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Synthesis and structural characterization of Pd(II) complexes containing 2,6-bis[(dimethylamino)methyl]-4-methylphenolate ligand

Debasish Ghorai, Ganesan Mani*

Department of Chemistry, Indian Institute of Technology, Kharagpur 721 302, India

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Dedicated to Professor S.S. Krishnamurthy on the occasion of his 70th birthday.

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ABSTRACT

Treatment of 2,6-bis[(dimethylamino)methyl]-4-methylphenol (1) with $[Pd(PhCN)_2Cl_2]$ in a 1:1 molar ratio gives the mononuclear Pd(II) complex $[PdCl_2(OC_6H_2(CH_2NMe_2)-2-Me-4-(CH_2NHMe_2)-6)]$ (2) containing one ligand with an ammonium hydrogen atom, which forms a bifurcated hydrogen bonding to the phenoxy oxygen and the chlorine atoms, as shown by the single crystal X-ray diffraction study. The reaction between the lithium salt of 1 and $[Pd(COD)Cl_2]$ gives the mononuclear Pd(II) complex $[Pd(OC_6H_2(CH_2NMe_2)_2-2,6-Me-4)_2]$ (3). The X-ray structure of 3 showed the presence of two ligands coordinated to one palladium metal center in a *trans* fashion with two dangling dimethylamine groups. The yield of the complex 3 was improved by carrying out the reaction between $[Pd(OAc)_2]$ and 1 in acetone. The solid state structures of the complexes 2 and 3 were confirmed by ¹H, ¹³C, HETCOR NMR, IR and elemental analysis methods. The ¹H NMR spectra of 2 and 3 showed two different chemical shifts corresponding to the coordinated and uncoordinated amine groups of the ligand. No decoalescence of signals for the chelate ring puckering process was observed in variable-temperature NMR spectra. © 2011 Elsevier B.V. All rights reserved.

1. Introduction

2,6-Bis[(dimethylamino)methyl]-4-methylphenol **1** is a versatile, an easy-to-synthesize and a bis-chelating phenoxy ligand. As it has an acidic phenolic proton, it can coordinate to a metal center as a monoanionic-monodentate, bidentate and bridging ligand and stabilizes metal complexes in various oxidation states [1].

N OH N

The steric and electronic nature of this aminophenolate ligand can readily be changed to fine tune the property of its complex, which is often used as a catalyst for reactions such as ring-opening metathesis polymerization of 2-norbornene [2], lactide polymerization [3] and the hydrolysis of β -lactam substrates [4]. van Koten and co-workers reported vanadium complexes of **1** which are catalysts for the polymerization of α -olefins [5]. Here, we first report the synthesis and the structural characterization of new Pd(II) complexes of **1**.

2. Experimental

2.1. General procedures

Solvents were distilled before use by following standard procedures. All reactions and manipulations were carried out under nitrogen atmosphere using standard Schlenk line techniques. ¹H NMR (200 and 400 MHz) and ¹³C NMR (51.3 and 102.6 MHz) spectra were recorded on Bruker ACF200 and AV400 spectrometers. Chemical shifts are referenced with respect to the chemical shift of the residual proton present in the deuterated solvents. FTIR spectra were recorded using Perkin-Elmer spectrophotometer RX-1. The [Pd(PhCN)₂Cl₂] [6], [Pd(COD)Cl₂] [7] and 2,6-bis[(dimethylamino)methyl]-4-methylphenol [8] were prepared according to the literature procedures. Other chemicals were obtained from commercial sources and used without further purification.

2.2. Synthesis of complex [PdCl₂(OC₆H₂(CH₂NMe₂)-2-Me-4-(CH₂NHMe₂)-6)] (**2**)

To a CH_2Cl_2 solution of 2,6-bis[(dimethylamino)methyl]-4methylphenol (0.0145 g, 0.065 mmol), [PdCl_2(PhCN)_2] (0.025 g, 0.065 mmol) was added at room temperature. The resulting solution was refluxed for 4 h to give a red color solution. The solution was cooled to room temperature and then concentrated to ~2 mL, to which diethyl ether (20 mL) was added to give orange-red precipitate of **2**. The precipitate was filtered, washed with diethyl ether and then dried under vacuum. Yield: 0.022 g, 84%. **2** was



Note



^{*} Corresponding author. Tel.: +91 3222 282320; fax: +91 3222 282252. *E-mail address*: gmani@chem.iitkgp.ernet.in (G. Mani).

