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Note

Regiospecific C(naphthyl)–H bond activation: Isolation and characterization of cyclopalladates

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Abstract

In ethanol medium disodium tetrachloropalladate selectively activates the C8–H bond of naphthyl group present in 1-(2′-hydroxy-5′-methylphenylazo)naphthalene(H_2L^1) at room temperature and produces cyclometallate [$PdL^1(PPh_3)$] in presence of triphenylphosphine. Under similar reaction condition, the C(naphthyl)–H bond activation of 1-(2′-methoxy-5′-methylphenylazo)naphthalene (HL^2) occurs at C2 and cyclopalladate [PdL^2Cl] has been isolated as product. The labile nature of the Pd ←:O(R) bond of [PdL^2Cl] in solution is established from the electronic and NMR spectra. Cyclopalladate [PdL^2Cl] reacts with thallium(I) cyclopentadienide and yields [$PdL^2(Cp)$], where both σ- and π-palladium(II)-carbon bonds exist. All the cyclopalladates have been isolated in pure form and characterized on the basis of their spectral data. The molecular structure of cyclopalladate [$PdL^1(PPh_3)$] has been determined by single crystal X-ray diffraction method. In [$PdL^1(PPh_3)$], the metal ion is bonded to C8 (*peri*-position of 1-azonaphthyl fagment), N1 of diazene function, O1 of phenolic group and P1 of triphenylphosphine. The tetra-coordination around palladium(II) is in a distorted square-planar geometry.

Keywords: Cyclopalladation; Palladium(II)-C(naphthyl) bond; Peri-palladates; Ortho-palladates; Crystal structure

1. Introduction

The regioselective intramolecular C–H bond activation of aryl ring *via* cyclometallation and subsequent functionalization at metallated carbon is of special significance in organic synthesis [1]. There has been intense interest in the chemistry of cyclopalladated compounds [2,3], which have numerous applications in organic synthesis [4–8], catalysis [9–15], photochemistry [16–20] and metallomesogen chemistry [21–23]. The C(naphthyl)–H bond activation by palladium(II) following cyclometallation route has received much less attention in contrast to the C(phenyl)–H bond activation [2,3], in spite of the fact that the naphthalene ring is more reactive towards electrophilic metal ions [24]. The naphthalenes with donor bearing substituent like diazene function at C1 can offer either the *ortho*- (C2) or the *peri*- (C8) position for metallation. Rys et al. have thoroughly studied [25–27] the cyclopalladation reaction of 1-(phenylazo)naphthalene and established that the palladium-carbon bond formation occurs at C2 (naphthyl) exclusively. The *peri*-position (C8) of 1-(phenylazo)naphthalene was palladated by disodium tetracloropalladate, only when all the *ortho*-positions of both naphthyl and phenyl rings were blocked by methyl groups [27].

In continuation of our interest [28–30] in the synthesis and reactivity of cyclopalladated naphthyl moiety, the present report deals with the regiospecific C(naphthyl)–H bond activation of 1-(phenylazo)naphthalene system, modified by 2′-substitution with auxiliary donors (**1** and **2**), by disodium tetrachloropalladate at room temperature. The effect of auxiliary donors on the selectivity of

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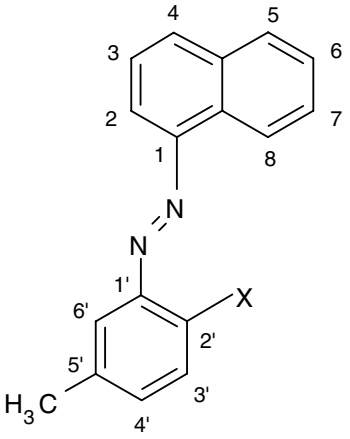
C(naphthyl)–H bond activation has been examined The isolation and characterization of the resulting cyclopalladates have also been described.



