Synthesis, characterization, crystal structure and catalytic activity of amido azo palladium(II) complex

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Abstract



Introduction

In the last three decades, palladium-catalyzed cross-coupling reactions leading to formation of C–C, C–N, C–O, and C–S bonds have become widely used in laboratory preparation, as well as industrial fine chemical synthesis [1–6]. Most of the works reported an efficient catalytic system for cross-coupling reactions based on the sterically demanding electron-rich phosphines, and its analogs [1, 2, 7–10]. However, palladium phosphines have several disadvantages, including toxicity, sensitivity to air and moisture, and difficult separation from the products [11–13]. To avoid this disadvantage several phosphine free systems have emerged

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as an alternative [14–20]. The unique steric and electronic properties of some ligands as well as their specific coordination mode and their strong binding ability to the formation of highly active and stable metal complexes which are more active catalyst for coupling reactions [21–23]. Still palladium phosphines are an important catalyst for crosscoupling reactions because phosphines control the catalytic cycle to maintain the steric and electronic properties to favor both the oxidative addition of the aryl halide and the reductive elimination of the coupled product [24–26]. The formation of amine complexes depends on the steric and electronic influences of the substituents that are conjugated with the chelate unit. N-alkyl substituents can control substantial steric effects on structure, reactivity and selectivity of the complexes. Optimistic to these points a balance between the steric and electronic properties of the ligand is an important part. Moreover, few reports are available on azo-imine ligands with palladium(II) and their applications as catalyst in cross-coupling reactions [27-31]. Therefore, we are interested in continuing our work focused on the synthesis of new catalysts, which are inexpensive and stable in catalytic reaction conditions. This work originates from our previous observations of ligands 2,2'-diaminoazobenzene and bis-2,2'-(N-alkylamino)azobenzene with palladium and their application as catalyst [28, 29]. The stability of the chloride or phosphine complex depends on the alkyl substituent



at the amino nitrogen of 2,2'-diaminoazobenzene [28, 29]. The present study where the ligand, 2-((2-aminophenyl) diazenyl)-*N*-benzylaniline, contains one alkyl substituent at the amino nitrogen of 2,2'-diaminoazobenzene gives a neutral phosphine complex with the ligand coordinated as a dianion, which is different from our previous observations [28, 29].

Herein, we report the synthesis of new ligand 2-((2-aminophenyl)diazenyl)-*N*-benzylaniline and its palladium complex. Both the ligand and complex were characterized by usual (UV–Vis, IR and ¹H NMR) spectroscopy. The structure of the complex **2** was determined by X-ray crystallography. The Pd(II) complex was investigated as a catalyst in Suzuki cross-coupling and Heck reactions in the presence of air and moisture for a variety of aryl halides.

Experimental

The solvents used in the reactions were of reagent grade (Merck, India) and were purified and dried by general procedure [32]. Palladium chloride, sodium chloride, benzyl chloride and potassium carbonate were purchased from Merck, India. Iodobenzene, Bromobenzene, 1-Iodo-2-nitrobenzene, 1-Iodo-3-nitrobenzene, 1-Iodo-3,5-dimethylbenzene, 4-Iodotoluene, 4-Nitroiodobenzene, 4-Nitrobromobenzene, 4-Bromotoluene, 1-Bromo-2-nitrobenzene, Phenyl boronic acid and Styrene were purchased from Aldrich. Sodium tetrachloropalladate and 2,2'-diaminoazobenzene were prepared following reported procedure [29, 33]. Microanalysis (C, H, N) was performed using a Perkin-Elmer 2400 C, H, N, S/O series II elemental analyzer. Infrared spectra were recorded on a Perkin Elmer BX-1 FT-IR spectrometer with the samples prepared as KBr pellets. Electronic spectra were recorded on a Shimadzu UV-1800 PC spectrophotometer. ¹H NMR spectra were obtained on Bruker 400 NMR spectrometer in CDCl₃ using TMS as the internal standard.