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crystallized from dichloromethane solution upon layering with diethyl ether. mp 70 °C (decomposed). ¹H NMR (CDCl₃): δ (ppm), 2.12 (s, 3H, Ph-CH₃), 2.62 (s, 6H, coordinated NMe₂), 2.89 (d, 6H, ³J(H,H) = 4.8 Hz, HN⁺Me₂), 2.95 (s, 2H, coordinated Me₂NCH₂), 4.11 (d, 2H, ³J(H,H) = 5.2 Hz, Me₂N⁺HCH₂), 6.81 (s, 1H, Ph), 6.89 (s, 1H, Ph), 10.22 (br s, 1H, NH). ¹³C NMR (CDCl₃): δ (ppm) 20.4 (Ph-CH₃), 42.7 (HN⁺Me₂), 50.8 (coordinated NMe₂), 60.5 (CH₂N⁺H), 63.3 (coordinated CH₂NMe₂), 117.6 (Ph), 125.9 (Ph), 126.9 (Ph), 131.5 (Ph), 132.3 (Ph), 161.1 (Ph). IR (KBr, ν , cm⁻¹): 3440 (br, m, NH), 3015 (m), 2918 (m), 2843 (m), 1720 (w), 1636 (w), 1468 (vs), 1266 (s), 1101 (m), 1018 (w), 839 (w), 798 (m), 593 (w), 527 (w). Anal. Calc. for C₁₃H₂₂N₂OPdCl₂ (399.63): C, 39.07; H, 5.55; N, 7.01. Found: C, 38.24; H, 5.33; N, 6.58%.

2.3. Synthesis of complex $[Pd(OC_6H_2(CH_2NMe_2)_2-2,6-Me-4)_2]$ (3)

2.3.1. Method a

To a solution of **1** (0.099 g, 0.44 mmol) in acetone, $[Pd(OAc)_2]$ (0.05 g, 0.22 mmol) was added and stirred at room temperature for 24 h, resulting in a yellow–orange solution. The solvent was removed under vacuum and the resultant residue was extracted with petroleum ether (10 mL) two times. The solvent was removed again to give **3** (0.1 g, 0.18 mmol, 82% yield). A solution of **3** in acetone/hexane (1:2 v/v) mixture was allowed to stand for the slow evaporation and crystals of **3** formed after 5 days.

2.3.2. Method b

To a solution of 1 (0.31 g, 1.4 mmol) in diethyl ether (50 mL) at -78 °C, ⁿBuLi (0.9 mL, 1.6 M, hexanes solution) was added with stirring. The solution was brought slowly to room temperature. After stirring for 1 h, [Pd(COD)Cl₂] (0.2 g, 0.7 mmol) was added and stirred for additional 12 h. The solvent was removed under vacuum and the residue was extracted with petroleum ether (30 mL) to give yellow-orange solution. The solution was concentrated to ~ 15 mL and left at room temperature for seven days to give needle shaped yellow colored crystals of 3 (0.127 g, 33%). mp 165 °C (decomposed). ¹H NMR (CDCl₃): δ (ppm) 2.15 (s, 6H, PhCH₃), 2.19 (s, 12H, dangling NMe₂), 2.63 (s, 12H, coordinated NMe₂), 3.24 (s, 4H, coordinated CH₂), 3.33 (s, 4H, dangling CH₂), 6.62 (s, 2H, Ph), 6.92 (s, 2H, Ph). ¹³C NMR (CDCl₃): δ (ppm) 20.4 (PhCH₃), 45.6 (dangling NMe₂), 48.4 (coordinated NMe₂), 59.3 (dangling CH2), 64.5 (coordinated CH2), 123.5 (Ph), 124.2 (Ph), 126.7 (Ph), 129.7 (Ph), 132.2 (Ph), 161.8 (Ph). IR (Nujol, v, cm⁻¹): 2807 (m), 2758 (m), 1607 (w), 1364 (w), 1316 (w), 1264 (m), 1145 (w), 1092 (w), 1024 (m), 979 (w), 877 (w), 846 (w), 807 (m) 627 (w), 548 (w). Anal. Calc. for C₂₆H₄₂N₄O₂Pd (549.04): C, 56.87; H, 7.71; N, 10.21. Found: C, 57.01; H, 7.64; N, 9.99%.

2.4. X-ray crystallography

Single crystal X-ray diffraction data collections were performed using Bruker-APEX-II CCD diffractometer with graphite monochromated Mo K α radiation (λ = 0.71073 Å). The crystals were glued onto the tip of glass fibers using Wembley's Quickfix. The unit cell parameters were determined by using three matrices each consists of 12 frames of reflections and then the data were collected with a set up of 10 seconds per frame for every 0.5°. The structures were solved by direct methods, which successfully located most of the non-hydrogen atoms. Subsequently, least square refinement was carried out on F^2 using SHELXL-97 [9] (WinGX version) to locate the remaining non-hydrogen atoms. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms except H2 attached to N2 were fixed on calculated positions using riding models and were refined isotropically. The hydrogen atom attached to N2 was located in difference map and refined isotropically with thermal parameter equivalent to 1.2 times that of N2.