1 : X = OH; H_2L^1

2 : X = OMe; HL^2

2. Experimental

2.1. Materials and general methods

All chemicals and solvents used for the work were of reagent grade and used without purification. 1-(2′-hydroxy-5′-methylphenylazo) naphthalene (HL^1) was prepared following the reported method [31]. 1-(2′-methoxy-5′-methylphenylazo) naphthalene (HL^2) was prepared by methylation of 1-(2′-hydroxy-5′-methylphenylazo)naphthalene [32]. Thallium (I) cyclopentadieneide was prepared following reported method [33].

Elemental microanalyses (C, H and N) were done by either Perkin–Elmer (Model 240C) or Heraeus Carlo Erba 1108 elemental analyzer. The IR and Electronic spectra were recorded on Jasco 5300 FT-IR spectrophotometer and Shimadzu UV-240 spectrophotometer respectively. NMR spectra were obtained by using Bruker DPX 300 NMR Spectrometer.

A suitable single crystal of $[PdL^1(PPh_3)]$ (**3**) was mounted on a thin glass fiber and transferred to a Siemens Smart-CCD diffractometer equipped with a normal focus, 2.4 kW sealed tube X-ray source (MoKα radiation, $\lambda = 0.71073$ Å) operating at 50 kV and 40 mA. The structure was solved by direct methods using SHELXS-97 [34] and difference Fourier syntheses. An empirical absorption correction based on symmetry equivalent reflections was applied for the compound using SADABS [35] program. All the hydrogen positions for the compound were initially located in the difference Fourier map, and for the final refinement, the hydrogen atoms were placed geometrically and held in the riding mode. The last cycles of refinement included atomic positions for all the atoms, anisotropic thermal parameters for all non-hydrogen atoms and isotropic thermal parameters for all the hydrogen atoms. Full-matrix-least-squares structure refinement against $|F^2|$ was carried out using SHELXL-97 [36] package of programs.

2.1.1. Synthesis of $[Pd(L^1)(PPh_3)]$ (3)

A solution of 1-(2′-hydroxy-5′-methylphenylazo)naphthalene (H_2L^1) (0.06 g, 0.23 mmol) in ethanol (20 mL) was added drop-wise to an ethanolic solution (20 mL) of disodium tetrachloropalladate (0.08 g, 0.27 mmol) with stirring at room temperature.

The stirring of the mixture was continued for 2 h and precipitate started to appear slowly. A solution of triphenyl phosphine (0.08 g, 0.30 mmol) in benzene (10 mL) was added drop-wise to the above mixture. The mixture was further stirred for 0.5 h and the colour of the reaction mixture was changed from red to pink. After the removal of the solvent, the residue was washed with water and ethanol respectively and finally dried. The solid mass was dissolved in dichloromethane and chromatographed on silica gel (60–120 mesh size). An orange yellow band of unreacted (**1**) was eluted by a mixture of petroleum ether and benzene (1:1, v/v) followed by a pink band of cyclopalladate **3**, which was eluted by pure benzene. The removal of solvent afforded microcrystals of cyclopalladate $[PdL^1(PPh_3)]$ which was finally dried in vaccuo. (Yield: 0.08 g, 0.127 mmol, 55.2%). *Anal.* Calc. for $C_{35}H_{27}N_2OPPd$: C, 66.83; H, 4.29; N, 4.45. Found: C, 66.78; H, 4.10; N, 4.52%. IR (KBr); ν/cm^{-1}): 1433 (N=N), 1099 (Pd bound PPh_3). UV/VIS (UV/VIS (CH_2Cl_2), λ/nm (ε, $dm^3\ mol^{-1}\ cm^{-1}$): 548(9600), 463(7700), 438(6900), 337(7700). ^1H NMR in $CDCl_3$ (ppm): 2.27 (s, 3H, 5′-Me), 6.32–8.37 (aromatic protons).

