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trans-Dibromobis(triphenylphosphine)palladium(II) Tetrahydrofuran Solvate and *trans*-Dichlorobis(triphenylphosphine)palladium(II) Bis(chloroform) Solvate

J. L. Stark and **K. H. Whitmire**

Abstract

Trans-dibromobis(triphenylphosphine)palladium(II), $[(C_6H_5)_3P]_2PdBr_2$ (1), crystalizes in the triclinic space group P-1. Trans-dichlorobis(triphenylphosphine)palladium(II), $[(C_6H_5)_3P]_2PdCl_2$ (2) also crystalizes in the triclinic space group P-1. $[(C_6H_5)_3P]_2PdBr_2$ is solvated with one THF molecule and has a square planar geometry with the bromide ligands coordinated in a *trans* configuration. $[(C_6H_5)_3P]_2PdCl_2$ is solvated with two chloroform molecules and has a square planar geometry with the palladium, Pd1, residing on an inversion center.

Comment

The title compounds were obtained while trying to synthesize a palladium-bismuth cluster. Related compounds are *trans*-$[PdI_2(PPh_3)_2]\cdot CHCl_3$ (Kubota, Ohba & Saito, 1991), *trans*-$[PdI_2(PPh_3)_2]\cdot 2CH_2Cl_2$ (Debaerdemaeker, Kutoglu, Schmid, & Weber, 1973), *trans*-$[PdI_2(PPhMe_2)_2]$ (Bailey & Mason, 1968), *trans*-$[PdCl_2(PPh_3)_2]$ (Ferguson, McCrindle, McAlees & Parvez, 1982), and *trans*-$[PdCl_2(PPh_3)_2].1/2(p\text{-}C_6H_4Cl_2)$ (Kitano, Kinoshita, Nakamura & Ashida). While the structure is known for $[(C_6H_5)_3P]_2PdCl_2$ this structure is solvated with two chloroform molecules and has a unique unit cell.

Experimental

Synthesis was carried out by reaction of $Pd[PPh_3]_4$ and $BiBr_3$, or $BiCl_3$, (molar ratio 1:1) in tetrahydrofuran (thf) solution for 15 h. Orange block-like crystals of $[(C_6H_5)_3P]_2PdBr_2$ were obtained by recrystalization from THF. $[(C_6H_5)_3P]_2PdCl_2$ was recrystalized from chloroform to give yellow crystals suitable for X-ray analysis.

Refinement

For compound (1) $\Delta\rho_{max}$ is 0.77Å from Pd1 and $\Delta\rho_{min}$ is 0.57Å from Br1. For compound (2) $\Delta\rho_{max}$ is 1.18Å from Cl11 and $\Delta\rho_{min}$ is 0.43Å from Cl11.

Computing details

For both compounds, data collection: *TEXSAN* 5.0; cell refinement: *TEXSAN* 5.0; data reduction: *TEXSAN* 5.0; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* for (1); *SHELXL*PLUS for (2). For both compounds, software used to prepare material for publication: *SHELXL93*

CIF access

trans-dibromobis(triphenylphosphine)palladium(II)

Crystal data

$[PdBr_2(C_{18}H_{15}P)_2]\cdot C_4H_8O$	$\gamma = 98.73\ (3)^o$
$M_r = 862.86$	$V = 1820.1\ (6)\ Å^3$
Triclinic, $P\overline{1}$	$Z = 2$
$a = 9.356\ (2)$ Å	Mo $K\alpha$
$b = 9.609\ (2)$ Å	$\mu = 2.82\ mm^{-1}$
$c = 20.529\ (4)$ Å	$T = 223\ (2)$ K
$\alpha = 92.16\ (3)^o$	0.25 × 0.25 × 0.20 mm
$\beta = 92.81\ (3)^o$	

Data collection

Rigaku AFC%-5 diffractometer	3991 reflections with $I > 2\sigma(I)$
Absorption correction: ψ scan	$R_{int} = 0.021$
$T_{min} = 0.517$, $T_{max} = 0.568$	3 standard reflections
5097 measured reflections	every 150 reflections
4758 independent reflections	intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$	416 parameters
$wR(F^2) = 0.142$	H-atom parameters constrained
$S = 1.05$	$\Delta\rho_{max} = 0.91\ e\ Å^{-3}$
4756 reflections	$\Delta\rho_{min} = -1.26\ e\ Å^{-3}$