3. Results and discussion

3.1. Synthesis and spectroscopic characterization of the palladium(II) complexes

The Pd(II) complex **2** was synthesized by the reaction between an equimolar amount of the ligand 1 and [Pd(PhCN)₂Cl₂] in 84% yield (Scheme 1). 2 is an orange solid that is soluble in solvents such as CH₂Cl₂, CHCl₃ and THF. The structure of this complex 2 was first established by single crystal X-ray diffraction study which was then confirmed by NMR, IR and elemental analysis methods. The ¹H NMR spectrum of **2** displays two sets of resonances for the coordinated and the protonated (dimethylamino)methyl groups. The NMe₂ methyl groups and the benzylic protons of the protonated amine group show doublets at δ 2.89 and 4.11 ppm, respectively, due to the coupling with N⁺H proton, which resonates as a broad singlet at δ 10.22 ppm. The chemical shift assignments are based on the HETCOR spectrum of **2** (see Supplementary data). In addition, the ¹H NMR spectrum of **2** shows a considerable change in the chemical shift of other protons of the ligand with respect to the ¹H NMR spectrum of the free ligand and their integrated intensities are in accord with the structure. The presence of NH group is further confirmed by the stretching frequency observed at 3440 cm^{-1} .

Variable-temperature NMR study of the puckered six membered chelate ring has been reported for W and B complexes of **1** [1a,h]. To detect the formation of the conformers resulting from the ring-flipping process variable-temperature ¹H NMR spectra of **2** in CD₂Cl₂ were recorded. When the temperature is reduced from the room temperature to -80 °C with 10 °C incremental reductions the signals due to the benzylic and the NMe₂ methyl groups are broadened without decoalescence (see Supplementary data). This indicates a fast exchange going on between the conformers resulting from the puckering of the six membered chelate ring on the NMR time scale even at -80 °C.

When the ligand **1** is treated with $[Pd(OAc)_2]$ in acetone at room temperature the mononuclear Pd(II) complex **3** is formed in 82% yield. This complex is formed by substituting both the acetate anions of $[Pd(OAc)_2]$ with the anions of the ligand, resulting in the concomitant formation of acetic acid. The complex **3** was also obtained in 33% crystalline yield when two equivalents of the lithium salt of the ligand, which was generated in situ by the addition of "BuLi to a solution of **1** in diethylether, is treated with one equivalent of $[Pd(COD)Cl_2]$ (Scheme 2).

The complex **3** was characterized by both spectroscopic and Xray methods. In the ¹H NMR spectrum, **3** shows two distinctive chemical shifts for the NMe₂ and the benzylic groups, as shown by **2**, and its chemical shifts are assigned based on the HETCOR spectrum of **3** (see Supplementary data). The resonances due to the other protons of the complex are also changed on comparison with that of the free ligand and their integrated intensities are matching each other, which confirm the observed solid state structure (see below). Since **3** has dangling (dimethylamino)methyl groups and coordinated arms the ¹H NMR of **3** was recorded for



Scheme 1. Synthesis of [PdCl₂(OC₆H₂(CH₂NMe₂)-2-Me-4-(CH₂NHMe₂)-6)] (2).



Scheme 2. Synthesis of [Pd(OC₆H₂(CH₂NMe₂)₂-2,6-Me-4)₂] (3).

every 10 °C increase from room temperature to 60 °C to check whether there is an exchange between them. The elevated temperature spectra appeared the same as the room temperature one, indicating no exchange between the coordinated and the free (dimethylamino)methyl groups at the Pd metal center and the rigidness of the Pd–N bond on the NMR time scale, as reported for the tungsten complex [1a]. Variable-temperature ¹H NMR spectra (see Supplementary data) show the broadening of signals and no decoalescence, indicating the ring-flipping process does not slow down even at -60 °C in CDCl₃ on the NMR time scale.