Preparation of ligand 1

A mixture of 2,2'-diaminoazobenzene (0.5 g, 2.36 mmol), benzyl chloride (0.3 g, 2.36 mmol), 5 g of K₂CO₃ in 40 mL dry ethanol was refluxed for 2 h. The orange solid mass that was obtained after evaporation of the solvent, afforded the ligand, 1, which was isolated by thin layer chromatography (R_f =0.6) on silica gel using the eluent petroleum ether–toluene (70/30 v/v). Yield: 50%. Anal. Calc. for C₁₉H₁₈N₄: C, 75.47; H, 6.00; N, 18.53. Found: C, 75.18; H, 6.13; N, 18.35%. UV/VIS (λ_{max} /nm (ε /dm² mol⁻¹), dichloromethane): 480 (25,290), 315 (21,810), 265 (33,520), 230 (45,760). IR (KBr, cm⁻¹): 3382,3271 v(NH, NH₂), 1433 v(N=N). ¹H NMR (400 MHz, CDCl₃): 4.46 (CH₂, s, 2H), 4.70 (NH₂, s, 1H), 5.50 (NH₂, s, 1H), 6.70–6.78 (ArH, m, 4H), 7.11–7.13 (ArH, m, 1H), 7.19–7.23 (ArH, m, 1H), 7.28–7.31 (ArH, m, 1H), 7.36 (ArH, t, 2H), 7.40 (ArH, t, 2H), 7.54 (ArH, d, 1H), 7.72 (ArH, d, 1H), 8.55 (NH, s, 1H).

Preparation of complex 2

A solution of 1 (0.103 g, 0.34 mmol) in 10 mL methanol was added to a solution of Na₂PdCl₄ (0.1 g, 0.34 mmol) in 10 mL methanol. The mixture was stirred for 4 h and then added PPh₃ (0.52 mmol). The dark solid precipitate was separated by filtration and purified by thin layer chromatography ($R_f = 0.3$) on silica gel using the eluent toluene. Yield: 65%. Anal. Calc. for PdC₃₇H₃₁N₄P: C, 66.42; H, 4.67; N, 8.37. Found: C, 66.65; H, 4.42; N, 8.35%. UV/VIS (λ_{max}/nm $(\varepsilon/dm^2 mol^{-1})$, dichloromethane): 585 (3450), 345 (28,750), 230 (65,560). IR (KBr, cm⁻¹): 3346 ν (NH), 1653 ν (C=N), 1484 ν (N=N), 1478, 692 ν (PPh₃). ¹H NMR (400 MHz, CDCl₃): 4.66 (CH₂, s, 2H), 5.21 (NH, d, 1H), 6.31 (ArH, d, 1H), 6.53 (ArH, d, 2H), 6.65 (ArH, t, 1H), 6.71 (ArH, t, 2H), 6.86-6.94 (ArH, m, 2H), 7.01-7.07 (ArH, m, 2H), 7.23 (ArH, t, 1H), 7.30–7.34 (ArH, m, 6H(PPh₃)), 7.43 (ArH, t, 3H(PPh₃)), 7.76–7.70 (ArH, m, 6H(PPh₃)), 8.07(ArH, d, 1H), 8.67 (ArH, d, 1H).

General procedures for the Suzuki reaction

To the catalyst (1.0 mol%) dissolved in 10 mL THF, aryl halide (1.0 mmol), phenyl boronic acid (1.2 mmol), K_2CO_3 (2.0 mmol) were all inserted in a 25 mL round-bottom flask. The mixture was heated at 80 °C under stirring for 3 h. The reaction was monitored using thin layer chromatography (R_f =0.4–0.7) on silica gel. After the completion of the reaction the mixture was evaporated to dryness, water was added and the product was extracted with dichloromethane. The organic layer was washed with brine, dried over Na₂SO₄, filtered, passed through silica column (60–120 mesh), the complex remain trapped. Upon removal of the solvent, solid of pure products were obtained. The yields of the products obtained (based on the aryl halides) from all the reactions were determined after isolation, and characterized by ¹H NMR spectra and melting points with the literature data.

General procedures for the Heck reaction

A similar procedure to that described for the Suzuki reaction was followed to carry out the Heck reaction using aryl halide (1.0 mmol), styrene (1.1 mmol), K_2CO_3 (2.0 mmol) with (1.0 mol%) catalyst and 10 mL of 2-propanol. The reaction mixture was heated to 70 °C under stirring during 4 h. The yields of the products obtained (based on the aryl halides) from all the reactions were determined after isolation, and

characterized by ¹H NMR spectra and melting points with the literature data.