Table 1
Selected geometric parameters (Å, °)

Pd1—P1	2.336 (2)	Pd1—Br1	2.4148 (13)
Pd1—P2	2.337 (2)	Pd1—Br2	2.4169 (13)
P1—Pd1—P2	176.06 (6)	P1—Pd1—Br2	92.76 (6)
P1—Pd1—Br1	86.82 (6)	P2—Pd1—Br2	88.12 (6)
P2—Pd1—Br1	92.18 (6)	Br1—Pd1—Br2	178.09 (4)

trans-dichlorobis-(triphenylphosphine)palladium(II)

Crystal data

$[PdCl_2(C_{18}H_{15}P)_2]\cdot 2CHCl_3$	$\gamma = 74.65\ (3)^o$
$M_r = 940.58$	$V = 996.6\ (4)\ Å^3$
Triclinic, $P\overline{1}$	$Z = 1$
$a = 7.880\ (2)$ Å	Mo $K\alpha$
$b = 11.201\ (2)$ Å	$\mu = 1.11\ mm^{-1}$

c = 12.043 (2) Å	T = 223 (2) K
α = 89.19 (3)º	0.40 × 0.40 × 0.40 mm
β = 76.76 (3)º	

Data collection

Rigaku AFC5-S diffractometer	R_{int} = 0.022
Absorption correction: ψ scans	θ_{max} = 23.9º
T_{min} = 0.504, T_{max} = 0.641	3 standard reflections
2828 measured reflections	every 150 reflections
2647 independent reflections	intensity decay: none
2420 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053	223 parameters
$wR(F^2)$ = 0.140	H-atom parameters constrained
S = 1.03	$\Delta\rho_{max}$ = 1.81 e Å^{-3}
2647 reflections	$\Delta\rho_{min}$ = −1.41 e Å^{-3}

Table 2
Selected geometric parameters (Å, °)

Pd1—Cl1^{i}	2.293 (2)	P1—C11	1.814 (6)
Pd1—Cl1	2.293 (2)	P1—C21	1.819 (6)
Pd1—P1^{i}	2.343 (2)	P1—C31	1.824 (6)
Pd1—P1	2.343 (2)		
Cl1^{i}—Pd1—Cl1	180.0	P1^{i}—Pd1—P1	180.0
Cl1^{i}—Pd1—P1^{i}	92.88 (6)	C11—P1—Pd1	112.5 (2)
Cl1—Pd1—P1^{i}	87.12 (6)	C21—P1—Pd1	111.1 (2)
Cl1^{i}—Pd1—P1	87.12 (6)	C31—P1—Pd1	117.2 (2)
Cl1—Pd1—P1	92.88 (6)		

Symmetry codes: (i) −*x*, −*y*, −*z*.

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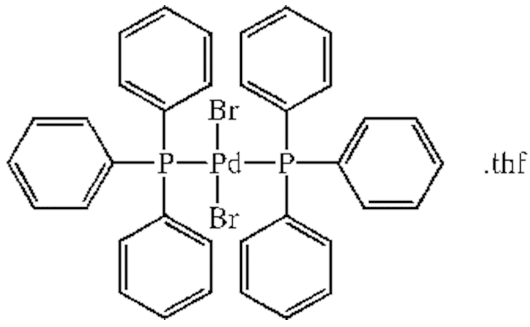
References

Bailey, N. A. & Mason, R. (1968). *J. Chem. Soc. A*, 2594–2605.
Coucouvanis, P., Swenson, D., Stremple, P., Baenziger, N.C., J. Am. Chem. Soc., 101, 1979, 3392-3394.
Dedaerdemaeker, T., Kutoglu, A., Schmid, G. & Weber, L. (1973). *Acta Cryst.* B**29**, 1283–1288.
Ferguson, G., McCrindle, R., McAlees, A. J. & Parvez, M. (1982). *Acta Cryst.* B**38**, 2679–2681.
Kitano, Y., Kinoshita, Y., Nakamura, R. & Ashida, T. (1983). *Acta Cryst.* C**39**, 1015–1017.
Kubota, M., Ohba, S. & Saita, Y. (1991). *Acta Cryst.* C**47**, 1727–1729.

