New boron complex based on 3-(2-pyridyl)pyrazole Chelates of bis(4-tert-butylphenyl): Synthesis and X-ray Crystal Structure

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Abstract: The synthesis and crystal structure of (B*t*-*Bu*Ph_2(3-(2-pyridyl)pyrazolate)) complex is described. The structure of the complex was determined by using single crystal X-ray diffraction. The compound crystallized in symmetry cell setting orthorhombic of P_{212121} with a = 11.067(2) Å, b = 11.202(2) Å, c = 20.542(4) Å and Z = 4. The structure of complex reveals that the boron centre is four coordinated. The formed compound, emitting materials which could be of interest for practical applications.

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Keywords: 3-(2-pyridyl)pyrazole; boron complex; crystal structure

1. Introduction

Considerable attention has been focused on studying the organboron compounds due to their interest for practical applications (Song et al., 2001; Liu et al., 2002; Lee et al., 2004) such as organic light emitting devices (OLED) (Esteghamatian et al., 2000; Li et al. 2000) as well as biomolecular porbes (James, et al.1996; Rurack et al., 2001). It has been interested to study the physical properties of the boron complex by design a new ligand chromophores so that can be fine-tuned to achieve a designated function. Boron compound with N,N-chelates(McDonnell and O' Shea 2006) and N,O-chelates ligand(Cui, et al., 2005); have been used widely in important applications. The imidazoles and its relative such as pyrazole have been known as good chelating ligands(Balamurugan and Palaniandavar, 2001); and the attachment of the pyridyl ring at 3-position of pyrazole would allow the ligand to form stable compounds upon treatment with a boron compound such as BPh_3, giving a nearly planar, conjugated π-system that was perfect for studying the photoluminescent properties (Cheng et al. 2003). In this present work, the synthesis, crystal structure of a new born compound B*t*-*Bu*Ph_2(3-(2-pyridyl)pyrazolate) is reported.

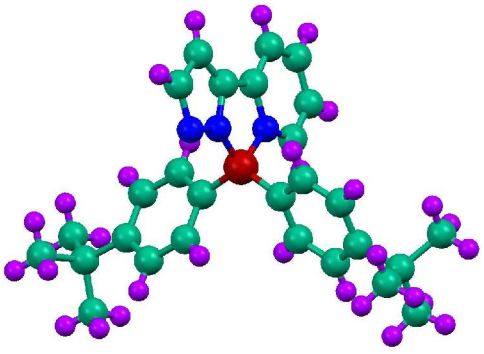
2. Material and Methods

Synthesises. Ligand 3-(2-pyridyl)pyrazole was prepared as previously reported (Bell et al., 2003). Complex B*t*-*Bu*Ph_2(3-(2-pyridyl)pyrazolate) was prepared in THF by reacting, in 1:1.1 molar ratio Potassium tetrakis(4-tert-butylphenyl)borate with 3-(2-pyridyl)pyrazole. The solutions were refluxed for 8h under nitrogen. After the solvent was removed, the residues were purified by sublimation method. Selected analytical data concerning the complex is a following. White powder, 46% yield. M. P.>200 ºC. ^{1}H NMR (250 MHz,$CDCl_3$): 8.56 (1H, d, pyridyl H6), 7.18(1 H, t, pyridyl H5), 7.45(1H, t, pyridyl H4), 7.23 (1H, d, pyridyl H3), 7.07 (1H, d, pyazolyl H5), 6.80 (1H, d, pyrazolyl H4), 7.67(4H, d, phenyl H4,H6), 7.46(4H, d, phenyl) H3,H5), 1.35(18H, s, methyl). ES mass spectrum: m/z 421. Found: C, 79.43; H, 7.42; N, 9.91 %. $C_{23}H_{19}N_7$ required C, 79.81; H,7.65; N, 9.97 %.

