at the final stage of refinement F(25) had shifted abnormally towards P(2) to give a P(2)—F(25) distance of 1.01 Å. Although the difference Fourier calculation after refinement without F(25) shows a peak around 1.3 Å from P(2), succesive refinement including F(25) again converged with a shift of F(25) towards the former position, so refinement was terminated at this stage. No attempt was made to locate H atoms. All computations were performed using *UNICS*III (Sakurai & Kobayashi, 1979) and *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic thermal parameters and complete geometry have been deposited with the IUCr (Reference: AS1122). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Cyclopalladated Complex, $[PdCl(C_{37}H_{32}N)]$

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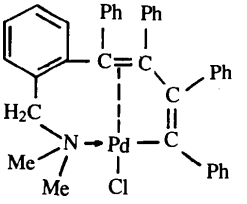
Abstract

In the structure of chloro{4-[2-(dimethylaminomethyl)phenyl]-1,2,3,4-tetraphenyl-(3,4-η)-buta-1,3-dienyl-C^1,N}palladium(II), (1), the Pd atom is surrounded by five atoms [C(1), C(3), C(4), Cl and N], but if the midpoint of the ligand C(3)=C(4) bond is considered as a coordination site, the environment around the Pd atom is nearly square planar.

Comment

Cyclopalladated compounds are an important part of modern organometallic chemistry and they have attracted much attention owing to their structural features (Dehand *et al.*, 1983; Caires, Mauro, Santos, Gambardella & Lechat, 1994) and applications in organic synthesis and catalysis (Ryabov, 1985). Insertion of alkynes into the Pd—C bond of cyclopalladated complexes has proved to be a very interesting route for the preparation of novel organometallic molecules. In the present paper we report the X-ray structure analysis of (1).



(1)

The compound was prepared by the interaction of diphenylacetylene with [Pd(*N*,*N*-dimethylbenzylamine)Cl]$_2$, as described in the literature (Bahsoun *et al.*, 1979). The results of this study are illustrated in

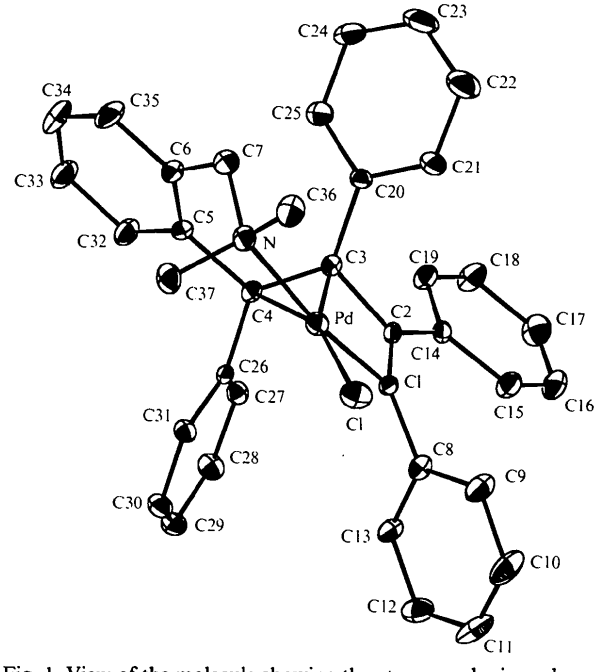


Fig. 1. View of the molecule showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% level.

Fig. 1 which also shows the labelling scheme. The atoms bonded directly to the Pd atom, C(1), Cl and N, and the midpoint of C(3) and C(4), almost lie in the same plane, the largest deviation from the mean plane being 0.124 (5) Å. Thus, the environment around the Pd atom is nearly square planar. The Pd—C(1) distance is significantly shorter than the Pd—C(3) and Pd—C(4) distances (Table 2).

Experimental

Crystal data

$[PdCl(C_{37}H_{32}N)]$	Mo $K\alpha$ radiation
M_r = 632.51	λ = 0.71069 Å
Orthorhombic	Cell parameters from 25 reflections
Pbca	θ = 11–21°
a = 14.300 (1) Å	μ = 0.72 mm^{-1}
b = 18.202 (1) Å	Room temperature
c = 23.347 (1) Å	Plate-like
V = 6077.0 (6) Å^3	0.3 × 0.2 × 0.1 mm
Z = 8	Yellow
D_x = 1.383 Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4 diffractometer	θ_{max} = 30.3°
ω–2θ scans	h = 0 → 20
Absorption correction: none	k = 0 → 25
9173 measured reflections	l = 0 → 33
9173 independent reflections	3 standard reflections
4926 observed reflections [$I > 2.5\sigma(I)$]	frequency: 120 min
	intensity variation: none