CIF access

Rauchfuss, T.B., Dev, S., Wilson, S.R., Inorg. Chem., 1992, 31, 154-156.
Sheldrick, G. M. (1986). *SHELXS86*. Program for structure solution of crystal structures. Univ. of Gőttingen, Germany.
Sheldrick, G. M. (1993). *SHELXL93*. In preparation for J. Appl. Cryst..
SHELXTL PLUS (1995). Siemens Xray Cooporation, Madison, Wisconson.
TEXSAN 5.0 (1990). The Molecular Structure Company, The Woodlands, Texas.

Scheme 1



supplementary materials

trans-dibromobis(triphenylphosphine)palladium(II)

Crystal data

$[PdBr_2(C_{18}H_{15}P)_2]\cdot C_4H_8O$	$Z = 2$
$M_r = 862.86$	$F_{000} = 864$
Triclinic, $P\overline{1}$	$D_x = 1.574$ Mg m^{-3}
$a = 9.356$ (2) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 9.609$ (2) Å	Cell parameters from 25 reflections
$c = 20.529$ (4) Å	$\theta = 3.7$–6.1º
$\alpha = 92.16$ (3)º	$\mu = 2.82$ mm^{-1}
$\beta = 92.81$ (3)º	$T = 223$ (2) K
$\gamma = 98.73$ (3)º	Block, orange
$V = 1820.1$ (6) Å^3	0.25 × 0.25 × 0.20 mm

Data collection

Rigaku AFC%-5 diffractometer	$R_{int} = 0.021$
Radiation source: normal-focus sealed tube	$\theta_{max} = 24.9$º
Monochromator: graphite	$\theta_{min} = 2.2$º
$T = 223(2)$ K	$h = -10\rightarrow9$
ω–scans	$k = 0\rightarrow10$
Absorption correction: ψ scan	$l = -24\rightarrow22$
$T_{min} = 0.517$, $T_{max} = 0.568$	3 standard reflections
5097 measured reflections	every 150 reflections
4758 independent reflections	intensity decay: none
3991 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.047$	H-atom parameters constrained
$wR(F^2) = 0.142$	Calculated $w = 1/[\sigma^2(F_o^2) + (0.0589P)^2 + 11.8164P]$ where $P = (F_o^2 + 2F_c^2)/3$?
$S = 1.05$	$\Delta\rho_{max} = 0.91$ e Å^{-3}
4756 reflections	$\Delta\rho_{min} = -1.26$ e Å^{-3}
416 parameters	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0013 (4)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement on F^2 for ALL reflections except for 2 with very negative F^2 or flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating _refine_ls_R_factor_obs *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R–factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Pd1	0.73514 (5)	0.74706 (5)	0.25547 (2)	0.0268 (2)
Br1	0.90861 (10)	0.92286 (10)	0.20917 (4)	0.0578 (3)
Br2	0.56790 (9)	0.56652 (9)	0.30252 (4)	0.0494 (3)
P1	0.8344 (2)	0.8383 (2)	0.35770 (9)	0.0289 (4)
P2	0.6500 (2)	0.6484 (2)	0.15245 (8)	0.0277 (4)
C111	0.8612 (8)	1.0295 (7)	0.3624 (3)	0.031 (2)
C112	0.7458 (9)	1.0948 (8)	0.3426 (4)	0.045 (2)
H11A	0.6576 (9)	1.0397 (8)	0.3278 (4)	0.054*
C113	0.7583 (11)	1.2393 (10)	0.3443 (4)	0.057 (2)
H11B	0.6789 (11)	1.2826 (10)	0.3309 (4)	0.068*
C114	0.8878 (13)	1.3207 (9)	0.3655 (4)	0.062 (3)
H11C	0.8968 (13)	1.4195 (9)	0.3665 (4)	0.074*
C115	1.0019 (11)	1.2577 (9)	0.3850 (5)	0.063 (3)
H11D	1.0902 (11)	1.3135 (9)	0.3990 (5)	0.076*
C116	0.9901 (9)	1.1111 (8)	0.3845 (4)	0.046 (2)
H11E	1.0690 (9)	1.0683 (8)	0.3991 (4)	0.055*
C121	0.7385 (7)	0.7900 (8)	0.4311 (3)	0.032 (2)
C122	0.6755 (8)	0.8846 (8)	0.4674 (4)	0.041 (2)
H12A	0.6802 (8)	0.9782 (8)	0.4548 (4)	0.049*
C123	0.6044 (9)	0.8412 (10)	0.5231 (4)	0.051 (2)
H12B	0.5606 (9)	0.9060 (10)	0.5476 (4)	0.062*
C124	0.5974 (9)	0.7057 (10)	0.5425 (4)	0.054 (2)
H12C	0.5492 (9)	0.6773 (10)	0.5801 (4)	0.065*
C125	0.6608 (10)	0.6133 (10)	0.5070 (4)	0.054 (2)
H12D	0.6570 (10)	0.5202 (10)	0.5201 (4)	0.065*
C126	0.7310 (9)	0.6539 (8)	0.4516 (4)	0.044 (2)
H12E	0.7744 (9)	0.5881 (8)	0.4275 (4)	0.053*
C131	1.0092 (7)	0.7819 (7)	0.3752 (3)	0.031 (2)
C132	1.0761 (8)	0.7169 (8)	0.3264 (4)	0.038 (2)
H13A	1.0297 (8)	0.6993 (8)	0.2846 (4)	0.046*
C133	1.2105 (9)	0.6779 (9)	0.3387 (4)	0.048 (2)