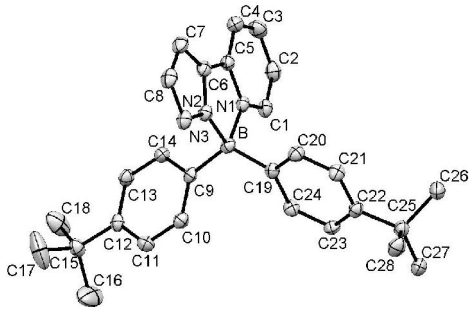
3. Results and discussion

The compound is air-stable in the solid state and in solution. Single structure of the compound was grown by slow diffusion of diethyl ether into a concentrated solution of the complex in dichloromethane. The molecular structure of the complex is shown in figure 1 and the selected bond lengths and angles are listed in Table 1. In this structure (see table 2), the boron atom in compound has a characteristic tetrahedral geometry with angles N(1)-B-C(9) = 107.46(16) Å and C(19)-B-C(9) = 119.05(18) Å. The 3-(2-pyridyl)pyrazole is potentially tridentate ligand which chelated to boron centre as bidentate ligand through its pyridyl and pyrazole nitrogen (N2) atoms, and two 4-*tert*-*Bu*phenyl ring occupy the other boron sites. The bonds length of B-N(1) pyridyl and B-N(2) pyrazole is (1.635(3) Å) and (1.570(3) Å) respectively. The B-N(1) of pyridyl is shorter than that of B-N(2) pyrazole which is indicating that the N(1)-atom of pyridyl is anionic and hence a better donor than the N(2)-atom of pyrazole. This observation is consistent with the structural data reported in other tetrahedrally arranged BPh_2 complexes with chelating ligands of (2-pyridyl)-7-azaindole and (2-pyridyl)-7-indole derivatives (Liu et al., 2000; Liu et al., 2002).

The photophysical properties of the boron complex are currently in progress.



(A)



(B)

Figure 2. Molecular structure of (a) complex with hydrogen atoms and (b) with atom labeling. Hydrogen atoms omitted for clarity.

Table 1. Selected bond distance (Å) and angles (°) for complex.

N(1)-C(5)	1.366(3)	C(1)-N(1)-C(5)	120.85(18)
N(1)-B	1.635(3)	C(1)-N(1)-B	125.75(17)
N(2)-N(3)	1.345(2)	N(3)-N(2)-C(6)	111.46(17)
N(2)-B	1.570(3)	N(3)-N(2)-B	133.42(17)
N(3)-C(8)	1.349(3)	N(2)-B-C(9)	110.78(17)
C(1)-C(2)	1.369(3)	C(19)-B-C(9)	119.05(18)
B-C(19)	1.598(3)	N(1)-C(1)-C(2)	120.8(2)

Table 1. Crystal data and structure refinement summery for complex.

formula	C_{29} H_{36} B N_3O
Formula weight	453.42
Temperature (K)	150(2)
Wavelength (Å)	0.71073
Crystal system	Orthorhombic
Space group	P212121
Unit cell dimensions	
a (Å)	11.067(2)
b (Å)	11.202(2)
c (Å)	20.542(4)
α (o)	90
β (o)	90
γ (°)	90
V, (Å^3)	2546.5(8)
Z	4
D. calcd, (Mg/m^3)	1.183
μ, (mm-1)	0.071
F(000)	976
Cryst. size (mm)	0.34 x 0.32 x 0.26
Theta range for data collection (°)	1.98 to 28.63
Limiting indices	-14<=h<=14,
	-14<=k<=14,
	-26<=l<=27
Reflections collected	29716
Refinement method	Full-matrix
	Last squares on F2
Data/restraints/ parameters	3521 / 0 / 316
Goodness-of-fit on F^{-2}	1.049
Final R indices [1 >2σ(1)]	R1 = 0.0426, wR2 = 0.0899
R indices (all data)	R1 = 0.0659, wR2 = 0.0962
Largest diff. peak and hole (e Å^{-3})	0.263 and -0.181

_Supplementary material

Crystalographic data for the structural analysis has been deposited with the Cambridge Crystalographic Data Centre as supplementary publication. CCDC 923562. Copies of data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 01223 336033 or e-mail: deposit@ccdc.cam.ac.uk).

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