2.1.2. Synthesis of $[Pd(L^2)Cl]$ (4)

An orange yellow solution of 1-(2′-methoxy-5′-methylphenylazo)naphthalene (HL^2) (0.06 g, 0.22 mmol) in ethanol (20 mL) was added to a brown ethanolic solution (20 mL) of sodium tetrachloropalladate (0.07 g, 0.24 mmol) with stirring. Green precipitate started to appear with the stirring, which was continued for 2 h at room temperature. The green precipitate was collected by filtration and thoroughly washed with water and ethanol respectively. The solid mass was recrystallised from a mixture of dichloromethane and petroleum ether. Finally the cyclopalladate $[Pd(L^2)Cl]$ (**4**) was dried in vacuo (Yield: 0.06 g, 0.144 mmol, 65.4%). *Anal.* Calc. for $C_{18}H_{15}N_2OClPd$: C, 51.80; H, 3.60; N, 6.71. Found: C, 51.42; H, 3.34; N, 6.35%. IR (KBr); ν/cm^{-1}): 1452 (N=N). UV/VIS (CH_2Cl_2), λ/nm(ε, $dm^3\ mol^{-1}cm^{-1}$): 604(7820), 574(7620), 430^{sh}(7260), 410(8030), 388(8340), 308(14200). ^1H NMR in $CDCl_3$ (ppm): 2.39 (s,3H, 5′-Me), 4.18 (s, 3H, 2′-OMe) 7.01–8.59 (aromatic protons).

2.1.3. Synthesis of [Pd(L^2)(Cp)] (5)

Solid thallium cyclopentadienide (TlCp) (0.036 g, 0.134 mmol) was added to a solution of [Pd(L^2)Cl] (**4**) (0.04 g, 0.096 mmol) in benzene (40 mL) and the mixture was stirred for 2 h at room temperature. The colour of the solution became deep reddish violet. The precipitate of thallium chloride was removed by filtration through glass sintered (G-4) funnel. The filtrate was chromatographed on cellulose column and a deep reddish violet band was eluted using benzene as eluant. The removal of the solvent afforded red microcrystals of [Pd(L^2)(Cp)] (**5**). (Yield: 0.026 g, 0.058 mmol, 60.4%). *Anal.* Calc. for $C_{23}H_{20}N_2OPd$: C, 61.82; H, 4.48; N, 6.27. Found: C, 61.68; H, 4.24; N, 6.12%. IR (KBr); ν/cm^{-1}): 1452 (N=N), 771 (Cp). UV/VIS (CH_2Cl_2), λ/nm (ε, dm^3 $mol^{-1}cm^{-1}$): 552(3200), 473(3700), 362(3000), 280 (11700). ^1H NMR in $CDCl_3$ (ppm): 2.4 (s,3H, 5′-Me), 3.9 (s, 3H, 2′-OMe), 5.79 (s, 5H, Cp), 6.90–8.81 (aromatic protons).