H13B	1.2548 (9)	0.6337 (9)	0.3053 (4)	0.057*
C134	1.2802 (9)	0.7032 (9)	0.3995 (4)	0.047 (2)
H13C	1.3731 (9)	0.6794 (9)	0.4073 (4)	0.056*
C135	1.2131 (9)	0.7633 (9)	0.4486 (4)	0.051 (2)
H13D	1.2592 (9)	0.7782 (9)	0.4905 (4)	0.061*
C136	1.0777 (9)	0.8024 (8)	0.4371 (4)	0.044 (2)
H13E	1.0322 (9)	0.8429 (8)	0.4713 (4)	0.053*
C211	0.4530 (7)	0.6108 (8)	0.1429 (3)	0.031 (2)
C212	0.3752 (8)	0.7072 (9)	0.1699 (4)	0.046 (2)
H21A	0.4239 (8)	0.7868 (9)	0.1941 (4)	0.056*
C213	0.2261 (9)	0.6873 (10)	0.1615 (4)	0.053 (2)
H21B	0.1737 (9)	0.7542 (10)	0.1794 (4)	0.063*
C214	0.1535 (9)	0.5695 (10)	0.1270 (4)	0.052 (2)
H21C	0.0519 (9)	0.5556 (10)	0.1217 (4)	0.063*
C215	0.2291 (9)	0.4743 (9)	0.1009 (4)	0.052 (2)
H21D	0.1797 (9)	0.3939 (9)	0.0775 (4)	0.062*
C216	0.3801 (8)	0.4942 (8)	0.1083 (4)	0.039 (2)
H21E	0.4319 (8)	0.4278 (8)	0.0896 (4)	0.047*
C221	0.6960 (8)	0.7504 (7)	0.0808 (3)	0.032 (2)
C222	0.8413 (8)	0.7708 (8)	0.0631 (4)	0.041 (2)
H22A	0.9118 (8)	0.7336 (8)	0.0884 (4)	0.049*
C223	0.8800 (9)	0.8448 (8)	0.0091 (4)	0.048 (2)
H22B	0.9769 (9)	0.8576 (8)	−0.0025 (4)	0.057*
C224	0.7796 (11)	0.8995 (9)	−0.0275 (4)	0.054 (2)
H22C	0.8072 (11)	0.9500 (9)	−0.0643 (4)	0.065*
C225	0.6368 (10)	0.8816 (9)	−0.0109 (4)	0.053 (2)
H22D	0.5674 (10)	0.9198 (9)	−0.0365 (4)	0.064*
C226	0.5963 (8)	0.8075 (8)	0.0431 (4)	0.041 (2)
H22E	0.4991 (8)	0.7958 (8)	0.0542 (4)	0.049*
C231	0.7197 (7)	0.4846 (7)	0.1347 (3)	0.030 (2)
C232	0.7160 (8)	0.4276 (8)	0.0710 (4)	0.038 (2)
H23A	0.6798 (8)	0.4752 (8)	0.0362 (4)	0.045*
C233	0.7651 (9)	0.3018 (9)	0.0592 (4)	0.047 (2)
H23B	0.7614 (9)	0.2635 (9)	0.0162 (4)	0.056*
C234	0.8191 (9)	0.2319 (8)	0.1089 (4)	0.049 (2)
H23C	0.8509 (9)	0.1453 (8)	0.1001 (4)	0.058*
C235	0.8273 (9)	0.2879 (9)	0.1719 (4)	0.047 (2)
H23D	0.8663 (9)	0.2405 (9)	0.2061 (4)	0.057*
C236	0.7778 (8)	0.4141 (8)	0.1845 (4)	0.038 (2)
H23E	0.7838 (8)	0.4524 (8)	0.2276 (4)	0.046*
O1	0.4270 (11)	1.0129 (11)	0.2580 (5)	0.118 (3)
C1	0.3183 (18)	1.0605 (18)	0.2881 (7)	0.118 (5)
H1A	0.3458 (18)	1.0785 (18)	0.3347 (7)	0.142*
H1B	0.2321 (18)	0.9882 (18)	0.2840 (7)	0.142*
C2	0.284 (2)	1.189 (2)	0.2604 (13)	0.165 (8)
H2A	0.181 (2)	1.180 (2)	0.2464 (13)	0.198*
H2B	0.309 (2)	1.270 (2)	0.2916 (13)	0.198*
C3	0.374 (4)	1.2021 (18)	0.2057 (8)	0.191 (13)
H3A	0.315 (4)	1.2049 (18)	0.1652 (8)	0.230*

