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Syntheses, characterisation and structure of new diazoketiminato chelates of palladium(II) incorporating a tridentate (*N*,*N*,*N*) azo ligand

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Abstract

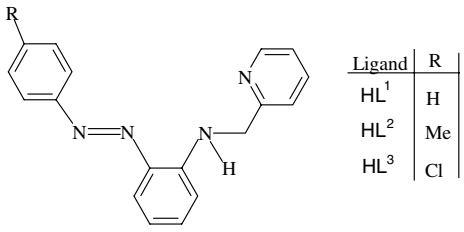
The 1*N*-(2-pyridyl-2-methyl)-2-arylazo aniline, HL [where HL = ArN=NC$_6$H$_4$N(H)(CH$_2$C$_5$H$_4$N); Ar=C$_6$H$_5$ (for HL^1) or *p*-MeC$_6$H$_4$ (for HL^2) or *p*-ClC$_6$H$_4$ (for HL^3)], ligands were prepared by treating the appropriate 2-(arylazo) aniline with 2-chloromethyl pyridine. Reactions of Na$_2$PdCl$_4$ with HL afforded (L)PdCl complexes. HL binds palladium(II) in a tridentate fashion (*N*,*N*,*N*) forming a new diazoketiminato chelate, dissociating the amino proton. The X-ray structure of (L^1)PdCl was determined.

Keywords: 1*N*-(2-pyridyl-2-methyl)-2-arylazo aniline; Sodium tetrachloropalladate; Diazoketiminato species

1. Introduction

The π-acidity and metal binding ability of the azo nitrogen have drawn interest to the exploration of the chemistry of transition metal complexes incorporating azo ligands. Notable examples of these ligands are arylazobenzene [1], arylazooxime [2], arylazophenol [3], arylazopyridine [4], arylazoimidazole [5], arylazoaniline [6] and related ligands. The coordination chemistry of transition metals with azo ligands is being studied due to the observation of several interesting properties. Facile metal–carbon bond formation and the subsequent reactions of some orthometallated azobenzene and related molecules demonstrated their importance in C–H bond activation [3,7,8]. Fascinating electron transfer [3–5,9] and photophysical [10] behaviour of delocalised transition metal chelates of bi and tridentate azo ligands have drawn attention during the recent years. Such remarkable properties were attributed to the low-lying ligand π* orbitals [6].

Herein we describe the synthesis of some new azoamine ligands, 1*N*-(2-pyridyl-2-methyl)-2-arylazo anilines, HL (**1**) and reactions of these new ligands with Na$_2$PdCl$_4$. The coordination mode of HL toward palladium (II) has been compared critically with that of other 1*N*-alkyl-2-arylazo aniline ligands, HL^a (**2**), reported earlier [7]. Formation of the diazoketiminato chelate, in the case of HL, rather than orthometallation has been rationalised on the basis of product formation and amino proton dissociation.



Ligand	R
HL^1	H
HL^2	Me
HL^3	Cl

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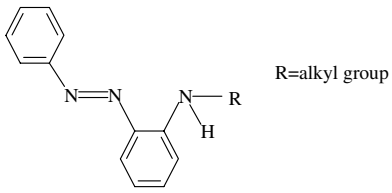


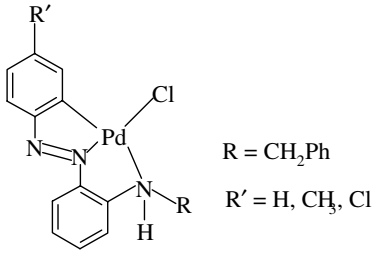
2 (HL[a])

2. Results and discussion

2.1. Syntheses

The 1*N*-(2-pyridyl-2-methyl)-2-arylazo aniline, HL (**1**), ligands were prepared by refluxing 2-(arylazo) aniline with 2-chloromethyl pyridine in acetonitrile in the presence of potassium carbonate and a catalytic amount of potassium iodide (Scheme 1). The ligands were isolated as orange solids.