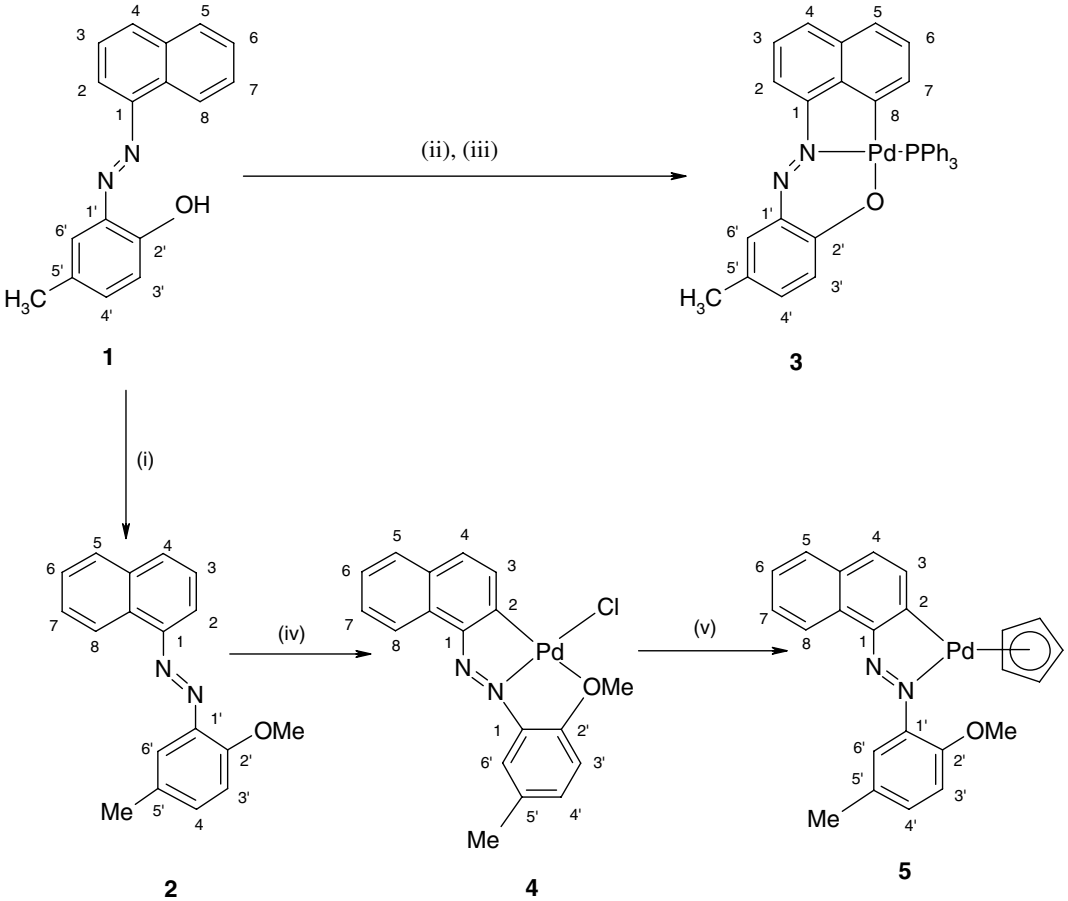
Caution: Thallium compounds and benzene are highly hazardous chemicals. These should be handled with great care.

3. Results and discussion

3.1. Synthesis

Scheme 1 shows the compounds prepared in this work and the numbering of the carbon atoms for the following discussion.

An ethanolic solution of disodium tetrachloropalladate is treated with an equimolar solution of 1-(2′-hydroxy-5′-methylphenylazo) naphthalene (**1**) at room temperature and gradually starts to produce solid precipitate, which readily reacts with PPh_3 and yields soluble pink coloured cyclometallate [PdL^1 (PPh_3)] (**3**). 1-(2′-methoxy- 5′-methylphenylazo)naphthalene (**2**) also undergoes facile cyclopalladation reaction with disodium tetrachloropalladate in ethanol and yields green coloured cyclopalladate [PdL^2Cl] (**4**). Cyclopalladate [PdL^2Cl] (**4**) reacts with thallium(I) cyclopentadienide and yields red violet [PdL^2(Cp)] (**5**), where palladium(II) is bound to carbon by both σ- and π-bonds. The proposed compositions of all the cyclopalladates are consistent with the observed elemental analysis data (Section 2).



Scheme 1. (i) MeI, K_2CO_3, 2-butanone, reflux, 3 h; (ii) $Na_2[PdCl_4]$ (1 equivalent), EtOH, room temperature, 2 h; (iii) PPh_3 (1 equivalent), benzene, 0.5 h; (iv) $Na_2[PdCl_4]$ (1 equivalent), EtOH, room temperature, 2 h; (v) TlCp, benzene, room temperature.