H3B	0.444 (4)	1.2893 (18)	0.2107 (8)	0.230*
C4	0.445 (2)	1.088 (2)	0.2032 (8)	0.148 (7)
H4A	0.549 (2)	1.120 (2)	0.1988 (8)	0.177*
H4B	0.409 (2)	1.027 (2)	0.1647 (8)	0.177*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.0264 (3)	0.0272 (3)	0.0264 (3)	0.0046 (2)	−0.0011 (2)	−0.0024 (2)
Br1	0.0665 (6)	0.0507 (6)	0.0526 (6)	−0.0012 (4)	−0.0003 (4)	0.0020 (4)
Br2	0.0441 (5)	0.0534 (5)	0.0494 (5)	0.0030 (4)	0.0068 (4)	−0.0018 (4)
P1	0.0288 (10)	0.0295 (10)	0.0287 (10)	0.0070 (8)	−0.0008 (8)	−0.0020 (8)
P2	0.0245 (9)	0.0311 (10)	0.0274 (9)	0.0054 (8)	−0.0003 (7)	−0.0024 (8)
C111	0.038 (4)	0.030 (4)	0.026 (4)	0.009 (3)	0.001 (3)	−0.002 (3)
C112	0.052 (5)	0.039 (5)	0.046 (5)	0.015 (4)	0.001 (4)	0.002 (4)
C113	0.074 (7)	0.052 (6)	0.054 (5)	0.034 (5)	0.015 (5)	0.015 (4)
C114	0.111 (9)	0.029 (5)	0.049 (5)	0.019 (5)	0.021 (6)	0.001 (4)
C115	0.076 (7)	0.041 (5)	0.067 (6)	−0.005 (5)	−0.002 (5)	−0.008 (5)
C116	0.044 (5)	0.041 (5)	0.051 (5)	0.007 (4)	−0.007 (4)	−0.010 (4)
C121	0.028 (4)	0.039 (4)	0.028 (4)	0.004 (3)	−0.004 (3)	−0.001 (3)
C122	0.039 (4)	0.042 (5)	0.043 (5)	0.010 (4)	0.002 (4)	−0.006 (4)
C123	0.045 (5)	0.070 (6)	0.041 (5)	0.015 (4)	0.009 (4)	−0.009 (4)
C124	0.043 (5)	0.078 (7)	0.039 (5)	−0.004 (5)	0.007 (4)	0.007 (5)
C125	0.058 (6)	0.052 (5)	0.049 (5)	−0.003 (4)	0.001 (4)	0.012 (4)
C126	0.054 (5)	0.036 (5)	0.042 (5)	0.005 (4)	0.007 (4)	0.001 (4)
C131	0.027 (4)	0.028 (4)	0.039 (4)	0.007 (3)	−0.001 (3)	0.003 (3)
