of (ppy)Pt(O^O) complexes: general procedure

Synthetic strategy for the complexes (ppy)Pt(O^O) and their chemical structures were shown in Scheme 1 [27,28]. One equivalents of dimer (ppy)Pt(μ-Cl)$_2$Pt(ppy), 1 equiv. of the appropriate

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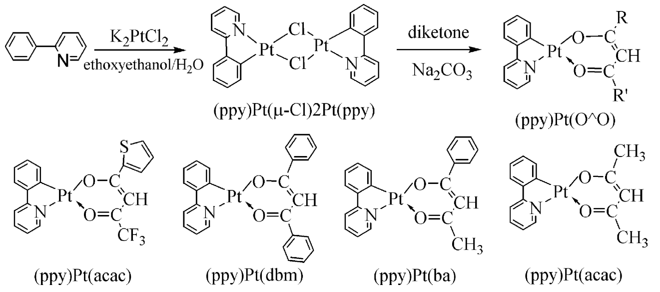

Scheme 1. The synthetic route of the platinum complexes.

diketone and 10 equiv. of Na_2CO_3 in 50 ml of 2-ethoxyethanol were reacted at 100 °C for 16 h. The solvent was removed under reduced pressure, and the compound was purified by chromatography using dichloromethane as the eluent. The product was recrystallized in dichloromethane/methanol solution. All the complexes are air-stable and yellow-orange crystals with moderate yield (30–50%).

2.2.1. (ppy)Pt(tta)

^{1}H NMR ($CDCl_3$, 400 MHz) δ, ppm: 8.84 (dd, 1H, J = 5.6, 5.7 Hz), 7.80 (m, 3H), 7.61 (d, 2H, J = 6.4 Hz), 7.43 (q, 1H, J = 3.8 Hz), 7.12 (m, 4H), 6.40 (d, 1H, J = 4.4 Hz). *Anal.* Calc. for $C_{19}H_{12}NF_3O_2SPt$: C, 39.93; H, 2.11; N, 2.46. Found: C, 39.23; H, 1.50; N, 2.45%.

2.2.2. (ppy)Pt(dbm)

^{1}H NMR ($CDCl_3$, 400 MHz) δ, ppm: 9.15 (s, 1H), 8.07 (t, 4H, J = 9.6 Hz) 7.80 (m, 2H), 7.67 (d, 1H, J = 7.6 Hz), 7.53 (m, 8H), 7.16 (m, 2H), 6.77 (s, 1H). *Anal.* Calc. for for $C_{26}H_{19}NO_2Pt$: C, 54.45; H, 3.32; N, 2.45. Found: C, 54.43; H, 3.43; N, 2.79%.

2.2.3. (ppy)Pt(ba)

^{1}H NMR ($CDCl_3$, 400 MHz) δ, ppm: 9.06 (d, 1H, J = 27.3 Hz), 7.99 (s, 2H) 7.65 (m, 3H), 7.44 (s, 3H), 7.25 (s, 2H) 7.10 (s, 2H), 6.11 (s, 1H), 2.11 (s, 3H). *Anal.* Calc. for $C_{21}H_{17}NO_2Pt$: C, 49.41; H, 3.33; N, 2.75. Found: C, 49.20; H, 3.00; N, 2.43%.

2.2.4. (ppy)Pt(acac)

^{1}H NMR ($CDCl_3$, 400 MHz) δ, ppm: 8.99 (d, 1H, J = 5.6 Hz), 7.78 (t, 1H, J = 7.6 Hz), 7.61 (t, 2H, J = 6.8 Hz), 7.43 (d, 1H, J = 7.5 Hz) 7.16 (m, 3H), 5.47 (s, 1H), 2.05 (s, 3H), 2.00 (s, 3H). *Anal.* Calc. for $C_{16}H_{15}NO_2Pt$: C, 42.86; H, 3.35; N, 3.12. Found: C, 42.52; H, 3.37; N, 3.15%.