3.2. Crystal structure analyses

3.2.1. Complex 2

The molecular structure of **2** is shown in Fig. 1. Crystallographic refinement data, and selected bond lengths and bond angles are given in Tables 1–3, respectively. The X-ray structure reveals that one ligand **1** as a zwitterionic form is coordinated through its N and O atoms to the Pd(II) metal center, forming a six membered chelate ring which is puckered. The protonated amine group (N2) acts as a bifurcated donor and forms a bifurcated hydrogen bond with the coordinated oxygen atom (O1) and chloride atom (Cl2) of the Pd(II) metal center. The N2–O1 distance is considerably shorter than the sum of the van der walls radii (3.07 Å), while the N2–Cl2 distance is only slightly lower than the sum of the van der wals radii (3.30 Å). The hydrogen atom sits in the plane formed by N2, O1 and Cl2 atoms and the sum of the angles of N(2)-H(2)···O(1) (140.38°), N(2)-H(2)···Cl(2) (143.63°) and



Fig. 1. Molecular structure of 2. The thermal ellipsoids are drawn at the 50% probability level.

Cl2-H2-O1 (75.78°) equals to 359.79°, indicating the efficacy of a bifurcated hydrogen bond [10].

The zwitterionic form with its bifurcated hydrogen bond is probably formed by the abstraction of proton from the acidic OH group of the ligand. Similar structures containing the zwitterionic form of 2,6-bis[(dimethylamino)methyl]-4-methylphenol 1 with a bifurcated hydrogen bond have been reported for Zn and V metal complexes [1f,11]. The geometry around the Pd(II) metal center is a distorted square planar with O(1)-Pd-Cl(1) and N(1)-Pd-Cl(2) angles of 174.90(5)° and 176.59(8)°, respectively. Owing to the presence of less bulky groups on N2 the average C-N-C bond angle around the protonated amine nitrogen atom N2 is higher (112.2°) than that (109.5°) found at N1 which is bonded to Pd metal. The Pd–N1 distance (2.060 Å) is shorter than that reported for an analogous sp³ nitrogen coordinated Pd(II) complexes (2.1037, 2.092 and 2.110 Å) containing five membered chelate rings [12]. The bite angle of the ligand $(O(1)-Pd-N(1) = 92.20^{\circ})$ is higher than the bite angles (82.00° and 81.94°) reported for these Pd(II) complexes. But the Pd-N1 distance is longer than that reported for a Pd(II) complex containing sp² coordinated nitrogen atom [13]. The Pd-O1 distance (2.0106) is slightly longer than that reported (1.987 and 1.974 Å) for the Pd(II) complexes [14,15].

3.2.2. Complex 3

The complex **3** crystallizes in C2/c space group and one half of the molecule is present in the asymmetric unit with the Pd metal at the inversion center. The crystal structure of **3** is shown in Fig. 2. Crystallographic refinement data, and selected bond length and bond angles are given in Tables 1 and 4, respectively.

The X-ray structure reveals that two phenolate ligands are coordinated to one Pd(II) metal center. Each ligand uses one of its NMe₂ nitrogen atoms and the phenoxide oxygen atom to form a six membered chelate ring which is puckered; the other (dimethylamino)methyl arm is dangling and away that reduces steric interactions. The geometry around the Pd metal center is distorted square planar, as shown by the bond angles. The two coordinated oxygen atoms are *trans* to each other, as are the two nitrogen atoms, probably owing to the steric hindrance of the (dimethylamino)methyl groups. As described for the structure of **2**, the Pd–N distance (2.082(5) Å) is shorter than that found in the reported Pd(II) complexes [12]. The Pd–O bond distance (2.021(4) Å) is slightly longer than that found in the complex **2** and in other Pd(II) complexes [14,15].

The used diaminophenoloate ligand is a bulky aryloxy ligand and there can be a repulsion between the lone pairs on the oxygen atom and the filled metal d orbitals. This can cause the oxygen atom to adopt sp² hybridization, as shown by the angles (C9– O1–Pd = 118.68° for **2** and C10–O1–Pd1 = 117.3° for **3**) and the puckering of the chelate rings observed in both complexes. As a result the lone pair on the oxygen atom becomes increasingly suitable for forming hydrogen bond with the protonated amine hydrogen atom (Fig. 1) and the Pd–O bond distances of both complexes are longer. Since Pd(II) metal has filled d orbitals and is bonded to two hard donors (O, N) the lone pairs on the chlorine atom (Cl2) rather involved in hydrogen bond than does act as π donor ligand. This is reflected in the bond distances; the Pd–Cl2 is longler (2.3125(6) Å) than the Pd–Cl1 distance (2.2975(7) Å) [16].

In conclusion, two new mononuclear Pd(II) complexes of 2,6bis[(dimethylamino)methyl]-4-methylphenol ligand are synthesized and structurally characterized. Owing to a fast exchange the conformers resulting from the ring-flipping process could not be detected by variable-temperature NMR studies for both the complexes. Catalytic properties of these Pd(II) complexes are under investigation. In addition the complex **3** contains two uncoordi-

Table 1

Crystal data and details of the structure refinement for 2 and 3.

