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Platinum(II) Complexes with Bis(pyrazolyl)borate Ligands: Increased Molecular Rigidity for Bidentate Ligand Systems

Johannes Soellner,^[a] Piermaria Pinter,^[a] Sergej Stipurin,^[a] and Thomas Strassner*^[a]

Abstract: The structural motif of platinum(II) complexes bearing cyclometalating *N*-heterocyclic carbene ligands can be used to design deep-blue phosphors for application in organic light-emitting diodes. However, the photophysical properties of the resulting molecules are also highly dependent on the auxiliary ligand. These often allow molecular deformations in the excited state which contribute to non-radiative decay processes that diminish the attainable quantum yield. The use of bis(pyrazolyl)borate based auxiliary ligands enforces a high molecular rigidity due to their unique geometry. The steric crowding in the coordination sphere inhibits deformation processes and results in highly efficient deep-blue platinum(II) emitters with CIE coordinates below (0.15; 0,15).

Displays using organic light-emitting diode (OLED) technology are now widely used for screens in televisions and mobile devices. They mostly employ a RGB color scheme, where the polychrome image is generated by an interplay of red, green and blue OLEDs. Highly efficient phosphorescent organometallic molecules^[1] for low energetic red and green emissions are already well established and commercially available.^[2] However, phosphors for deep-blue OLEDs, necessary to cover a broad color range, still fail to excel in terms of color-purity, efficiency and stability, keeping them a relevant research topic.^[2] A prominent approach to obtain deep- to sky-blue phosphors is to combine a platinum(II)^[3] center with C[^]C*-cyclometalating and β-diketonatebased auxiliary ligands.^[3k, 4] Studies on that motif showed that the emission color is largely predefined by the N-heterocyclic carbene (NHC) ligand^[4f] which led to an increased research focus on the optimization of these ligands. Complexes of that type bearing bidentate cyclometalating imidazolylidene and acetylacetonate (acac) ligands generally show very good results regarding their emission color and quantum yields yet still leave some room for improvement. Nevertheless, the straightforward structural variability of two separate bidentate ligand platforms renders this motif an attractive synthetic target. However, bidentate ligands significantly trail behind terdentate or tetradentate ligands in terms of molecular rigidity, which is a prime criterion for phosphorescence efficiency.^[1, 3j, 5] Some modifications to wellestablished auxiliary ligands, like β -diketonates^[4g, 4h, 6] or β diketoiminates^[6c, 7], had positive effects on the quantum yields of the resulting complexes, yet in a number of cases^[6a-c, 7] induced significant bathochromic shifts of the emission, which complicates

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their application in deep-blue emitting platinum(II) complexes. However, these studies proved valuable to verify the significant influence of auxiliary ligands on the photophysical performance of the emitter as a whole. Therefore, we were and still are looking for promising substitutes for established auxiliary ligands and alternative structural approaches to cure the drawbacks of current platinum(II) emitters with bidentate ligand platforms. To our surprise, the bis(pyrazolyl)borate ligands, first synthesized in the laboratories of Trofimenko^[8] in the 1960s, later used by Thompson for iridium complexes have only been scarcely investigated concerning their applicability in phosphorescent platinum(II) complexes.^[9] Due to the tetrahedral geometry of the boron center in these ligands and their relative proximity to the opposing ligand in square-planar platinum(II) complexes we explicitly wanted to study the effect of borate auxiliary ligands on the molecular rigidity and photophysical properties of platinum(II) phosphors. As cyclometalating carbene ligand we chose an imidazolylidene-based motif, since this kind of ligand helped to achieve deep-blue emissions in earlier studies.[4]

The synthesis of ligand precursor **2** starts from 1-(2,4,6-trimethylphenyl)-1*H*-imidazole^[10] **1** which was synthesized according to a literature procedure (Scheme 1)^[10b]. Imidazole **1** is then quarternized utilizing a copper(II)-catalyzed arylation protocol^[11] to yield the imidazolium salt **2** in 72 % yield after column chromatography. Subsequently, a silver(I)-carbene complex is formed upon reaction of **2** with silver(I)-oxide and the NHC is then transmetalated to platinum by addition of dichloro-



Scheme 1. A: General synthetic approach for the syntheses of ligand precursor **2** and target complexes **3-5**. (i) [Ph₂]][BF₄], Cu(OAc)₂·H₂O, DMF, 100 °C. (ii) Ag₂O, DMF, 45 °C. (iii) Pt(COD)Cl₂, DMF, RT to 115 °C. (iv) β-diketone, base, DMF, RT to 100 °C *or* potassium bis(pyrazolyl)borate, DMF, 50 °C. **B:** Synthesized platinum(II) complexes **3-5**.