X-ray structure analysis

Single crystal of **2** was grown by diffusion of dichloromethane into petroleum ether at room temperature. Data were collected by ω -scan technique on a Bruker Smart CCD diffractometer with Mo-K_{α} radiation monochromated by graphite crystal. Structure solution was done by direct method with SHELX program [34, 35]. Full matrix least square refinements on F² were performed using SHELX program [34, 35]. All non-hydrogen atoms were refined anisotropically

 Table 1
 X-ray data of compound 2

Formula	$PdC_{37}H_{31}N_4P$
Mol. wt	669.05
Crystal system	Monoclinic
Space group	P2 _{1/c}
<i>A</i> (Å)	15.4207(6)
B (Å)	21.7425(10)
$C(\text{\AA})$	9.0226(3)
α (°)	90
β (°)	96.728(2)
γ (°)	90
Wavelength (Å)	0.71073
Volume (Å)	3004.3(2)
F(000)	1368
Crystal dimensions (mm)	0.11×0.14×0.26
Ζ	4
Temperature (K)	296
Density/mg/m ⁻³	1.479
Abs. coeff (mm^{-1})	0.705
Theta Min–Max (°)	1.3, 28.5
Reflections measured	27,136
Unique reflections (R_{int})	5738, 0.054
No. of reflections used $[I > 2\sigma(I)]$	3830
Final R_1 (wR_2) (all)	0.0535, 0.0732
Final R_1 , (wR_2) $[I > 2\sigma(I)]$	0.0423, 0.0754
Goodness of fit	1.01

Scheme 1 Synthesis of ligand and Palladium(II) complex



CCDC 1958999 contains the supplementary crystallographic data for compound 2. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Results and discussion

Synthesis

The new ligand 2-((2-aminophenyl)diazenyl)-*N*-benzylaniline, **1** was prepared by substituting one of the amino proton of one amino group of 2,2'-diaminoazobenzene with benzyl chloride in the presence of potassium carbonate in refluxing dry ethanol (Scheme 1). The ligand was isolated as orangered liquid after chromatographic purification.

The reaction of ligand 1 with Na₂[PdCl₄] in methanol at room temperature in the presence of PPh₃ afforded a blue colored complex of composition 2 (Scheme 1). In the complex 2, the ligand binds in a tridentate (N,N,N) fashion through amido nitrogen, azo nitrogen and amino nitrogen atoms forming both six membered and five membered chelate rings. The fourth coordination site of the palladium in the complexes is occupied by a PPh₃ ligand. Reactions of 2,2'-diaminoazobenzene with Pd(II) gives ionic phosphine complex [29], whereas the presence of alkyl substituents at both the amino nitrogen, i.e., bis-2,2'-(N-alkylamino) azobenzene gives phosphine free neutral complex [28]. The presence of only one benzyl substituent in the ligand, 2-((2-aminophenyl)diazenyl)-N-benzylaniline, is expected to stabilize the dianionic neutral phosphine complex by changing electronic and steric properties, which is further supported by two other unsuccessful reactions of 2-((2-aminophenyl)diazenyl)-N-alkylaniline, where alkyl substituents are N-Me or Et.



Characterization

Both the ligand and complex are soluble in common organic solvents and furnished orange-red and blue color. UV-Vis spectra of the ligand and complex were recorded in dichloromethane solutions and these spectra are given in Fig. 1. The absorption spectrum of the free ligand and its complex consists of four and three absorption peaks in the range 230-480 nm and 230-585 nm respectively. First three (230, 265 and 315 nm) high energy bands of the ligand can be assigned to $\pi - \pi^*$ transitions of the phenyl rings and the last one (480 nm) characteristic of $n-\pi^*$ transitions of azo (-N=N-) function [28-31, 36, 37]. Upon coordination of the ligand, the π - π * transitions of the phenyl rings in the complex were shifted to different wavelengths (230 and 345 nm), which indicated the coordination of the palladium. A characteristic broad low intense absorption peak was observed at 585 nm in the electronic spectrum of the complex and can be assigned to a mixed metal ligand charge transfer transition [28-31, 36, 37].

The IR spectrum of the ligand in solid KBr support the presence of $\nu_{\rm NH2}$ and $\nu_{\rm N-H}$ which appeared as a twin band at 3382 and 3271 cm⁻¹, whereas in the complex only one $\nu_{\rm NH}$ appeared as singlet band at 3346 cm⁻¹ indicating the dissociation of two amino protons on complexation [28–31]. The $\nu_{\rm N=N}$ band of the ligand 1484 cm⁻¹ shifted to lower frequency in the complex 1433 cm⁻¹, consistent with coordination of the azo nitrogen [28–31]. Two new bands near 1478 cm⁻¹ and 692 cm⁻¹ of complex **2** are assigned for the coordinated triphenylphosphine [29, 38]. A new band at 1653 cm⁻¹ in the IR spectrum of the complex unlike the ligand has been attributed to imine formation [28–31].