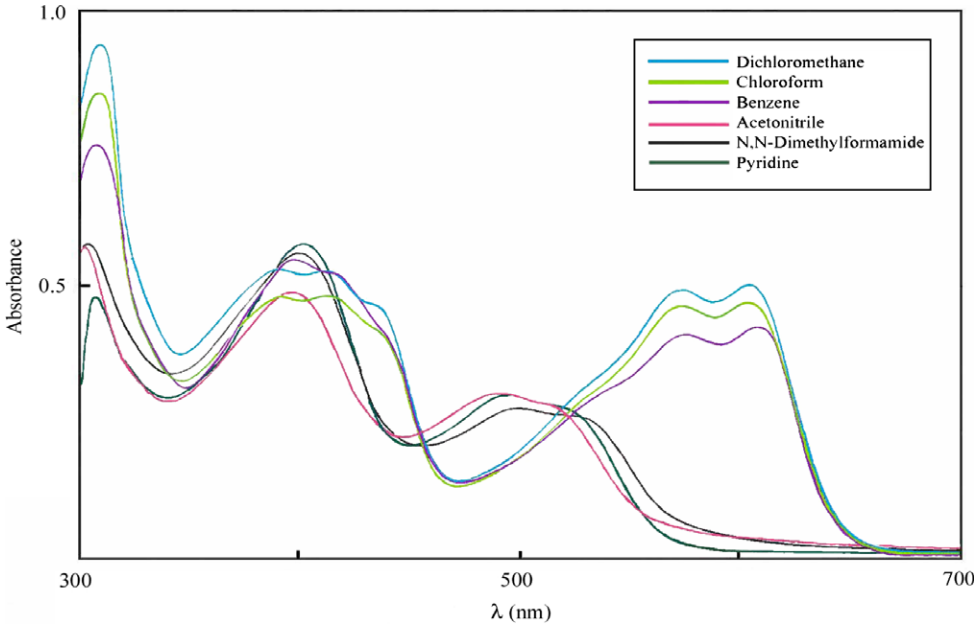


Fig. 1. Electronic spectra of [Pd(L^2)Cl] (**4**) (6.476×10^{-5} M) in different solvents at 298 K.

3.2. Spectral properties

The IR spectra of all the cyclopalladates (**3**–**5**) show absorption in the region of 1430–1450 cm^{-1} which can be assigned to the N=N group [37]. The IR spectrum of [PdL^1(PPh_3)] (**3**) shows absorption at 1099 cm^{-1} due to palladium (II) bound triphenyl phosphine [38]. Cyclopalladate [PdL^3(Cp)] (**5**) displays a strong absorption around 771 cm^{-1} in its IR spectrum, which is characteristic of π-bonded cyclopentadienyl ring to palladium(II) [39,40].

All the cyclopalladates are soluble in common organic solvents. The electronic spectral data of the cyclometallates are given in the experimental section. The electronic spectra of the cyclopalladates show one or more strong absorptions in the visible region. These transitions are assigned to metal to ligand charge transfer (MLCT) [d(Pd) → π (azo)] bands. The bands observed in the high-energy region with high molar extinction co-efficient arise from intraligand charge transfer transition [41].

The compound [PdL^2Cl] (**4**) shows solvent dependent electronic spectra (Fig. 1). In non-coordinating solvents like dichloromethane, benzene and chloroform, it shows a strong absorption above 600 nm, which is similar to the absorption profile of palladium(II) bound terdentate [C2(phenyl), N(azo), O(phenolato)] phenylazophenol [42,43]. This observation suggests that Pd(II)–OCH_3 bond remains intact in non-cordinating solvents and monoanionic L^2 behaves as a terdentate ligand. In coordinating solvents like acetonitrile, dimethylformamide and pyridine, the absorption band of [PdL^2Cl] shifts to 520–530 nm, which is characteristic of cyclopalladated [C2, N] naphthylazobenzene [27]. Excess donor solvents like acetonitrile, dimethylformamide and pyridine open the labile palladium(II)–OCH_3 bond and coordinate to the metal centre of [PdL^2Cl] in the solution (Table 1).

Table 1
Electronic spectral data of cyclopalladate (**4**) in different solvents

Solvent	λ_{max} (ε, $dm^3\ mol^{-1}\ cm^{-1}$)
Dichloromethane	604(7820), 574(7620), 430^{sh}(7260), 410(8030), 388(8340), 308(14200)
Benzene	608(6640), 578(6330), 409^{sh}(8030), 394(8500), 305(11890)
Chloroform	607(7410), 574(7260), 412^{sh}(7110), 390(7100), 307(13280)
Acetonitrile	515(4320), 487(4630), 396(7570), 302(8800)
Dimethylformamide	528(3090), 497(4170), 402(8800), 306(8960)
Pyridine	520(4010), 495(4170), 400(8960), 309(7410)