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(cycloocta-1,5-diene)platinum(II). In the following, the formation of the metallacycle is thermally induced and the appropriate auxiliary ligand is introduced. In the cases of complexes **3a,b** the respective β -diketones are added and deprotonated *in situ* to give the target molecules. For complexes **4** and **5a,b** only the potassium salts of the appropriate borate ligands are added at this stage to form the desired molecules. This was accommodated by slightly milder reaction conditions. All platinum(II) complexes were isolated by chromatography on a silica column and obtained in yields ranging from 16 % to 58 %.

The steric environment around the central platinum atom should vary greatly between the different motifs 3-5. In complexes 3a,b the coordination of the auxiliary ligand is achieved via carbonyl groups, which consequently directs the steric bulk of that ligand away from the coordination sphere. In contrast, the spatial organization around the metal center in complexes 4 and 5a,b seems much more compact, leading to rigid ligand arrangement. Indeed, this is confirmed by the structural characterization via NMR and X-ray diffraction experiments (Figure 1). In the βdiketonate complexes 3a,b equivalent resonances are obtained for the ortho-methyl groups of the NHC-ligand. In complexes 4, 5a and 5b however, the methyl groups are non-equivalent in the NMR experiment, which is best visible in their ¹³C spectra. This is due to the tetrahedral geometry of the boron center, which allows only the nitrogen atoms directly coordinated to the platinum(II) center to be within the coordination plane. This results in the formation of enantiomers (SI, Figure S1) and the non-equivalency of the aforementioned methyl resonances indicates that an interconversion of these enantiomers does not occur at room temperature, unlike observed before.[12]



Figure 1. Solid-state structures of complexes a) **3b**, b) **4**, c) **5a** and d) **5b**. Thermal ellipsoids drawn at 50 % probability. For c) and d): labelling of NHC ligand analogous to a) and b) and along with H atoms omitted for clarity. For d): only one molecule out of the two in the asymmetric unit shown.

In accordance to the experimental results, DFT calculations suggest that for this class of compounds this conversion is hindered by the steric demand of the bis(pyrazolyl)borate ligands (SI, Figure S1). The presence of enantiomers for the complexes with borate ligands can also be concluded from their solid-state structures due to their crystallization in centrosymmetric space groups (4 and 5a) or the explicit presence of both isomers in the asymmetric unit (5b). Notably, the carbon-metal bond lengths are enlarged by approximately 0.02-0.04 Å in the solid-state for complexes 4, 5a and 5b compared to their β-diketonate congener 3b, which reflects the altered steric and bonding situation (SI, Table S1). Futher, the arrangement of the borate ligands relative to the coordination plane proves to be sensitive to substitution of the pyrazole moieties in ortho-position to the metalation. In order to visualize this, we calculated the angle spanned by the planes through Pt(1)/N(3)/N(5) and B(1)/N(3)/N(5) of the solid-state structures of molecules 4, 5a and 5b, respectively (SI, Figure S2). For the structures of 5a and 5b with unsubstituted pyrazole fragments (5a) or substituents pointing away from the core molecule (5b) angles between 51.9 ° to 54.1° are obtained. In the structure of 4 which features methyl substituents in 3- and 5position of the pyrazole moiety, the dihedral is increased significantly to 66.5°. A different kind of steric interaction is visible also for molecules 5a and 5b with bicyclic borate substituent. The hydrogen of the bridgehead CH-group facing the platinum atom projects into the d_7^2 -orbital of the metal (Figure 1, c and d). The assignment of these protons in the crystal structures by the riding model only allows for an approximation of the interatomic distance. Still, the observed Pt-H distances of 2.45-2.58 Å are well below the sum of van-der-Waals radii (2.85 Å^[13]) and support the presence of an atomic interaction. The resonance of the respective protons in the ¹H-NMR thereby experience significant downfield shifts compared to the corresponding protons of the bridgehead group facing away from the core of the molecule (SI, Figure S3).