The reactions of the new HL ligands with Na_2PdCl_4 in methanol afforded pink complexes of palladium (II) of the composition (L)PdCl (**3**) incorporating a new tridentate (*N*,*N*,*N*) ligand (Scheme 1). The deprotonated secondary amino and the azo nitrogens bind the metal, forming a six membered diazoketiminato chelate, while the other 1*N*-alkyl-2-arylazo aniline, HL[a] (**2**) [7] produced the orthopalladated species (L[a])PdCl (**4**) upon treatment with Na_2PdCl_4. Formation of a six membered azoimine chelate in the case of HL has been attributed to the delocalisation of the negative charge within the ligand backbone.



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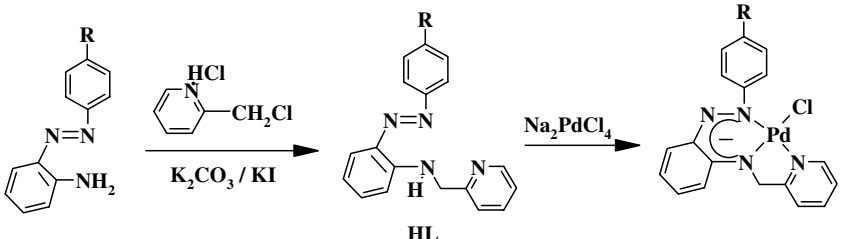
The general mechanism of the formation of orthopalladated azobenzene was described to proceed via initial coordination of azo nitrogen followed by electrophilic substitution at the pendant aryl ring [11]. This mechanism was plausible in the cases of HL[a] since these ligands also underwent facile orthopalladation upon treatment with Na_2PdCl_4 (Scheme 2). Whereas reaction of HL with Na_2PdCl_4 did not afford the orthopalladated product and the reaction pathway has been proposed as shown in Scheme 3. Coordination of the amino nitrogen upon concomitant dissociation of its proton was assumed to occur as a result of prior binding of the pyridyl nitrogen to Pd(II). These preceding coordinations of pyridyl and amido nitrogens enabled the ligand to form the diazoketiminato chelate upon subsequent binding through the azo nitrogen and delocalisation of negative charge, which grows upon amino proton dissociation.

2.2. Characterisation

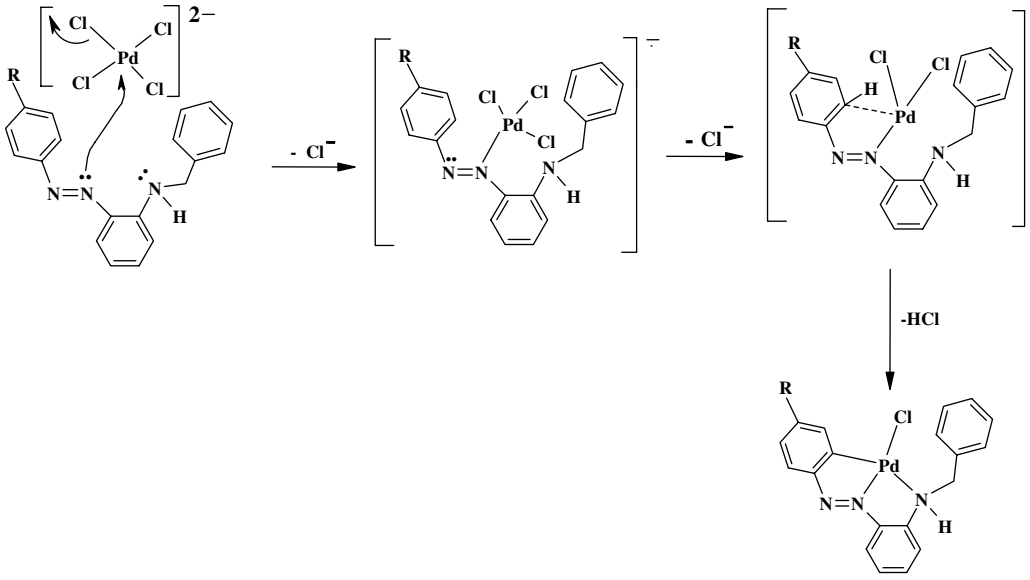
All the ligands displayed characteristic UV–Vis spectra with an absorption near 325 nm for the n–π^* transition of azo compounds [6]. The (L)PdCl complexes exhibited a characteristic low energy absorption near 560 nm, which was assigned to a MLCT transition. Representative UV–Vis spectra of the ligand HL[3]and (L[3])PdCl are shown in Fig. 1. Relevant data are collected in Section 3.