2.3. X-ray data collection and structure determination

Diffraction data were collected at room temperature with graphite monochromatized Mo Kα radiation (λ = 0.71073 Å) on a Brucker CCD X-ray diffractometer. The data were corrected for absorption using the SADABS program. The structures were solved by program SHELXS 97 direct methods and refined by full-matrix least squares on F^2 using the SHELXL 97 program package. The non-hydrogen atoms were refined anisotropically. Some details of (ppy)Pt(dbm) single crystal data collection and refinement is summarized in Table 1.

2.4. Measurements and instrumentation

^{1}H NMR was collected by a Bruker 400 MHz spectrometer. Absorption spectra were obtained by a Perkin Elmer Lambda-35 UV–Vis spectrometer. Photoluminescence spectra (PL) were carried out with a Hitachi F-4500 fluorescence spectrophotometer. Emission quantum yields were carried out at room temperature in a distilled 2-methyltetrahydrofuran (2-MeTHF) solution. Before measuring, the solution was degassed by several freeze-pump-thaw cycles performed in a Mbraun glove box at <0.1 ppm O_2 and H_2O level. Quinine in 1.0 N H_2SO_4 (Φ = 0.48 at 313 nm and Φ = 0.54 at 366 nm) [29] was used as a reference. To obtain the quantum yield data as precise as possible, the absorption and PL must be measured in a single machine run to compare the integrated emission area. The difference between refractive index of the solvent and that of the standard should also be accounted [24,29]. The quantum yield data obtained in this paper was strikingly consistent with data reported for (ppy)Pt(acac) in degassed 2-MeTHF (Φ = 0.15) [24]. Emission life times were obtained on a FLS 920 combined fluorescence lifetime and steady state spectrometer in CH_2Cl_2 solution at room temperature.

Table 1
Crystallographic data for (ppy)Pt(dbm)

Formula	$C_{26}H_{19}NO_2Pt$
Formula weight	572.51
Radiation (Å)	Mo Kα 0.71073
Crystal system	monoclinic
Space group	$P2(1)/c$
a (Å)	13.949(4)
b (Å)	7.779(2)
c (Å)	19.541(5)
α (°)	90.00
β (°)	109.869
γ (°)	90.00
V (Å^3)	1994.2 (9)
Z	4
D_{calc} (g/cm^3)	1.907
Absorption coefficient (mm^{-1})	7.060
F(000)	1104
Goodness-of-fit	1.037
R_1, R_w	0.0309, 0.0645
Temperature (K)	294 (2)

3. Results and discussion

3.1. Crystal structure

Single crystals of (ppy)pt(dbm) were grown from dichloromethane/ethanol solution and characterized by X-ray crystallography. The N(1)–Pt(1)–O(2) and C(7)–Pt(1)–O(1) angles are 175.74(17)° and 174.29(19)°, respectively (Fig. 1) indicating a very slight distortion of the square plane. The Pt1–O1 (2.082(4) Å) and Pt1–O2 (2.006(4) Å) bond lengths and the C7–Pt1–N1 (80.9(2)°) and O2–Pt1–O1 (90.55(15)°) bond angles are typical for metallocyclic and β-diketonate derivatives of Pt [15,24,30]. There is only one molecule in a single crystal cell. The molecules pack head-to-tail from unit to unit with a plane-to-plane separation of 3.422 Å indicative of moderate π–π interaction. Metal–metal interaction is very weak because the closest Pt–Pt distance is too long, 4.960 Å (Fig. 2).