Thermal stability is of great importance for molecules with potential application in OLEDs since vapor deposition techniques are often used in the assembly of such devices. Thermogravimetric analyses of complexes 3-5, confirmed that the stability platinum(II) complexes with borate-based auxiliary ligands is in a similar region compared to their β-diketonate congeners (SI, Figures S4-S8). For the acetylacetonate complex 3a a mass loss of 5 % occurred at 295 °C. This point of decomposition was notably moved to higher temperature (361 °C) for 3b. Decompositions of complexes 4, 5a and 5b were observed at 315 °C (4), 343 °C (5a) and 357 °C (5b). Additional DSC spectra (Figures S9-S12) are given in the supporting information. The absorption properties of molecules 3-5 were studied in dichloromethane solution and the resulting spectra are given in Figure 2. All complexes show an absorption onset at around 360 nm, with the exception of 3b, which starts to absorb at 380 nm. Aside from that, all absorption spectra possess similar characteristics and show good qualitative agreement: a first clear absorption band is visible at approximately 315 nm and is followed by a second in the range between 280-285 nm before the absorptions reach their maximum at around 275 nm.

At room temperature and in PMMA matrices (Figure 2, Table 1) all complexes **3-5** show emissions in the deep-blue to sky-blue range of the visible spectrum (for solution measurements see SI,

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Table S2, Figure S13). The acetylacetonate parent complex **3a** produces a structured emission spectrum – characteristic for a metal-perturbed ILCT-dominated process – with an emission maximum at 444 nm and a quantum yield of 24 %. Upon introduction of the mesityl substituents in **3b** we observed a complete loss of structure in the respective spectrum, which is accompanied by a significant bathochromic shift, an increase of the efficiency and lowering of the excited state lifetime τ_0 (Table 1). These effects hint towards a distinctly different emission mechanism for **3b** compared to the parent platinum(II) complex **3a**, which has been analyzed by TDDFT calculations (SI, Figures S14,S15). The results point to a LLCT process for **3b**.



Figure 2. Absorption (dashed) and emission (solid) spectra of complexes **3-5**. Absorption spectra recorded as dichloromethane solutions (c = 5×10^{-5} mol/l). Emission spectra recorded as PMMA films (2 wt.-% emitter concentration, N₂ atm., λ_{exc} = 320 nm)

Table 1. Photoluminescence data for complexes **3-5** in a PMMA matrix (2 wt.-% emitter load, room temperature, N₂ atm. λ_{exc} = 320 nm). Data for higher concentrations are given in Table S3 (SI).

	Φ ^[a] [%]	λ _{em} ^[b] [nm]	CIE [c] x;y	το ^[d] [µs]	k _r ^[e] [10 ³ ⋅s ⁻¹]	k _{nr} ^[f] [10 ³ ⋅s ⁻¹]
3a	24	420 , 444	0.16; 0.10	20.1	49.8	157.0
3b	88	485	0.20; 0.34	3.4	296.1	39.6
4	77	425 , 452	0.15; 0.12	10.9	91.8	26.9
5a	87	423 , 449	0.15; 0.11	9.8	102.1	15.5
5b	91	423 , 449	0.15; 0.11	9.1	110.0	11.0

[a] PLQY at RT and λ_{exc} = 320 nm. [b] Emission wavelength of E₀₋₀ transition (bold) and maximum intensity emission wavelength (normal). [c] CIE color coordinates. [d] $\tau_0 = \tau_{exp}/\Phi$. [e] $k_r = \Phi/\tau_{exp}$. [f] $k_{nr} = (1-\Phi)/\tau_{exp}$.