The ^{1}H NMR spectra of all the cyclopalladates show that the methyl group attached to the phenyl ring (2′-Me) appear as singlet in the range of 2.27–2.40 ppm. In $CDCl_3$ the signal for methoxy group of [PdL^2Cl] (**4**) appears as a singlet at 4.2 ppm whereas the signal for the same group of [PdL^2(Cp)] (**5**) shifts to the higher field at 3.9 ppm, which demonstrates the opening of palladium(II)-OCH_3 bond in [PdL^2(Cp)] (**3**) due to the presence of Cp group. The ^{1}H NMR spectrum of [PdL^2(Cp)] (**5**) also shows a sharp signal at 5.79 ppm due to the five equivalent protons of cyclopentadienyl ring [40,44]. For all the cyclopalladates, the signals for aromatic protons appear as complex pattern in the region 6.3–8.8 ppm with correct integration ratio.

3.3. X-ray structure of [PdL^1(PPh_3)] (3)

Slow diffusion of the solvent from a solution of [PdL^1(PPh_3)] (**3**) in dichloromethane to hexane layer afforded monoclinic pink crystal of the space group $P2_1/c$ and

allowed the establishment of its crystal structure (Fig. 2). Significant crystal data and data collection parameters for $[PdL^1(PPh_3)]$ are listed in Table 2. The selected bond lengths and bond angles for $[PdL^1(PPh_3)]$ are given in Table 3.

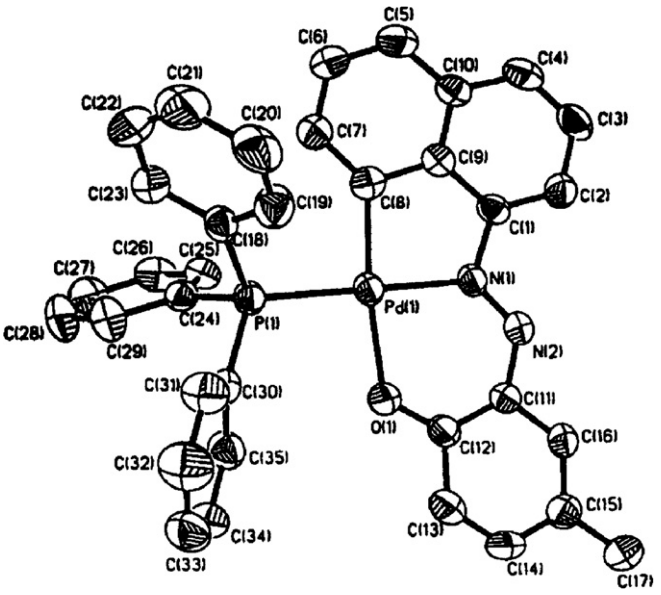


Fig. 2. Molecular structure of $[PdL^1(PPh_3)]$ (**3**).

Table 2
Crystal data and structure refinement for $[PdL^1(PPh_3)]$ (**3**)

Chemical formula	$C_{35}H_{27}N_2OPPd$
Formula weight	629.00
Temperature (K)	293(2)
Wavelength (Å)	0.71073
Crystal system	monoclinic
Space group	$P2_1/c$
Unit cell dimensions	
a (Å)	15.2140(5)
b (Å)	11.6433(3)
c (Å)	17.7950(5)
α (°)	90
β (°)	115.223(10)
γ (°)	90
Volume (Å^3), Z	2851.68(14), 4
Density (calculated) (Mg/m^3)	0.366
Absorption coefficient (mm^{-1})	0.184
$F(000)$	320
Crystal size (mm)	$0.35 \times 0.12 \times 0.12$
*Maximum and minimum transmission	1.000000 and 0.673573
θ range for data collection (°)	1.48 to 23.25
Limiting indices	$-16 \leqslant h \leqslant 16$, $-8 \leqslant k \leqslant 12$, $-19 \leqslant l \leqslant 19$
Reflections collected	11 541
Independent reflections	4084 ($R_{int} = 0.0409$)
Refinement method	full-matrix least-squares on F^2
Data/restraints/parameters	4084/0/469
Goodness-of-fit on F^2	0.994
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0308$, $wR_2 = 0.0605$
R indices (all data)	$R_1 = 0.0498$, $wR_2 = 0.0667$
Largest difference in peak and hole (e A^{-3})	0.289 and −0.467