The IR spectra of HL display a sharp singlet near 3189 cm^{-1} for ν_{N-H} which was absent in spectra of (L)PdCl, indicating dissociation of the amino proton on complexation. The $\nu_{N=N}$ band of the ligands ($\sim$1510 cm^{-1}) shifts to lower frequency after formation of the Pd(II) chelates (1350–1400 cm^{-1}), consistent with coordination of the azo nitrogen [6]. The ν_{Pd-Cl} of the palladium complexes appear in the range 290–333 cm^{-1} [7]. Relevant data are included in Section 3.

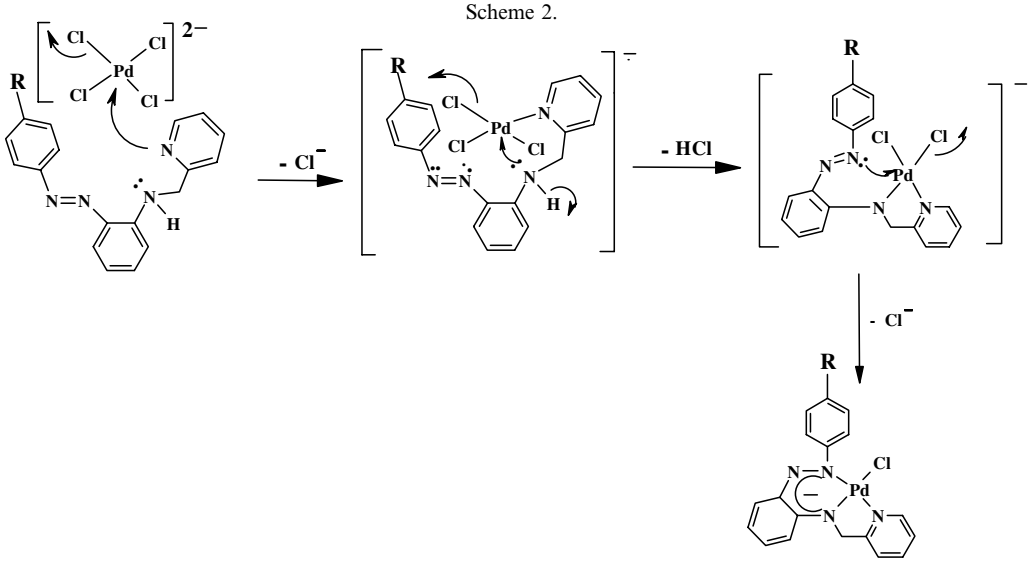
The ^1H NMR spectra of the ligands and the corresponding palladium complexes are consistent with the formulae and structures. Although the amino proton of HL appears as a broad signal, due to the vicinal coupling of the methylene protons its splitting into a broadened triplet can be observed. On the other hand, the methylene proton resonance also splits as a doublet due to vicinal coupling of the N–H proton. In contrast, the ^1H NMR spectra of (L)PdCl do not display the N–H resonance and the methylene protons appear as a singlet (the vicinal coupling of N–H is absent), signifying the dissociation of the amino proton upon complexation. The methylene proton reso-



Scheme 1.



Scheme 2.



Scheme 3.

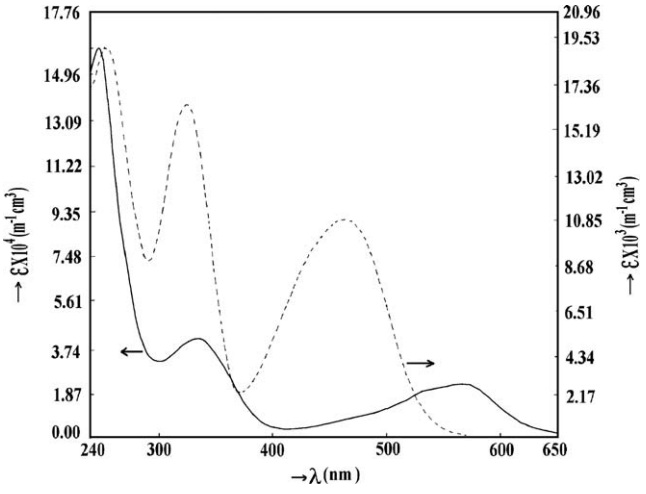


Fig. 1. UV–Vis spectra of HL^3 (---) and $(L^3)PdCl$ (—). The arrows indicate scales of the corresponding spectrum.

nance of (L)PdCl (~δ 5.24) is shifted downfield compared to that of the HL ligands (~δ 4.68). The ^{1}H NMR data are given in Section 3.