Refinement

Refinement on F	$(\Delta/\sigma)_{max}$ = 0.001
R = 0.052	$\Delta\rho_{max}$ = 0.83 e Å^{-3}
wR = 0.041	$\Delta\rho_{min}$ = −0.94 e Å^{-3}
S = 3.62	Extinction correction: none
4926 reflections	Atomic scattering factors from *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989)
361 parameters	
H-atom parameters not refined	
$w = 1/\sigma^2(F)$	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Pd	0.92415 (2)	0.09808 (2)	0.15403 (1)	2.87 (2)
Cl	1.03441 (9)	0.00936 (7)	0.17723 (5)	4.12 (6)
N	0.8168 (3)	0.0379 (2)	0.2042 (2)	3.1 (2)
C(1)	1.0139 (3)	0.1655 (2)	0.1145 (2)	2.1 (2)
C(2)	0.9674 (3)	0.2277 (3)	0.1170 (2)	2.2 (2)
C(3)	0.8704 (3)	0.2092 (2)	0.1387 (2)	2.3 (2)
C(4)	0.8184 (3)	0.1605 (2)	0.1045 (2)	2.2 (2)
C(5)	0.7154 (3)	0.1445 (2)	0.1163 (2)	2.6 (2)
C(6)	0.6795 (3)	0.1138 (2)	0.1675 (2)	3.0 (2)
C(7)	0.7369 (4)	0.0877 (3)	0.2176 (2)	3.7 (2)
C(8)	1.1022 (3)	0.1461 (2)	0.0870 (2)	2.6 (2)
C(9)	1.1817 (4)	0.1244 (3)	0.1176 (2)	4.5 (3)
C(10)	1.2623 (4)	0.1055 (3)	0.0897 (3)	5.7 (3)
C(11)	1.2666 (4)	0.1067 (3)	0.0309 (3)	5.4 (3)
C(12)	1.1906 (4)	0.1290 (3)	0.0003 (2)	4.3 (3)
C(13)	1.1088 (3)	0.1475 (3)	0.0282 (2)	3.2 (2)
C(14)	0.9942 (3)	0.3037 (3)	0.1024 (2)	2.4 (2)
C(15)	1.0875 (3)	0.3229 (3)	0.0921 (2)	3.4 (3)
C(16)	1.1117 (3)	0.3940 (3)	0.0772 (2)	4.5 (3)
C(17)	1.0438 (4)	0.4489 (3)	0.0733 (2)	4.5 (3)
C(18)	0.9528 (4)	0.4305 (3)	0.0839 (2)	4.1 (3)
C(19)	0.9276 (3)	0.3595 (3)	0.0984 (2)	3.0 (2)
C(20)	0.8372 (3)	0.2462 (2)	0.1920 (2)	2.5 (2)
C(21)	0.8985 (3)	0.2548 (3)	0.2369 (2)	3.9 (3)
C(22)	0.8713 (5)	0.2880 (3)	0.2879 (2)	5.7 (3)
C(23)	0.7822 (5)	0.3127 (3)	0.2938 (2)	5.6 (3)
C(24)	0.7191 (4)	0.3060 (3)	0.2500 (3)	4.4 (3)
C(25)	0.7465 (4)	0.2739 (2)	0.1980 (2)	3.4 (2)
C(26)	0.8453 (3)	0.1458 (3)	0.0431 (2)	2.1 (2)
C(27)	0.8514 (3)	0.2046 (2)	0.0049 (2)	2.6 (2)
C(28)	0.8730 (3)	0.1916 (3)	−0.0523 (2)	3.2 (3)
C(29)	0.8907 (3)	0.1215 (3)	−0.0713 (2)	3.5 (3)
C(30)	0.8841 (3)	0.0620 (3)	−0.0338 (2)	3.2 (2)
C(31)	0.8605 (3)	0.0748 (2)	0.0228 (2)	2.7 (2)
C(32)	0.6539 (3)	0.1653 (3)	0.0732 (2)	3.6 (3)
C(33)	0.5572 (4)	0.1602 (3)	0.0811 (2)	4.6 (3)
C(34)	0.5232 (4)	0.1341 (3)	0.1305 (3)	5.5 (3)
C(35)	0.5825 (4)	0.1092 (3)	0.1741 (2)	4.8 (3)
C(36)	0.8553 (3)	0.0140 (3)	0.2612 (2)	4.5 (3)
C(37)	0.7827 (3)	−0.0256 (3)	0.1718 (2)	3.8 (3)

Table 2. *Selected bond lengths* (Å)

Pd—Cl	2.321 (1)	Pd—N	2.220 (3)
Pd—C(1)	2.002 (4)	Pd—C(3)	2.193 (4)
Pd—C(4)	2.217 (4)	Pd—C(3-4)	2.090
C(1)—C(2)	1.316 (7)	C(2)—C(3)	1.514 (6)
C(3)—C(4)	1.406 (6)	C(4)—C(5)	1.527 (6)
C(5)—C(6)	1.415 (7)	C(6)—C(7)	1.506 (7)
C(7)—N	1.492 (7)		

Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Structure solution: *SHELXS*86 (Sheldrick, 1985). Structure refinement: *NRCVAX*. Molecular graphics: *ORTEP* (Johnson, 1965).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1095). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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