Both the ligand and complex shows highly resolved ¹H-NMR spectra in CDCl₃, which are in complete agreement with the structure of the ligand and complex. A sharp singlet at 8.55 ppm is assigned for an NH proton of the ligand which is absent in the complex. The NH₂ resonance of the ligand appeared as two singlets at 5.50 ppm and 4.70 ppm, probably



Fig. 1 UV–Vis spectra of 1 (dashed line) and 2(solid line) in dichloromethane

resulting from the unsymmetric nature of the protons due to hydrogen bonding. In the complex only one NH resonance appeared at 5.21 ppm as a doublet indicating the dissociation of amino proton on complexation [28]. A sharp singlet at 4.46 ppm is assigned for CH₂ protons of the ligand which is shifted to 4.66 in the complex. The aromatic protons (thirteen) appeared in the range of 6.70–7.73 ppm, and appeared as two doublets, three set of triplets, and three set of multiplets, which are matched well with the composition of the ligand in solution. In the complex the aromatic protons (except PPh₃) appeared in the range 6.31–8.67 ppm for thirteen protons as separate doublets, triplets and multiplets.

The structure of **2** is determined by single crystal X-ray diffraction method and relevant crystallographic parameters for data collection and crystal structure solution and refinement details are given in Table 1 and selected bond lengths and bond angles are given in Tables 2. The completeness was low (75.4%), but 5738 unique reflections were collected, enough to refine 388 structural parameters. The Goodness of Fit covered to 1.012 [$I > 2\sigma(I)$], suggesting that the data refinement was reasonable. The crystal structure of 2 is shown in Fig. 2. Complex 2 is monoclinic and crystallized in the space group $P2_{1/c}$ with Z=4. The palladium atom is located in a distorted square planar environment (mean deviation 0.08 Å), surrounded by the three nitrogen atoms of the ligand and the fourth coordination site of the palladium is occupied by a P atom of the PPh₃ ligand. Ligand **1** is coordinated with palladium in a dianionic tridentate (N,N,N)fashion to construct two chelate rings (six membered and five membered) with amino nitrogen (N4) and azo nitrogen (N3) and amido nitrogen (N1) atoms.

The geometry about Pd(II) is near square planar with N(1)-Pd-N(4), N(3)-Pd-Cl(1) bond angles 170.61(11) and 178.90(7). The Pd-N(1), Pd-N(4), Pd-N(3) and Pd-P bond lengths 1.968(3), 2.016(3), 2.009(3), 2.2862(9) Å are comparable with similar Pd(II) complexes [28, 29, 38-41]. The C(1)-N(1) and C(6)-N(2) lengths (~1.35 Å) is shorter than the C-N single bond C(7)-N(3) 1.41 Å in the same molecule is close to the imine distance [28, 29] and C(12)-N(4) distance 1.343(4) Å is comparably shorter due to dissociation of amino proton upon complexation. The bond distances within the ligand frame adjacent to six membered chelate rings (C(2)–C(3) and C(4)–C(5) (~1.35 Å)) are appreciably shorter than that of other four bonds (C(1)-C(2), C(3)-C(4),C(5)-C(6) and C(6)-C(1) (~1.41) Å), which indicates the presence of extensive delocalization of the negative charge along the ligand backbone [28-31].

The Suzuki and Heck coupling reactions

The palladium-catalyzed Suzuki and Heck cross-coupling reactions are a powerful strategy to form carbon–carbon bonds, and have been utilized in synthesizing biaryl

Table 2	Selected bond distances	(A)	and angles (°)	of compound 2

Distances			
Pd–P	2.2862(10)	P-C20	1.827(3)
Pd-N1	1.967(3)	C1–C6	1.417(5)
Pd-N3	2.009(3)	C1–C2	1.430(4)
Pd-N4	2.016(3)	C2–C3	1.352(6)
N1C1	1.329(4)	C3–C4	1.391(6)
N2-N3	1.290(4)	C4–C5	1.355(5)
N2-C6	1.372(4)	C5–C6	1.416(5)
N3-C7	1.411(4)	C13–C14	1.515(4)
N4-C12	1.343(4)	N4C13	1.457(4)
Angles			
P–Pd–N1	91.50(9)	Pd-N1-C1	128.0(2)
P-Pd-N3	178.90(7)	N3-N2-C6	121.4(3)
P-Pd-N4	97.88(8)	Pd-N3-N2	131.0(2)
N1-Pd-N3	88.65(11)	Pd-N3-C7	112.8(2)
N1-Pd-N4	170.61(11)	N2-N3-C7	116.2(3)
N3-Pd-N4	81.99(11)	Pd-N4-C12	112.5(2)
Pd-N4-C13	130.4(2)	C12-N4-C13	116.9(3)



