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Key indicators

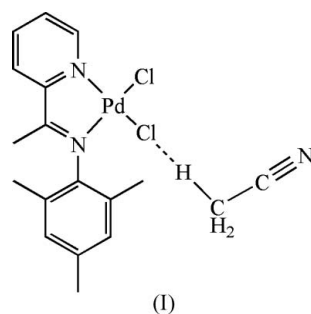
Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
 R factor = 0.039
 wR factor = 0.110
Data-to-parameter ratio = 17.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Dichloro{(E)-2,4,6-trimethyl-N-[1-(2-pyridyl)-
ethylidene]aniline- $\kappa^2\text{N},\text{N}'$ }palladium(II)
acetonitrile solvateThe title complex, $[\text{PdCl}_2(\text{C}_{16}\text{H}_{18}\text{N}_2)] \cdot \text{C}_2\text{H}_3\text{N}$, contains a slightly distorted square planar Pd^{II} center coordinated by two N and two Cl atoms. The crystal packing is stabilized by $\text{C}-\text{H} \cdots \text{Cl}$ interactions.

Received 12 June 2006

Accepted 28 June 2006

Comment

In the past few decades, palladium-catalyzed cross-coupling reactions, such as Heck (Beletskaya & Cheprakov, 2000), Suzuki (Miyaura & Suzuki, 1995; González-Arellano *et al.*, 2004), Sonogashira (Negishi & Anastasia, 2003) and Hartwig–Buchwald (Hartwig, 1998; Wolfe *et al.*, 1998) reactions, have become some of the most powerful and convenient methods for carbon–carbon bond formation. Recently, palladium-catalyzed Suzuki reactions involving cross-coupling of aryl halides with aryl boronic acids have emerged as the most important synthetic methods for preparation of biaryl compounds (Miyaura & Suzuki, 1995; Na *et al.*, 2004; Rajagopal *et al.*, 2002; Li, 2003; Tomioka *et al.*, 2004; Nicolaou *et al.*, 2005). Nevertheless, there has been a continuing interest in the further development of more efficient and selective catalytic systems for the synthesis of biaryls. For example, alternative ligands, such as sterically demanding trialkylphosphines (Wolfe *et al.*, 1998; Littke & Fu, 2002) and *N*-heterocyclic carbenes (Stauffer *et al.*, 2000; Navarro *et al.*, 2004), have been described as potentially effective ligands for Suzuki reactions under mild temperatures. However, only a few examples of pyridyl–imine palladium complexes have been reported as catalysts in coupling reactions (Pelagattia *et al.*, 2005; Lai *et al.*, 2005). We report here the synthesis and crystal structure of the title palladium(II) complex, (I), a potential catalyst in cross-coupling reactions.



Compound (I) is a mononuclear palladium(II) complex with the metal center bound to two N atoms, one from the imine group and one from the pyridine ring, and two Cl atoms (Fig. 1). The coordination geometry around Pd^{II} atom is slightly distorted square planar (Table 1). The Pd–N and Pd–Cl distances (Table 1) are similar to those observed in

other Schiff base palladium(II) complexes (Chen *et al.*, 2005). The *trans* angles (N2–Pd–Cl1 and N1–Pd–Cl2) in the PdN₂Cl₂ core do not deviate by more than 7° from the ideal value of 180°. The dihedral angle between the benzene and pyridine rings is 88.8(5)°. The crystal structure of (I) is stabilized by C–H···Cl interactions (Table 2).

Experimental

The title compound was prepared by the reaction of palladium(II) dichloride (1 mmol) with (*E*)-2,4,6-trimethyl-*N*-(1-(pyridin-2-yl)ethylidene)benzenamine (1 mmol) in acetonitrile (20 ml). The mixture was stirred for 12 h and filtered. The filtrate was slowly evaporated at room temperature to yield yellow crystals. Yield 0.42 g (92%).