2.3. X-ray structure

Suitable crystals of $(L^1)PdCl$ were grown by slow diffusion of hexane into a dichloromethane solution. The X-ray structure of the complex was determined and the perspective view of the $(L^1)PdCl$ molecule, along with the atom numbering scheme, is shown in Fig. 2. Selected bond distances and angles are collected in Table 1. The geometry about palladium is distorted square planar, where the mono anionic deprotonated ligand $(L^1)^-$ binds in a tridentate (*N*,*N*,*N*) fashion. A chloride ligand satisfies the tetracoordination. Three chelating nitrogens are: azo (*N*(azo)), pyridyl (*N*(py)) and amino (*N*(am)). The Pd–*N*(azo), Pd–*N*(py) and Pd–Cl lengths (1.988(4) Å, 2.037(5) Å and

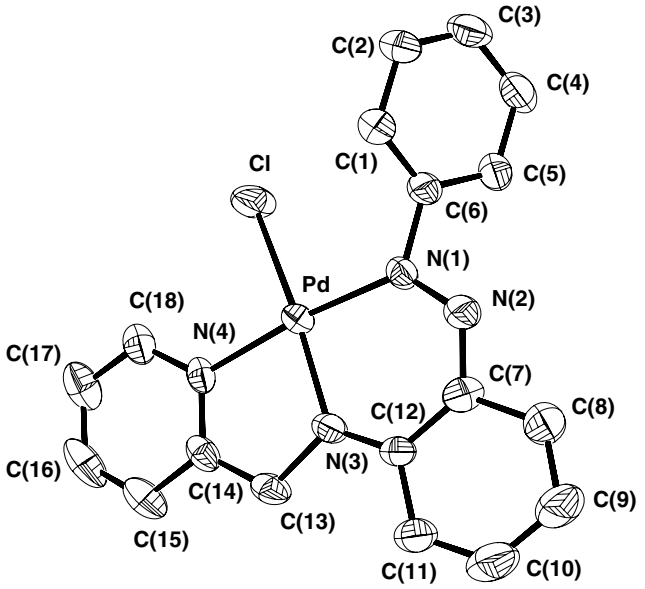


Fig. 2. Perspective view of (L^1)PdCl with the atom numbering scheme. Hydrogen atoms are omitted for clarity.

Table 1
Selected bond distances (Å) and angles (°) for (L^1)PdCl

Pd–N(4)	2.037(5)	N(3)–C(12)	1.330(9)
Pd–N(1)	1.988(4)	C(7)–C(8)	1.429(9)
Pd–N(3)	1.968(5)	C(8)–C(9)	1.349(10)
Pd–Cl	2.330(2)	C(9)–C(10)	1.385(13)
N(1)–C(6)	1.454(8)	C(10)–C(11)	1.368(12)
N(1)–N(2)	1.284(7)	C(11)–C(12)	1.419(10)
N(2)–C(7)	1.346(8)	N(3)–C(13)	1.466(8)
C(7)–C(12)	1.421(1)	C(13)–C(14)	1.454(10)
C(7)–C(8)	1.344(2)	C(14)–N(4)	1.350(9)
N(4)–Pd–N(3)	82.3(2)	N(1)–N(2)–C(7)	124.6(5)
N(1)–Pd–N(4)	173.7(2)	C(13)–C(14)–N(4)	118.0(6)
N(1)–Pd–Cl	94.8(2)	C(14)–N(4)–Pd	113.4(4)
N(3)–Pd–Cl	172.2(3)	Pd–N(1)–N(2)	126.7(4)
C(6)–N(1)–Pd	123.9(4)	C(7)–C(12)–N(3)	121.5(6)
C(13)–N(3)–Pd	114.6(4)	C(12)–N(3)–Pd	126.6(5)

2.330(2) Å, respectively) are within the normal range [9a]. The geometry about palladium is planar (mean deviation 0.042 Å) justifying the oxidation state of Pd(II) and the uninegative ligand since the complex is a non-electrolyte.