3.2. Absorption and emission spectroscopy

Absorption spectra of the four complexes were recorded in CH_2Cl_2 solution at room temperature (Fig. 2). Table 2 summarizes the data. Strong spin–orbit-coupling is needed to efficiently mix the singlet and triplet excited states in order for the complexes to be useful as phosphors in OLEDs. Clear evidence for significant mixing of the singlet and triplet excited states is seen in both the absorption and emission spectra. The four complexes show intense absorption from ligand π–π^* and MLCT transitions. The extinction coefficients for these bands are in the ranges expected for π–π^* ligand-centered and MLCT transitions, 10358–50526 cm^{-1} M^{-1} and >6210 cm^{-1} M^{-1}, respectively. The higher energy, more intense

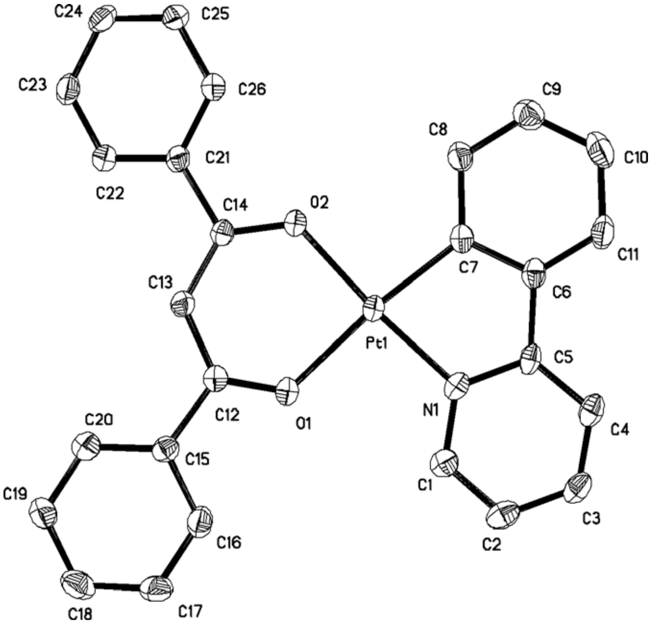

Fig. 1. Molecular structure of (ppy) Pt(dbm) (50% probability ellipsoids, H atoms omitted).

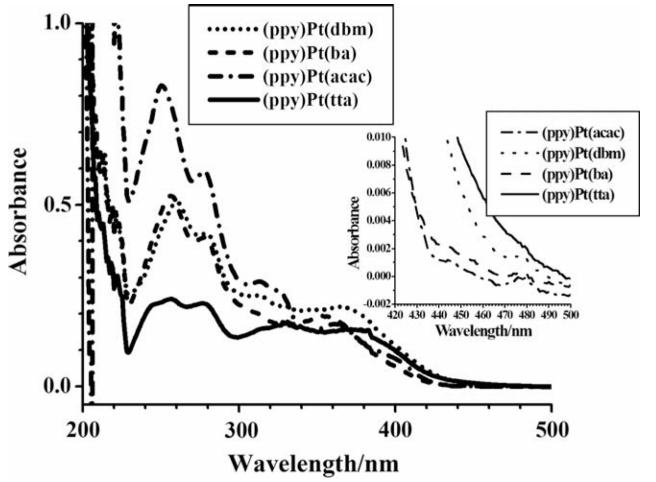

Fig. 2. Absorption spectra of the four complexes in CH_2Cl_2 at room temperature.

absorption bands are assigned to π–π^* LC transitions. In this region, the extinction coefficient decreases regularly as the aromaticity of the β-diketone ring decreases in the order of (ppy)Pt(dbm) > (ppy)Pt(ba) > (ppy)Pt(acac) (except for the 256 nm band for (ppy)Pt (ba)). ppyPt(tta) has the lowest absorption value in the 250–280 nm region, even though the aromaticity of the thiophene ring in tta ligand is thought to be higher than that of methyl group in acac of (ppy)Pt(acac). This may due to the electron-withdrawing character caused by "inductive effect" of the $-CF_3$ group. However, in the lower energy region in the range of 347–480 nm, the absorption for (ppy)Pt(tta) increases markedly. The heavy atom of S and F may contribute to the increase of the MLCT absorption. In addition, the spectrum feature of ppyPt(tta) is different from those of the other three. In this region, the absorption bands are not observed in the free HC^N and O^O ligand (see Fig. 3). The weaker absorption bands are assigned as metal-to-ligand charge transfer (MLCT) transitions. Note that for (ppy)Pt(acac), since 347 nm, the extinction coefficient increases till the maxima at 364 nm (6880 $cm^{-1}M^{-1}$). Although the extinction coefficients are sometimes greater than 6000 cm^{-1} M^{-1} for (ppy)Pt(ba) and (ppy)Pt(dbm), the absorption in the range 347–480 nm cannot be attributed to π–π^* LC transitions. It is caused by the increased aromaticity of the ancillary β-diketone ligand.