Crystal data

[PdCl ₂ (C ₁₆ H ₁₈ N ₂)]·C ₂ H ₃ N	<i>Z</i> = 4
<i>M_r</i> = 456.68	<i>D_x</i> = 1.547 Mg m ⁻³
Monoclinic, <i>P</i> 2 ₁ / <i>n</i>	Mo <i>K</i> α radiation
<i>a</i> = 8.1699 (7) Å	<i>μ</i> = 1.22 mm ⁻¹
<i>b</i> = 19.7209 (17) Å	<i>T</i> = 298 (2) K
<i>c</i> = 12.2323 (11) Å	Parallelepiped, yellow
<i>β</i> = 95.783 (2)°	0.45 × 0.33 × 0.14 mm
<i>V</i> = 1960.8 (3) Å ³	

Data collection

Bruker SMART 1000 CCD diffractometer	10980 measured reflections
<i>φ</i> and <i>ω</i> scans	3854 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	2980 reflections with <i>I</i> > 2σ(<i>I</i>)
<i>T_{min}</i> = 0.774, <i>T_{max}</i> = 1.000 (expected range = 0.652–0.843)	<i>R_{int}</i> = 0.037
	<i>θ_{max}</i> = 26.0°

Refinement

Refinement on <i>F</i> ²	H-atom parameters constrained
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.039	<i>w</i> = 1/[σ ² (<i>F_o</i> ²) + (0.065 <i>P</i>) ²]
<i>wR</i> (<i>F</i> ²) = 0.110	where <i>P</i> = (<i>F_o</i> ² + 2 <i>F_c</i> ²)/3
<i>S</i> = 1.05	(Δ/ <i>σ</i>) _{max} < 0.001
3854 reflections	Δ <i>ρ</i> _{max} = 0.74 e Å ⁻³
220 parameters	Δ <i>ρ</i> _{min} = -0.42 e Å ⁻³

Table 1

Selected geometric parameters (Å, °).

Pd–N2	2.022 (3)	Pd–Cl2	2.2916 (11)
Pd–N1	2.028 (3)	N1–C2	1.294 (5)
Pd–Cl1	2.2780 (12)		
N2–Pd–N1	79.92 (13)	N2–Pd–Cl2	94.54 (10)
N2–Pd–Cl1	174.99 (10)	N1–Pd–Cl2	173.57 (10)
N1–Pd–Cl1	95.24 (10)	Cl1–Pd–Cl2	90.37 (5)
C2–N1–C8–C9	89.6 (5)		

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
C18–H18C···Cl1	0.96	2.61	3.502 (8)	154
C1–H1B···Cl2 ⁱ	0.96	2.76	3.684 (5)	161

Symmetry code: (i) *x* + 1, *y*, *z*.

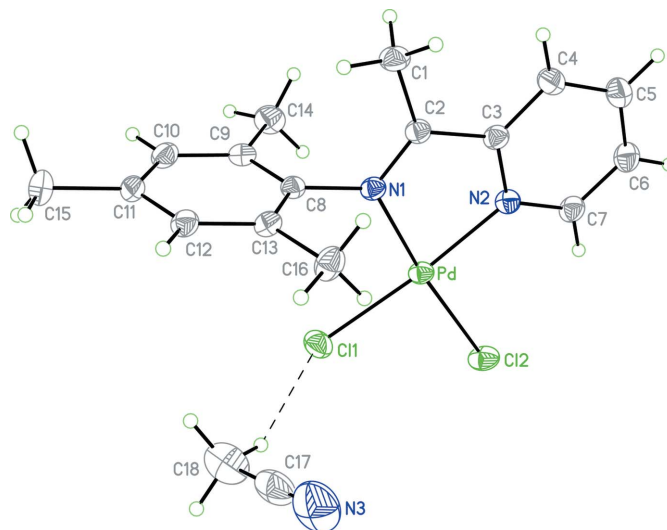


Figure 1

A view of the molecular structure of (I), with displacement ellipsoids shown at the 20% probability level. H atoms are represented by circles of arbitrary size and the dashed line indicates a hydrogen bond.

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry with C–H distances of 0.96 Å and *U*_{iso}(H) = 1.5*U*_{eq}(C), but each group was allowed to rotate freely about its C–C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C–H distances of 0.93 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

Financial support from the National Science Council of the Republic of China is gratefully appreciated. Helpful comments from the reviewers are also greatly appreciated.

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