Fig.2 Molecular structure of 2 (40% probability ellipsoids) with atom numbering scheme. Hydrogen atoms are omitted for clarity except at the N1 atom

compounds which are utilized in various fields such as pharmacology, medicine, and agriculture [1, 2, 7–9]. In most cases, phosphine ligands, used in combination with Pd precatalyst, have demonstrated efficient catalytic activity. In continuation of our effort toward the sustainable chemistry employing metal catalysis under mild reaction conditions, we investigated efficient, less expensive, easily accessible and air-stable azo-imine based palladium(II)-catalysts for C–C coupling reactions [29–31].

The complex 2 was screened for the catalytic Suzuki coupling reactions (Eq. 1). In this study we report the coupling reactions of several aromatic halides under atmospheric pressure with changing three parameters base, solvent and reaction temperature. Before detailed study on the catalytic reactions, we first optimized the reaction conditions by choosing a model substrate (iodobenzene with phenyl boronic acid). The important three factors for the reactions, base (NEt₃ and K₂CO₃), temperature (25, 40 and 65/80 °C) and solvents (THF, MeOH and CH₃CN) were compared for the purpose of attaining the optimum conditions. The progress of the reaction was monitored by TLC. Among the preliminary studies, K₂CO₃ was found to be best base and the THF was found to be appropriate solvent at 80 °C (Table S1). After optimization of the reaction conditions, the reactions of several aryl halides (iodide and bromide) with different substituent in the aromatic ring were also examined and moderate to good yields were obtained using fixed amount of catalysts (1.0 mol%). The results obtained are given in Table 3. All the reactions were carried out in presence of air and moisture. At the completion of the reaction the catalyst was isolated and reused for the next cycle. The reusability of the catalyst was checked up to three times for a reaction without sufficient loss of its catalytic activity where all other conditions remained intact.

Table 3 Suzuki cross-coupling reaction of various aryl halides with phenyl boronic acid catalyzed by $\mathbf{2}$



$$R \xrightarrow{R} X + A \xrightarrow{R} B(OH)_2 \xrightarrow{Reflux} R \xrightarrow{R} A \xrightarrow{R}$$

In the similar way, the complex **2** was tested as catalyst in another important carbon–carbon bond forming reaction in synthetic organic chemistry, known as the Heck reaction (Eq. 2). Before a detailed study on the catalytic reactions was initiated, we first optimized the reaction conditions by choosing a model substrate, iodobenzene with styrene, keeping other parameters (base, solvent and temperature) as mentioned in Suzuki reaction. After optimization, K_2CO_3 was found to be best base and 2-propanol was found to be an appropriate solvent at 80 °C (Table S2). The reactions of several aryl halides (iodide and bromide) with different substituents in the aromatic ring were also examined and Table 4 Heck reaction of various aryl halides with styrene catalyzed by 2a

Aryl halide	Styrene derivative	Isolated Yield
		92
		86
		88
Br	$\bigcirc - \bigcirc$	76
- Br		70
Br NO ₂		74

moderate to good yields (Table 4) were obtained using fixed amount of catalysts (1.0 mol%). All the reactions were carried out in presence of air and moisture. Similarly as mentioned in Suzuki coupling reactions, at the end of the reaction the catalyst was isolated and reused for the next cycle. The reusability of the catalyst was checked up to three times for a reaction without sufficient loss of its catalytic activity where all other conditions remained intact.



Conclusion

In summary, a tridentate azo ligand and its palladium(II) complex have been synthesized and characterized. The effect of steric and electronic influences on the chelating tendency of 2-((2-aminophenyl)diazenyl)-*N*-benzylaniline with Palladium(II) ion has been investigated using *N*-substituted 2-((2-aminophenyl)diazenyl)-*N*-alkylaniline. The easily accessible and air-stable palladium(II) azoimine catalyst is highly active and efficient catalyst for the formation of unsymmetrical biaryls diversities with high yields under comparatively mild experimental conditions. The effects of bases, temperature and solvents on the model reaction are also screened. The catalytic reactions are simple and efficient at low catalyst loading. The complex is stable under the condition of Suzuki and Heck reactions, signifying the catalytic activity of the molecular complex itself.

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