Table 2
Absorbances and extinction coefficients of the Pt complexes

Complex	Abs, λ_{max}/nm, (ε/cm^{-1} M^{-1})
ppyPt(acac)	250 (33157), 277 (23760), 313 (11560), 327 (10358), 347 (6210), 364 (6880), 410 (1864), 430 (184), 479 (10.9)
ppyPt(ba)	256 (52526), 278 (41032), 312 (20086), 330 (17933), 347 (18633), 353 (19355), 410 (3206), 430 (494), 476 (24.4)
ppyPt(dbm)	260 (50827), 279 (42129), 312 (25057), 328 (22459), 347 (21148), 366 (21958), 410 (7735), 430 (2456), 475 (144)
ppyPt(tta)	256 (24093), 276 (22861), 328 (17786), 347 (15473), 369 (15766), 410 (6474), 430 (2227), 477 (242)

Absorbances at 347 nm, 410 nm, 420 nm are not maxima, but the others are.

As the aromaticity of the free ligand decreases in the order of dbm > ba > acac, the first absorption maxima show bathochromic shifts, indicating a remarkable decrease in the S_1 energy state of the ligands (Fig. 3).

All of the complexes are emissive both in solution and solid state at room temperature. The emission spectra in 2-meTHF are shown in Fig. 4. The highly structured emission spectra for (ppy)-Pt(acac) and (ppy)Pt(ba) display vibronic progressions of ca. 1400 cm^{-1}, which are typical for the breathing modes of the aromatic ppy ring. The highly structured emission may originate from

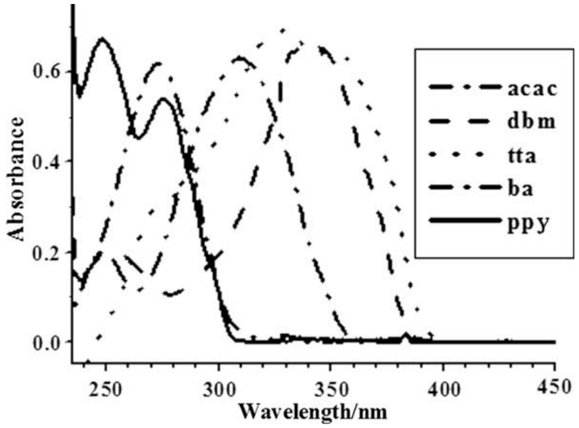

Fig. 3. Absorption spectra of ppy ligand and the four *O^O* ligands in CH_2Cl_2 at room temperature.

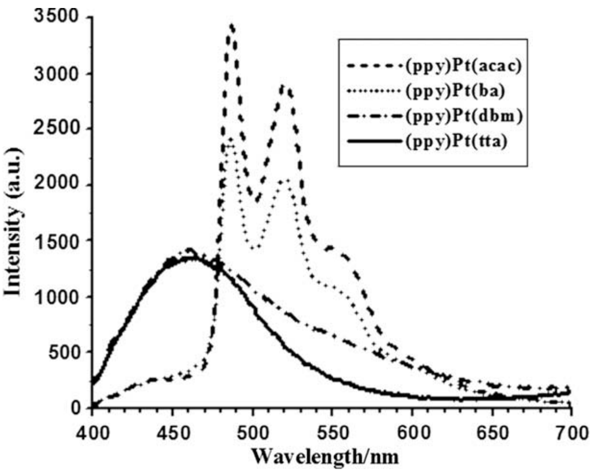

Fig. 4. Emission spectra of the four platinum complexes in 2-meTHF solution, excitation wavelength 313 nm.

