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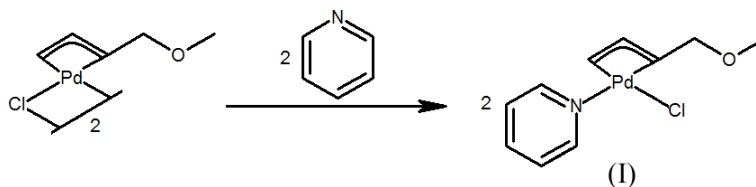
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Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$
 R factor = 0.041
 wR factor = 0.105
Data-to-parameter ratio = 21.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Chloro(1-methoxymethyl- π -allyl)-
(pyridine- κN)palladium(II)The structure of the novel title π -allyl palladium complex, $[\text{PdCl}(\text{C}_5\text{H}_9\text{O})(\text{C}_5\text{H}_5\text{N})]$, has been determined. The allyl fragment is slightly skewed.Received 5 January 2004
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Comment

Palladium is one of the most widely used transition metals in catalytic reactions (Tsuji, 1998). Synthetically, the reaction of allylic alkylation *via* π -allyl palladium complexes is a very useful, versatile and powerful tool for C–C, C–O and C–N bond formation (Blystone, 1989; Trost & Vranken, 1996; Ritleng *et al.*, 2002; Feuerstein *et al.*, 2001). In several cases, the intermediate complexes are stable enough for X-ray crystallographic determination, and a number of structures have been reported (Cambridge Structural Database, Version 5 of November 2003; Allen, 2002). The regulating ligands play a crucial role in regio- and stereoselectivity in nucleophilic addition reactions (Pregosin & Salzmann, 1996; Albinati *et al.*, 1991) and consequently in the products of reaction. Therefore, it is important to establish what influence these regulating ligands have on π -allyl conjugated systems. The molecular structure and atomic numbering of the title compound, (I), are given in Fig. 1.



The pyridine ligand coordinates to the Pd atom using its unshared pair of electrons of atom N1. The pyridine is located *trans* to the substituted C atom of the allyl fragment. The result of this present X-ray analysis shows that the structural features of pyridine remain practically unchanged on coord-

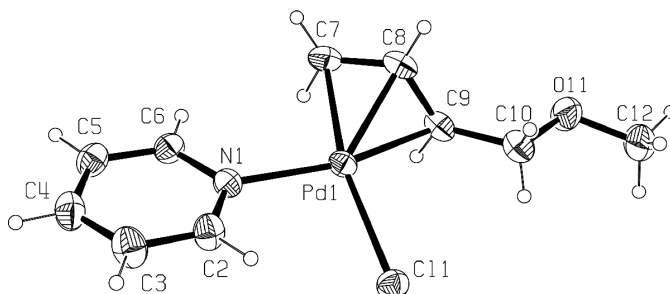


Figure 1
ORTEP-3 (Farrugia, 2003) plot of the title compound. Displacement ellipsoids are shown at the 30% probability level.

ination. Atom Pd1 is slightly displaced from the plane of the pyridine ring [0.072 (6) Å]; the dihedral angle between the pyridine plane and the N1–Pd1–C11 plane is 25.0 (2)°; the N1–Pd1–C11 angle is 94.4 (1)°; the distance from Pd1 to the plane of the π -allyl ligand (C7–C8–C9) is 1.667 (8) Å; the dihedral angle between the plane of the π -allyl ligand and the N1–Pd1–C11 plane is 67.2 (5)°; the C7–C8–C9 angle in the π -allyl ligand is 118.9 (5)°; the distance from Pd1 to *cis*-C7 is 2.124 (5) Å and the distance from Pd1 to *trans*-C9 is 2.136 (5) Å, reflecting the influence of the –CH₂OCH₃ group. This group has a (–)antiperiplanar orientation with respect to Pd1 [Pd1–C9–C10–O11 = –179.0 (3)°]. The O11–C12 bond is perpendicular to the plane of the allyl ligand. Although the non-planar molecule is chiral, the crystal structure is racemic. Insertion of pyridine into the coordination sphere of palladium leads to a slight skewing of the π -allyl ligand. This distortion may lead to an increase in the reactivity of π -allyl–palladium complexes, and may influence the direction of attack of nucleophilic reagents (*e.g.* in direct allylic alkylation).