The delocalisation of the negative charge within the ligand backbone causes the bond lengths to be suitably altered [6]. The C(12)–N(3) length (1.330(9) Å) is shorter compared to the C–N single bond (N(1)–C(6), 1.454(8) Å) in the same molecule and is similar to the imine (C=N) distance [6]. Also the effect of imine formation due to delocalisation has been reflected in the adjacent phenyl ring (C(7), C(8), C(9), C(10), C(11) and C(12)) which is distorted with two short (~1.35 Å) and four long (~1.4 Å) bonds. Thus the formation of an azoimine chelate could be inferred from the X-ray studies [6] and the structural formula of (L^1)PdCl has been drawn accordingly in **3** of Scheme 1 (vide supra). All the non-hydrogen atoms of (L^1)PdCl, excluding the pendant phenyl ring (C(1)–C(6)), make a good plane (mean deviation 0.114 Å).

3. Experimental

3.1. Materials

The solvents used in the reactions were of reagent grade, obtained from E. Merck, Kolkata, India and purified and dried by reported procedures [6]. 2-(Arylazo) anilines were prepared according to the reported procedure [6]. Palladium chloride and potassium carbonate were purchased from E. Merck, Kolkata, India. 2-(Chloromethyl) pyridine hydrochloride was purchased from Lancaster, England. Disodium tetrachloropalladate was prepared by a reported procedure [6a].

3.2. Syntheses of the ligands

All the ligands, HL^1, HL^2 and HL^3, were prepared following similar procedures. A representative procedure for HL^1 is given below.

3.2.1. HL^1

A mixture of 2-(phenylazo) aniline (0.3 g, 1.52 mmol), 2-chloromethyl pyridine hydrochloride (0.25 g, 1.52 mmol), 1 g of K_2CO_3 and a catalytic amount of KI (0.01 g, 0.06 mmol) in 30 cm^3 dry acetonitrile was refluxed for 3 h. The orange solid mass that was obtained after evaporation of the solvent, afforded the ligand, HL^1, which was isolated by column chromatography on silica gel (60–120 mesh) using the eluent petroleum ether–benzene (3/1 v/v). Upon evaporation of the solvent after chromatography, the orange-red solid of pure HL^1 was obtained. Yield: 0.262 g, 60%. *Anal.* Calc. for $C_{18}H_{16}N_4$, HL^1: C, 75.00; H, 5.20; N, 19.44. Found: C, 74.42; H, 5.35; N, 19.35%. UV–Vis (λ_{max}/nm (ϵ/M^{-1} cm^{-1}) in dichloromethane): 465 (1620), 420 (9430), 325 (10 570). IR (KBr pellets, cm^{-1}): ν_{N-H} 3189, $\nu_{N=N}$ 1512. ^1H NMR ($CDCl_3$): $\delta = 9.50$ (b, NH); 8.66 (d, 1H, $J = 6.0$); 7.90 (d, 3H, $J = 9.0$); 7.66 (t, 1H, $J = 8.5$); 7.49 (t, 2H, $J = 7.5$); 7.41 (d, 1H, $J = 7.2$); 7.36 (t, 1H, $J = 6.0$); 7.28 –7.20 (m, 2H); 6.81 (t, 1H, $J = 7.2$); 6.73 (d, 1H, $J = 8.4$); 4.69 (d, 2H, $J = 4.5$).

3.2.2. HL^2 and HL^3

Ligands HL^2 and HL^3 were prepared using 2-(*p*-tolylazo) aniline and 2-(*p*-chlorophenylazo) aniline in place of 2-(phenylazo) aniline, respectively. Yield: HL^2, 62% and HL^3, 65%.

Anal. Calc. for $C_{19}H_{18}N_4$, HL^2: C, 75.50; H, 5.96; N, 18.54. Found: C, 75.42; H, 6.00; N 18.60%. UV–Vis (λ_{max}/nm (ϵ/M^{-1} cm^{-1}) in dichloromethane): 451 (37 287), 419 (32 681), 321 (61 303). IR (KBr pellets, cm^{-1}): ν_{N-H} 3196, $\nu_{N=N}$ 1512. ^1H NMR ($CDCl_3$): $\delta = 9.37$ (b, NH); 8.65 (d, 1H, $J = 5.0$); 7.86 (d, 1H, $J = 8.0$); 7.79 (d, 1H, $J = 8.5$); 7.64 (t, 1H, $J = 7.5$); 7.35 (d, 1H, $J = 10.0$); 7.29 (d, 2H, $J = 8.0$); 7.24–7.19 (m, 3H); 6.79 (t, 1H, $J = 7.5$); 6.73 (d, 1H, $J = 8.5$); 4.68 (d, 2H, $J = 5.5$); 2.42 (s, 3H).