C132	0.036 (4)	0.048 (5)	0.033 (4)	0.011 (4)	0.002 (3)	−0.002 (3)
C133	0.046 (5)	0.058 (5)	0.045 (5)	0.022 (4)	0.014 (4)	0.005 (4)
C134	0.035 (4)	0.058 (5)	0.052 (5)	0.018 (4)	0.005 (4)	0.015 (4)
C135	0.047 (5)	0.065 (6)	0.040 (5)	0.015 (4)	−0.013 (4)	−0.003 (4)
C136	0.049 (5)	0.051 (5)	0.035 (4)	0.022 (4)	−0.006 (4)	−0.008 (4)
C211	0.029 (4)	0.040 (4)	0.026 (4)	0.010 (3)	0.000 (3)	0.003 (3)
C212	0.033 (4)	0.063 (6)	0.043 (5)	0.013 (4)	−0.001 (4)	−0.017 (4)
C213	0.047 (5)	0.078 (7)	0.043 (5)	0.036 (5)	0.013 (4)	0.004 (5)
C214	0.028 (4)	0.070 (6)	0.058 (6)	0.003 (4)	0.000 (4)	0.014 (5)
C215	0.035 (5)	0.047 (5)	0.069 (6)	0.000 (4)	−0.009 (4)	−0.004 (4)
C216	0.037 (4)	0.036 (4)	0.043 (4)	0.003 (3)	−0.002 (3)	0.001 (4)
C221	0.036 (4)	0.025 (4)	0.033 (4)	0.002 (3)	0.002 (3)	−0.006 (3)
C222	0.035 (4)	0.043 (5)	0.044 (5)	0.002 (3)	0.004 (4)	0.003 (4)
C223	0.048 (5)	0.042 (5)	0.052 (5)	0.001 (4)	0.013 (4)	0.000 (4)
C224	0.078 (7)	0.044 (5)	0.037 (5)	−0.007 (5)	0.015 (5)	0.005 (4)
C225	0.066 (6)	0.054 (5)	0.040 (5)	0.011 (5)	−0.006 (4)	0.015 (4)
C226	0.039 (4)	0.045 (5)	0.040 (4)	0.010 (4)	−0.001 (4)	0.002 (4)
C231	0.026 (4)	0.031 (4)	0.035 (4)	0.004 (3)	0.005 (3)	0.002 (3)
C232	0.037 (4)	0.047 (5)	0.031 (4)	0.014 (4)	0.002 (3)	−0.001 (3)
C233	0.054 (5)	0.049 (5)	0.041 (5)	0.018 (4)	0.009 (4)	−0.009 (4)
C234	0.058 (5)	0.038 (5)	0.056 (6)	0.020 (4)	0.021 (4)	0.006 (4)
C235	0.058 (5)	0.047 (5)	0.043 (5)	0.025 (4)	0.013 (4)	0.015 (4)