a mixed ^{13}LC-MLCT excited state while the flattened emission spectra for (ppy)Pt(dbm) and (ppy)Pt(tta) may indicate a pure MLCT excited state contribution. Evidently, the β-diketonato ligand significantly perturb the excited state since the emission spectra for (ppy)Pt(dbm) and (ppy)Pt(tta) significantly differ from those of (ppy)Pt(acac) and (ppy)Pt(ba). This is in contrary to the conclusion of Ref. [24], in which fewer examples of β-diketonato ligands were observed.

The solid-state emission spectra of all of the four complexes become in some extent "fat" and show bathochromic shift and are poorly resolved (Fig. 5). These bathochromic shifts may be caused by the intermolecular interaction of the solid sample compared to molecules in solution, a phenomenon commonly observed for most organic dye compounds. These red-shifted flattened emissions may be called as excimer emission. As indicated in the first section of this paper, The molecules pack with a plane-to-plane separation of 3.422 Å, this distance is short enough to bring about excimer emissions [31].

3.3. The Quantum yield

The triplet level of acac lies well above the energies of the C^N ligand and the MLCT exited state [6]. Thus, the luminescence is dominated by ^{13}LC and MLCT transitions of PPY, leading to high efficiency. However, if the triplet state energy of the ancillary β-diketone ligand lies lower in energy than the ^{13}LC and MLCT of PPY, a triplet *O^O* level will be the lowest energy excited state. Thus, a switch from *C^N*Pt to *O^O*-based emission can be seen in the other complexes coordinated with different β-diketones (Table 3). The phosphorescence efficiency falls in the order of (ppy)Pt(acac) > (ppy)Pt(ba) > (ppy)Pt(dbm) > (ppy)Pt(tta). This efficiency increase order corresponds to the aromaticity decrease order dbm > ba > acac very well. Anyway it is confusing that (ppy)Pt(tta) possesses the lowest quantum yield for the F and S atom in tta ligand should contribute a "heavy atom" effect to increase the phosphorescence efficiency as it does to strengthen the MLCT absorption of the complex. The following two luminescent quenching mechanisms may come into play: (1) for (ppy)Pt(tta), the CF_3–group has a strong electron-withdrawing effect therefore greatly influence the electron density of the tta ligand as a whole and further decreases the triplet energy of tta ligand and therefore greatly induces energy back-flow; (2) as tta ligand's MLCT contribution increases, a switch of phosphorescence emission from previously mainly from *ppy*Pt centered to ttaPt centered makes the emission not as efficiently as the ppyPt centered. In contrast, (ppy)Pt(acac) gives the highest quantum efficiency of >0.14. The triplet level of the three β-diketones falls in the order acac > ba > dbm. Apparently, in the case of acac, the highest triplet energy level effectively hampers the energy back-flow from ppy to acac. In contrast, in complexes comprising *O^O* ligands ba, dbm and tta, energy back-flow or loss is inevitable, leading to weak phosphorescence.

Table 3
Quantum yields (%) of the platinum complexes

Complexes	Quantum yield at 313 nm	Quantum yield at 366 nm
(ppy)Pt(acac)	14.56	17.02
(ppy)Pt(ba)	3.85	3.93
(ppy)Pt(dbm)	1.33	1.47
(ppy)Pt(tta)	0.69	0.80