Anal. Calc. for $C_{18}H_{15}N_4Cl$, HL^3: C, 66.97; H, 4.65; N, 17.37. Found: C, 66.50; H, 4.70; N, 17.25%. UV–Vis (λ_{max}/nm (ϵ/M^{-1} cm^{-1}) in dichloromethane): 461 (10854), 426 (8483), 324 (17123), 253 (19435). IR (KBr pellets, cm^{-1}): ν_{N-H} 3196, $\nu_{N=N}$ 1511. ^1H NMR ($CDCl_3$): $\delta = 9.55$ (b, NH); 8.66 (d, 1H, $J = 4.0$); 7.88 (d, 1H, $J = 8.0$); 7.84 (d, 2H, $J = 7.0$); 7.66 (t, 1H, $J = 7.5$); 7.46 (d, 2H, $J = 9.0$); 7.33 (d, 1H, $J = 7.5$); 7.25 (m, 1H); 7.22 (t, 1H, $J = 6.5$); 6.83 (t, 1H, $J = 8.0$); 6.75 (d, 1H, $J = 8.5$); 4.68 (d, 2H, $J = 5.0$).

3.3. Syntheses of the complexes

All the complexes, $(L^1)PdCl$, $(L^2)PdCl$ and $(L^3)PdCl$ were prepared following similar procedures. A representative procedure for $(L^1)PdCl$, is given below.

3.3.1. $(L^1)PdCl$

A solution of HL^1 (0.150 g, 0.52 mmol) in 10 cm^3 methanol was added to a solution of Na_2PdCl_4 (0.153 g, 0.52 mmol) in 5 cm^3 methanol. The mixture was stirred for 5 h. The dark solid precipitate was separated by filtration and purified by column chromatography using silica gel (60–120 mesh). The eluent was benzene–acetonitrile (19/1 v/v) mixed solvent. Upon evaporation of the solvent, a violet solid of pure $(L^1)PdCl$ was obtained. Yield: 0.122 g, 55%. *Anal.* Calc. for $C_{18}H_{15}N_4PdCl$, $(L^1)PdCl$: C, 50.32; H, 3.49; N, 13.04. Found: C, 50.35; H, 3.50; N, 13.00%. UV–Vis (λ_{max}/nm (ϵ/M^{-1} cm^{-1}) in dichloromethane): 565 (14660), 525 (3230), 325 (31870). IR (KBr pellets, cm^{-1}): $\nu_{N=N}$ 1357, ν_{Pd-Cl} 333. ^1H NMR ($CDCl_3$): $\delta = 9.39$ (d, 1H, $J = 6.0$); 7.86 (t, 2H, $J = 7.2$); 7.52 (d, 1H, $J = 7.8$); 7.45–7.30 (m, 7H); 7.02 (d, 1H, $J = 9.0$); 6.68 (t, 1H, $J = 7.8$); 5.24 (s, 2H).

3.3.2. $(L^2)PdCl$ and $(L^3)PdCl$

Complexes $(L^2)PdCl$ and $(L^3)PdCl$ were prepared using the ligands HL^2 and HL^3 in place of HL^1, respectively. Yield: $(L^2)PdCl$, 55% and $(L^3)PdCl$, 55%.

Anal. Calc. for $C_{19}H_{17}N_4PdCl$, $(L^2)PdCl$: C, 51.46; H, 3.83; N, 12.64. Found: C, 51.40; H, 3.90; N, 12.70%. UV–Vis (λ_{max}/nm (ϵ/M^{-1} cm^{-1}) in dichloromethane): 563 (25711), 529 (22733), 460 (19844), 327 (37666). IR (KBr pellets, cm^{-1}): $\nu_{N=N}$ 1354, ν_{Pd-Cl} 332. ^1H NMR ($CDCl_3$): $\delta = 9.40$ (d, 1H, $J = 5.5$); 7.86 (m, 2H); 7.53 (d, 1H, $J = 8.0$); 7.43 (t, 1H, $J = 7.0$); 7.35–7.31 (m, 3H); 7.18 (d, 2H, $J = 8.0$); 7.01 (d, 1H, $J = 8.5$); 6.67 (t, 1H, $J = 7.5$); 5.22 (s, 2H); 2.39 (s, 3H).