Experimental

Chlorobis(1-methoxymethyl- π -allyl)palladium (25 mg, 0.055 mmol; Huttel, 1970) was dissolved in 1 ml of chloroform, and a double excess of pyridine (17.8 ml, 0.22 mmol) was added. The mixture was stirred at room temperature for 5 min. 15 ml of heptane was then added. A yellow precipitate was filtered off, washed with petroleum ether and dried in a vacuum at room temperature, giving 27.4 mg (81% yield). Crystals suitable for X-ray study were obtained by crystallization from chloroform/heptane and are air-stable.

Crystal data

[PdCl(C₅H₉O)(C₅H₅N)]
 $M_r = 306.07$
 Monoclinic, $P2_1/n$
 $a = 8.627$ (5) Å
 $b = 13.115$ (4) Å
 $c = 10.299$ (2) Å
 $\beta = 90.38$ (2)°
 $V = 1165.2$ (8) Å³
 $Z = 4$

$D_x = 1.745$ Mg m^{–3}
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 15$ – 16°
 $\mu = 1.79$ mm^{–1}
 $T = 293$ K
 Prism, yellow
 $0.3 \times 0.3 \times 0.3$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 Non-profiled $\omega/2\theta$ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.536$, $T_{\max} = 0.584$
 2962 measured reflections
 2815 independent reflections
 2177 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.051$
 $\theta_{\text{max}} = 28.0^\circ$
 $h = -11 \rightarrow 11$
 $k = 0 \rightarrow 17$
 $l = 0 \rightarrow 13$
 1 standard reflection every 238 reflections
 intensity decay: 3%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.105$
 $S = 1.06$
 2815 reflections
 130 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 0.091P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.99$ e Å^{–3}
 $\Delta\rho_{\text{min}} = -0.64$ e Å^{–3}

Table 1

Selected geometric parameters (Å, °).

Pd1–C8	2.105 (5)	C3–C4	1.371 (7)
Pd1–C7	2.124 (5)	C4–C5	1.366 (7)
Pd1–N1	2.128 (4)	C5–C6	1.362 (7)
Pd1–C9	2.136 (5)	C7–C8	1.390 (7)
Pd1–C11	2.3787 (13)	C8–C9	1.374 (7)
N1–C2	1.329 (5)	C9–C10	1.499 (7)
N1–C6	1.341 (5)	C10–O11	1.395 (6)
C2–C3	1.377 (7)	O11–C12	1.413 (6)
C8–Pd1–C7	38.4 (2)	C4–C3–C2	118.9 (5)
C8–Pd1–N1	132.14 (18)	C5–C4–C3	118.4 (5)
C7–Pd1–N1	98.69 (17)	C6–C5–C4	119.5 (5)
C8–Pd1–C9	37.81 (19)	N1–C6–C5	123.1 (4)
C7–Pd1–C9	68.0 (2)	C8–C7–Pd1	70.1 (3)
N1–Pd1–C9	165.92 (16)	C9–C8–C7	118.9 (5)
C8–Pd1–C11	130.00 (15)	C9–C8–Pd1	72.3 (3)
C7–Pd1–C11	166.82 (15)	C7–C8–Pd1	71.5 (3)
N1–Pd1–C11	94.42 (10)	C8–C9–C10	124.4 (4)
C9–Pd1–C11	98.85 (14)	C8–C9–Pd1	69.9 (3)
C2–N1–C6	116.9 (4)	C10–C9–Pd1	121.1 (3)
C2–N1–Pd1	122.6 (3)	O11–C10–C9	108.6 (4)
C6–N1–Pd1	120.5 (3)	C10–O11–C12	113.2 (4)
N1–C2–C3	123.0 (5)		

H atoms were positioned geometrically (C–H = 0.89–0.96 Å) and refined using a riding model, with $U_{\text{iso}} = 1.2$ or $1.5U_{\text{eq}}$ of the parent atom.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1994); cell refinement: *CAD-4 Software*; data reduction: *PROFIT* (Strel'tsov & Zavodnik, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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