Table 3
Selected bond lengths [Å] and angles [°] for $[PdL^1(PPh_3)]$ (**3**)

Bond lengths	(Å)	Bond lengths	(Å)
Pd(1)–C(8)	2.015(3)	Pd(1)–N(1)	2.032(3)
Pd(1)–O(1)	2.056(2)	Pd(1)–P(1)	2.2816(9)
P(1)–C(24)	1.820(3)	P(1)–C(18)	1.835(3)
P(1)–C(30)	1.821(3)	O(1)–C(12)	1.299(4)
N(1)–N(2)	1.275(3)	N(1)–C(1)	1.427(4)
N(2)–C(11)	1.374(4)	C(1)–C(2)	1.372(5)
Bond angles	**(°)**	**Bond angles**	**(°)**
C(8)–Pd(1)–N(1)	82.92(13)	C(8)–Pd(1)–O(1)	172.40(12)
N(1)–Pd(1)–O(1)	89.50(10)	C(8)–Pd(1)–P(1)	96.70(10)
N(1)–Pd(1)–P(1)	178.11(8)	O(1)–Pd(1)–P(1)	90.86(7)

1-(2′-hydroxy-5′-methylphenylazo) naphthalene binds palladium(II) center in terdentate fashion through C8 of naphthyl fragment, N1 of diazene function and O1 of phenolato group. Terdentate naphthylazophenol and P1 of phosphine make the tetra coordination environment around palladium(II) essentially planar but deviates from the square planar geometry. The maximum deviation is represented by the 'bite' angle C(8)–Pd(1)–N(1) of 82.92(13)°. This type of deviation is observed for cyclopalladates [45]. The Pd–C8 bond length of 2.015(3) Å is in agreement with reported palladium(II)-C(naphthyl) bond length of 2.014(8) Å [46]. The Pd–N1 bond distance of 2.032(3) Å is longer than Pd–N(azo) bond lengths of 1.970(4) Å [47]. The lengthening of Pd–N bond is due to the presence of strong *trans* influence of triphenylphosphine. Here the Pd-P1 bond distance (2.281 Å) is within the reported range [48]. The C1–N1 bond length 1.427(4) Å of azapalladacycle is longer than C11–N2 1.374(4) Å of diazametallacycle [27]. The shortening of C11–N2 distance of diazapalladacycle is expected due to the presence of enhanced π-electron density than that of azapalladacycle.

4. Conclusions

In this study we have begun to explore how the phenylazonaphthalenes that are modeled by variation of the substituents at 2′-position of the phenyl ring can be employed to achieve the preferential C–H bond activation of the naphthyl ring with palladium(II) compounds. The selection of C(naphthyl)–H bond for activation with disodium tetrachloropalladate is controlled by the nature of oxygen donor (auxiliary donor) present at 2′-position of 1-(phenylazo)naphthalene. The regiospecific activation of C8–H bond of naphthyl group in (**1**) by palladium(II) can be explained in terms of the formation of six-membered metal chelate [N,O] as an initial step, which can then only lead to the palladation at the peri-position of the naphthyl ring. The group of *o*-hydroxyarylazo compounds are well known to form stable six-membered chelates [O, N] with transition metal ions [49–51]. On the other hand disodium tetrachlo-

ropalladate regiospecifically activates C2–H bond of naphthyl group of (**2**), where an alkoxy group is present as auxiliary donor. Here, the diazene function being a stronger donor than alkoxy group towards electrophillic metal ions, preferentially binds palladium(II), followed by palladation at *ortho*-position of the naphthyl ring resulting in the formation of five-membered palladacycle like palladates of other arylazonaphthalenes [25–27]. The formation of Pd ←:OMe bond only occurs in the absence of coordinating solvents.

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Appendix A. Supplementary data

CCDC 277866 contains the supplementary crystallographic data for **1**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.ica.2006.10.009.

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