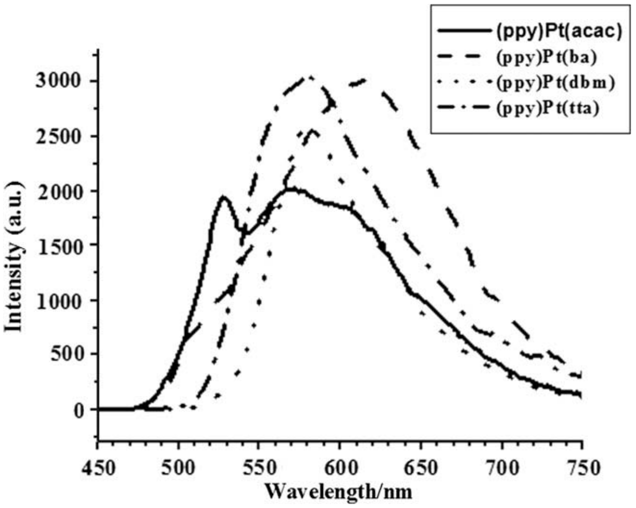

Fig. 5. Emission spectra of the solid crystalline platinum complexes.

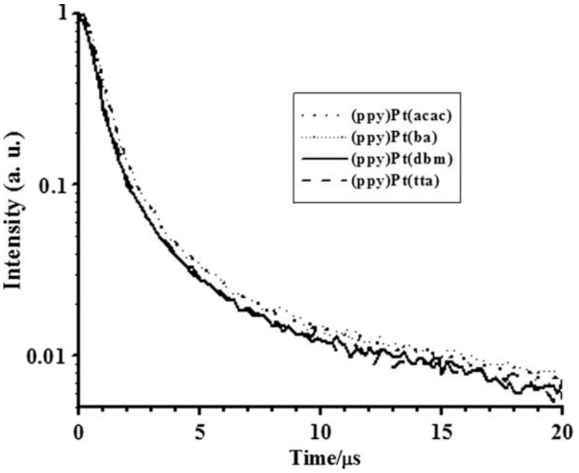

Fig. 6. Emission decay curves for the Pt complexes (measured in CH_2Cl_2 at room temperature).

3.4. The phosphorescence life time

Although the difference is small, the phosphorescence decay pattern can be divided into two groups with one group for (ppy)-Pt(acac) and (ppy)Pt(ba) faster and another slower for (ppy)Pt(dbm) and (ppy)Pt(tta) (Fig. 6). The phosphorescence of all of four complexes decays biexponentially with components of 1.0 μs and 13 μs for (ppy)Pt(acac) and (ppy)Pt(ba) and components of 0.7 μs and 9.0 μs for (ppy)Pt(dbm) and (ppy)Pt(tta). These are consistent with emission from a triplet excited state. The slower decay time for (ppy)Pt(acac) and (ppy)Pt(ba) and faster decay time for (ppy)Pt(dbm) and (ppy)Pt(tta) corresponds to the classification of the emission spectra shape of the four complexes: the former two's vibronic emission structure versus the latter two's broad and featureless emission. The decay time pattern is also in contrast to the quantum yields measurement: the faster decay time corresponds to the lower quantum yields for (ppy)Pt(dbm) and (ppy)Pt(tta).

4. Conclusion

In summary, we synthesized a series of platinum complex coordinating with ppy and different ancillary β-diketone *O^O* ligands and investigated their crystal structure, absorption, emission and quantum yield and emission lifetime. The ancillary *O^O* ligands perturb the excited state of the complex as a whole. In general, larger conjugating π systems lower the triplet state of the *O^O* ligand

and cause the energy back-flow from ppy ligand to the $O^\wedge O$ ligand and switch previously mainly *ppy*Pt centered phosphorescence emission to ttaPt centered emission, therefore cause spectral and quantum yield differences: the quantum yield decreases and the emission spectrum becomes featureless. The life time decay falls in the order of several microseconds, a clear indication of a triplet excited state for the complexes.

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Appendix A. Supplementary material

CCDC 687199 contains the supplementary crystallographic data for this paper. 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 associated with this article can be found, in the online version, at doi:10.1016/j.ica.2008.05.012.

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