Anal. Calc. for $C_{18}H_{14}N_4PdCl_2$, $(L^3)PdCl$: C, 46.60; H, 3.02; N, 12.08. Found: C, 46.55; H, 3.00; N, 12.10%. UV–Vis (λ_{max}/nm (ϵ/M^{-1} cm^{-1}) in dichloromethane): 566 (21488), 534 (18813), 334 (40348), 247 (159395). IR (KBr pellets, cm^{-1}): $\nu_{N=N}$ 1357, ν_{Pd-Cl} 333. ^1H NMR ($CDCl_3$): $\delta = 9.35$ (d, 1H, $J = 5.5$); 7.86 (t, 1H, $J = 7.5$); 7.79 (d, 1H, $J = 8.5$); 7.53 (d, 1H, $J = 7.5$); 7.44–7.32 (m, 6H); 7.02 (d, 1H, $J = 9.0$); 6.68 (t, 1H, $J = 7.5$), 5.23 (s, 2H).

Table 2
Crystallographic data for $(L^1)PdCl$

Chemical formula	$C_{18}H_{15}ClN_4Pd$
Formula weight	429.21
Space group	*Pna*21 cab (no. 33)
Crystal system	orthorhombic
a (Å)	6.9300(11)
b (Å)	13.3390(13)
c (Å)	17.738(2)
λ (Å)	0.71073
V (Å^3)	1639.7(4)
Z	4
Temperature (K)	293
ρ_{calc} (Mg/m^3)	1.739
μ (mm^{-1})	1.301
F (000)	856
R_1	0.0300
Unique reflections/[$I > 2\sigma(I)$]	1448/1269
wR_2	0.0805
Goodness-of-fit	1.04

3.4. Physical measurements

Microanalyses (C,H,N) were performed using a Perkin–Elmer 240C elemental analyser. Infrared spectra were recorded on a Perkin–Elmer L120-00A FT-IR spectrometer with the samples prepared as KBr pellets. Electronic spectra were recorded on a Shimadzu UV-2401 PC spectrophotometer. ^1H NMR spectra were obtained on Brucker Avance DPX 300 and Brucker RPX 500 NMR spectrometers in $CDCl_3$, using TMS as the internal standard.

3.5. Crystallography

A crystal of $C_{18}H_{15}N_4PdCl$ was grown by slow diffusion of hexane into a dichloromethane solution at 298 K. Data were collected by the ω-scan technique on a Enraf–Nonius CAD4 diffractometer with Mo Kα radiation monochromated by a graphite crystal. The structure solution was done by the direct method with the SHELXS-97 program. Full matrix least square refinements were performed using the SHELX-97 program (PC version). All non-hydrogen atoms were refined anisotropically using reflections $I > 2\sigma(I)$. Hydrogen atoms were included in calculated positions. The crystal data and data collection parameters are listed in Table 2.

4. Conclusion

Reactions of transition metal substrates with preformed ligands describe the actual metal–ligand interaction which is important to rationalise several biological and catalytic processes. The work presented here is a unique example in this regard. In this report we have described that the M–C interaction in polyfunctional organic molecules may be influenced not only electronically but also by suitable arrangements of the hetero donor atoms. The *N*-alkylated arylazo anilines, HL^a, were demonstrated to be appropriate for C–H bond activation via orthometallation [7] while

reactions of the newly synthesised ligands, HL with Na_2PdCl_4 afforded the remarkable azoimine chelates. A plausible rationale has been given considering the logical progress of the reactions.

Acknowledgments

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Appendix A. Supplementary data

Figs. S1–S6, UV–Vis spectra; Figs. S7–S12, IR spectra and Figs. S13–S18, ^{1}H NMR spectra are supplied as supplementary material. Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC reference number 250543. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, e-mail: deposite@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.poly.2006.03.019.

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