Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Chloro(*P*-{2-[*N*-(2,6-diisopropylphenyl)iminomethyl]phenyl}-*P*,*P*-diphenylphosphine-κ^2N,*P*)-methylpalladium(II)

Anthony L. Spek

Bijvoet Center for Biomolecular Research, Department of Crystal and Structural Chemistry, Utrecht University, Padualaan 8, 3584 CH Utrecht, The Netherlands

Correspondence e-mail: a.l.spek@chem.uu.nl

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.023
wR factor = 0.053
Data-to-parameter ratio = 19.5

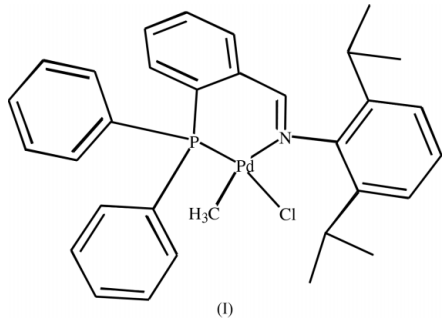
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, [Pd(CH$_3$)Cl(C$_{31}$H$_{32}$NP)], is a modification of the previously reported dimethoxy derivative. Both molecular structures are essentially identical.

Received 3 February 2003
Accepted 5 February 2003
Online 14 February 2003

Comment

The synthesis and catalytic properties of a number of new palladium iminophosphine catalysts for the oligomerization of ethene were reported by van den Beuken *et al.* (1998). The current paper reports the crystal structure of a minor modification of the crystal structure reported in that paper, *viz*. (*P*,*P*-di-*o*-anisyl-*P*-{2-[*N*-(2,6-diisopropylphenyl)-iminomethyl]phenyl}phosphine)chloromethylpalladium(II). The present structure, (I), does not contain any solvent of crystallization.



(I)

Experimental

See van den Beuken *et al.* (1998) for details of the synthesis of the title compound. Crystals were obtained from a dichloromethane/hexane mixture.

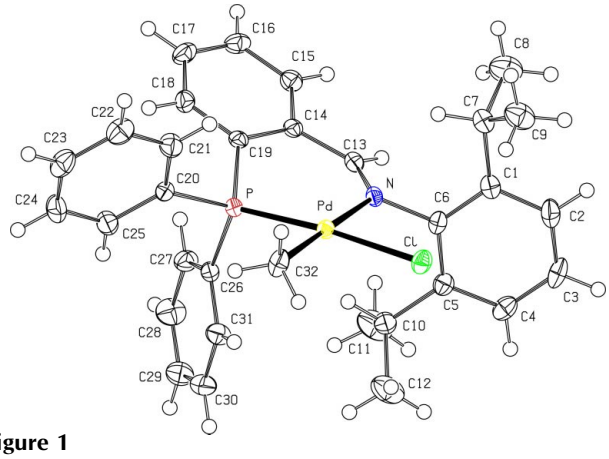


Figure 1
View of the title compound, with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

Crystal data

[Pd(CH$_3$)Cl(C$_{31}$H$_{32}$NP)]
M_r = 606.45
Orthorhombic, $P2_12_12_1$
a = 11.6099 (7) Å
b = 14.2132 (8) Å
c = 17.0508 (10) Å
V = 2813.6 (3) Å^3
Z = 4
D_x = 1.432 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 11.4–13.7°
μ = 0.83 mm^{-1}
T = 150 K
Block, yellow
0.50 × 0.38 × 0.25 mm

Data collection

Enraf–Nonius TurboCAD-4 diffractometer
ω scans
7202 measured reflections
6433 independent reflections
6164 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 27.4°
$h = 0 \rightarrow 15$
$k = -18 \rightarrow 18$
$l = -22 \rightarrow 0$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.053
S = 1.02
6433 reflections
330 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + 0.663P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = −0.020 (17)

Table 1
Selected geometric parameters (Å, °).

Pd—Cl	2.3668 (5)	P—C20	1.815 (2)
Pd—P	2.1910 (6)	P—C26	1.833 (2)
Pd—N	2.1694 (18)	N—C6	1.450 (3)
Pd—C32	2.048 (2)	N—C13	1.278 (3)
P—C19	1.821 (2)		
Cl—Pd—P	175.19 (2)	Pd—N—C6	114.55 (13)
Cl—Pd—N	93.32 (5)	Pd—N—C13	130.42 (15)
Cl—Pd—C32	87.32 (7)	C6—N—C13	114.77 (18)
P—Pd—N	89.58 (5)	N—C6—C1	119.17 (19)
P—Pd—C32	89.66 (7)	N—C6—C5	118.35 (19)
N—Pd—C32	178.03 (9)	N—C13—C14	127.5 (2)
Pd—P—C19	112.52 (7)	P—C19—C14	121.25 (15)
Pd—P—C20	119.79 (8)	P—C19—C18	119.88 (17)
Pd—P—C26	110.55 (7)	P—C20—C21	117.85 (16)
C19—P—C20	104.45 (10)	P—C20—C25	122.95 (17)
C19—P—C26	102.07 (10)	P—C26—C27	123.04 (17)
C20—P—C26	105.79 (9)	P—C26—C31	117.78 (17)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···Cl^{i}	0.93	2.78	3.584 (2)	145
C22—H22···Cl^{ii}	0.93	2.79	3.708 (3)	169

Symmetry codes: (i) $2-x, \frac{1}{2}+y, \frac{3}{2}-z$; (ii) $x-\frac{1}{2}, \frac{3}{2}-y, 2-z$.

No correction for absorption was deemed necessary in view of the only minor intensity variations observed with 360° ψ scans. H atoms were introduced at calculated positions and refined riding on their carrier atoms, with standard C—H values (*SHELXL*97; Sheldrick, 1997). The absolute structure is based on 2832 Bijvoet pairs (the molecule is achiral).

Data collection: locally modified *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *SET*4 (de Boer & Duisenberg, 1984); data reduction: *HELENA* (Spek, 1997); program(s) used to solve structure: *DIRDIF*99 (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2002); software used to prepare material for publication: *PLATON*.

Crystals were obtained from Professor B. L. Feringa and Dr E. K. van den Beuken. This work was supported in part by the Council for the Chemical Sciences of the Netherlands Organization for Scientific Research (CW–NWO).

References

Beuken, E. K. van den, Smeets, W. J. J., Spek, A. L. & Feringa, B. L. (1998). *Chem. Commun.* pp. 223–224.
Beurskens, P. T., Beurskens, G., de Gelder, R., García-Granda, S., Gould, R. O., Israel, R. & Smits, J. M. M. (1999). *The DIRDIF*99 *Program System*. Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
Boer, J. L. de & Duisenberg, A. J. M. (1984). *Acta Cryst.* A**40**, C-410.
Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (1997). *HELENA*. Utrecht University, The Netherlands.
Spek, A. L. (2002). *PLATON*. Utrecht University, The Netherlands.