However, the stark red-shift of the emission, observed here and in an earlier study,^[6a] is problematic for a use of this β -diketonate derivative in deep-blue platinum(II) emitters with cyclometalated imidazolylidene ligands. In contrast, the platinum(II) complexes **4**, **5a** and **5b** gave significantly improved quantum yields compared to the parent complex **3a**, while maintaining the ILCT-dominated

emission process with an only marginal shift of the emission to higher energies. Specifically, complex 4 achieved a quantum yield of 77 % - triple the efficiency of acetylacetonate complex 3a. Complexes 5a and 5b with the 9-BBN-derived borate ligands reached even higher quantum yields of 87 % and 91 %, respectively, with emission maxima at 449 nm (5a,b) and 452 nm (4). A rationalization of the observed effect may be derived from the calculated emission kinetics (Table 1). For the parent complex **3a** the non-radiative processes (k_{nr}) clearly outweigh the radiative process (k_r) in terms of the respective rate constants, corresponding to a rather low quantum yield. The situation changes drastically for 3b, for which the radiative decay is more than seven times faster than the non-radiative relaxation, which of course reflects in the efficiency of the emitter. Compared to 3a, k_r increases roughly six-fold for **3b**, while k_{nr} is reduced to a fourth of the value of 3a. Similarly, the radiative rate constants kr for complexes 4-5 are doubled with respect to 3a, k_{nr} on the other hand is reduced so a sixth (4) or even less than a tenth (5a,b) of the value of 3a. In other words, non-radiative decay processes are significantly hindered in the borate complexes 4-5. Theoretical studies of structurally closely related platinum(II) complexes showed,^[14] that these processes largely involve molecular deformations of the excited state molecules, such as an out-ofplane bending of the β -diketonate or even a rearrangement of the coordination geometry towards a pseudo-tetrahedral geometry which corresponds to a metal centered excited state^[14a] (SI, Figure S16). Based on recently published DFT studies,^[14c] we investigated the accessibility of these deformed molecular geometries. While the bent geometries could be optimized for the β-diketonate molecules 3a and 3b (SI, Table S4), comparable geometries were not observed for the borate complexes 4-5. This is consequent with respect to the steric crowding in the coordination sphere, since an out-of-plane bending of the borate ligand would significantly increase the already high steric interaction of carbene and borate ligand. The same holds true for the highly deformed metal-centered state. While the optimized ³MC geometries are more stable for **4-5** compared to **3a,b**, the corresponding activation barrier to access this geometry from the triplet state hypersurface is drastically increased for borate complexes 4-5, making these deformations harder to achieve (SI, Table S5 and Figure S17).

In additionally carried out cyclic voltammetry experiments complexes **3-5** gave almost identical results with irreversible reductions between -2.48 V to -2.54 V (vs. Fc/Fc⁺) and irreversible oxidations in the range of 0.30 V to 0.32 V (SI, Figures S18-S23).

In conclusion, we have prepared a series of platinum(II) complexes with bidentate imidazolylidene ligands combined with β -diketonate and bis(pyrazolyl)borate auxiliary ligands which were thoroughly studied concerning their structural, thermal and photophysical properties. Based on NMR spectra and solid-state structures the complexes with borate ligand showed a sterically more compact coordination sphere compared to their β -diketonate analogs. Both, results of photoluminescence measurements and DFT calculations show that this directly and positively affects the performance of these molecules as emitters. The existing steric interaction between the carbene and borate ligands notably increases the rigidity of the ligand arrangement around the metal center. This in turn prevents molecular

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deformations in the excited state molecules which are prone to cause non-radiative decay. Thereby, efficiencies in the range of 90 % could be achieved for deep-blue emissions. Ongoing experiments indicate that this approach is transferable to structurally related motifs, allowing for a straight-forward increase in efficiency, while still maintaining the structural versatility of bidentate ligand platforms.^[15]

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Deposition Numbers 2003958-2003961 contain the supplementary crystallographic data for this paper. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre at www.ccdc.cam.ac.uk/structures.

Keywords: deep-blue • platinum • *N*-heterocyclic carbene • OLED • phosphorescence

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