	2	3
Empirical formula	C13H22Cl2N2OPd	$C_{26}H_{42}N_4O_2Pd$
Formula weight	399.63	549.04
Radiation (Å)	0.7107	0.7107
T (K)	293(2)	293(2)
Crystal system	monoclinic	monoclinic
Color and shape	red needle	red needle
Space group	C2/c	C2/c
a (Å)	18.6557(7)	17.767(8)
b (Å)	15.4400(5)	17.659(8)
<i>c</i> (Å)	13.9569(5)	9.602(4)
α (°)	90	90
β(°)	110.4050(10)	117.090(5)
γ(°)	90	90
$V(Å^3)$	3767.9(2)	2682(2)
Ζ	8	4
$D_{\text{calcd}} (\text{mg m}^{-3})$	1.409	1.36
μ(Mo Kα) (mm ⁻¹)	1.263	0.72
F(0 0 0)	1616	1152
Crystal size (mm)	$0.46 \times 0.12 \times 0.10$	$0.38 \times 0.02 \times 0.02$
Total/unique number of reflections	27 895, 5980	14 113, 2840
R _{int}	0.0255	0.1475
R_1 , wR_2	0.0346, 0.0923	0.0646, 0.1571
R_1 , wR_2 (all data)	0.0501, 0.0983	0.1131, 0.1907
Largest different peak and hole	0.807 and -0.454	1.486 and -2.119
$(e Å^{-3})$		

Table 2

Hydrogen bonds distances (Å) and angles (°) in the structure of 2.

D–H…A	d(D-H)	d(H…A)	d(D…A)	<(DHA)
$N(2)-H(2)\cdots O(1)$	0.73(3)	2.08(3)	2.688(2)	140(3)
$N(2)-H(2)\cdots Cl(2)$	0.73(3)	2.63(3)	3.248(2)	144(3)

Table 3

Selected bond lengths (Å) and bond angles (°) for $\mathbf{2}$.

Pd-O(1)	2.0106(14)	N(1)-Pd-Cl(2)	176.59(8)
Pd-N(1)	2.060(2)	Cl(1) - Pd - Cl(2)	91.16(3)
Pd-Cl(1)	2.2975(7)	C(9)-O(1)-Pd	118.68(12)
Pd-Cl(2)	2.3125(6)	C(1)-N(1)-Pd	110.4(2)
O(1)-C(9)	1.340(3)	C(3)-N(1)-Pd	111.08(18)
N(1)-C(3)	1.500(4)	C(2)-N(1)-Pd	106.9(2)
N(2)-C(11)	1.493(3)	C(1)-N(1)-C(2)	111.2(3)
O(1) - Pd - N(1)	92.20(9)	C(1)-N(1)-C(3)	107.7(3)
O(1)-Pd-Cl(1)	174.90(5)	C(3)-N(1)-C(2)	109.6(2)
O(1)-Pd-Cl(2)	84.82(4)	C(12)-N(2)-C(13)	111.55(19)
N(1)-Pd-Cl(1)	91.91(8)	C(12)-N(2)-C(11)	112.5(2)
		C(13)-N(2)-C(11)	112.66(19)



Fig. 2. Molecular structure of **3**. The thermal ellipsoids are drawn at the 50% probability level. Hydrogen atoms are omitted for clarity.

Table 4

Selected bond lengths (Å) and bond angles (°) for 3.

Pd(1)-O(1)	2.021(4)	O(1)-Pd(1)-N(1)	92.65(18)
Pd(1)-N(1)	2.082(5)	O(1i)1-Pd(1)-O(1)	180.0(3)
O(1) - C(10)	1.327(8)	C(10) - O(1) - Pd(1)	117.3(3)
N(1)-C(3)	1.479(8)	C(3)-N(1)-Pd(1)	112.5(3)
C(4) - C(3)	1.507(8)	C(10)-C(4)-C(3)	117.2(6)
C(11) - C(9)	1.509(9)	N(1)-C(3)-C(4)	115.3(5)
C(11) - N(2)	1.461(8)	O(1)-C(10)-C(4)	122.6(5)
		C(10)-C(9)-C(11)	119.1(6)

nated amine groups which could be used to build heteronuclear complexes.

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Appendix A. Supplementary material

CCDC 794416 and 794417 contains the supplementary crystallographic data for **2** and **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 associated with this article can be found, in the online version, at doi:10.1016/j.ica.2011.01.098.

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