C236	0.042 (4)	0.041 (4)	0.034 (4)	0.015 (4)	0.009 (3)	0.002 (3)
O1	0.108 (7)	0.140 (9)	0.103 (7)	0.014 (6)	0.007 (6)	−0.006 (7)
C1	0.138 (14)	0.129 (13)	0.090 (10)	0.008 (11)	0.047 (10)	0.021 (9)
C2	0.114 (13)	0.115 (15)	0.27 (3)	0.011 (11)	0.043 (16)	0.017 (17)
C3	0.41 (4)	0.074 (11)	0.089 (12)	0.021 (17)	−0.027 (17)	0.033 (9)
C4	0.199 (19)	0.128 (15)	0.108 (13)	−0.023 (14)	0.064 (13)	0.021 (11)

Geometric parameters (Å, °)

Pd1—P1	2.336 (2)	C134—C135	1.368 (11)
Pd1—P2	2.337 (2)	C135—C136	1.387 (11)
Pd1—Br1	2.4148 (13)	C211—C216	1.373 (10)
Pd1—Br2	2.4169 (13)	C211—C212	1.379 (10)
P1—C111	1.815 (7)	C212—C213	1.380 (11)
P1—C131	1.823 (7)	C213—C214	1.380 (12)
P1—C121	1.830 (7)	C214—C215	1.350 (12)
P2—C211	1.823 (7)	C215—C216	1.397 (11)
P2—C231	1.824 (7)	C221—C226	1.374 (10)
P2—C221	1.831 (7)	C221—C222	1.411 (10)
C111—C112	1.380 (10)	C222—C223	1.374 (11)
C111—C116	1.381 (10)	C223—C224	1.355 (12)
C112—C113	1.375 (12)	C224—C225	1.382 (12)
C113—C114	1.380 (14)	C225—C226	1.378 (11)
C114—C115	1.356 (14)	C231—C236	1.383 (10)
C115—C116	1.396 (12)	C231—C232	1.394 (10)
C121—C122	1.372 (10)	C232—C233	1.375 (11)
C121—C126	1.382 (10)	C233—C234	1.360 (11)
C122—C123	1.397 (11)	C234—C235	1.376 (11)
C123—C124	1.369 (12)	C235—C236	1.382 (11)
C124—C125	1.351 (12)	O1—C1	1.34 (2)
C125—C126	1.380 (11)	O1—C4	1.36 (2)
C131—C132	1.384 (10)	C1—C2	1.45 (2)
C131—C136	1.389 (10)	C2—C3	1.44 (2)
C132—C133	1.380 (11)	C3—C4	1.36 (2)
C133—C134	1.375 (11)		
P1—Pd1—P2	176.06 (6)	C136—C131—P1	121.2 (5)
P1—Pd1—Br1	86.82 (6)	C133—C132—C131	120.4 (7)
P2—Pd1—Br1	92.18 (6)	C134—C133—C132	120.6 (7)
P1—Pd1—Br2	92.76 (6)	C135—C134—C133	119.4 (7)
P2—Pd1—Br2	88.12 (6)	C134—C135—C136	120.7 (7)
Br1—Pd1—Br2	178.09 (4)	C135—C136—C131	120.1 (7)
C111—P1—C131	108.0 (3)	C216—C211—C212	119.1 (7)
C111—P1—C121	103.6 (3)	C216—C211—P2	123.0 (5)
C131—P1—C121	102.2 (3)	C212—C211—P2	117.9 (6)
C111—P1—Pd1	111.8 (2)	C211—C212—C213	120.3 (8)
C131—P1—Pd1	110.6 (2)	C214—C213—C212	120.3 (8)
C121—P1—Pd1	119.7 (2)	C215—C214—C213	119.7 (7)
C211—P2—C231	107.7 (3)	C214—C215—C216	120.6 (8)
C211—P2—C221	102.2 (3)	C211—C216—C215	120.1 (7)

C231—P2—C221	102.8 (3)	C226—C221—C222	118.2 (7)
C211—P2—Pd1	113.3 (2)	C226—C221—P2	123.5 (6)
C231—P2—Pd1	111.5 (2)	C222—C221—P2	118.3 (6)
C221—P2—Pd1	118.3 (2)	C223—C222—C221	120.2 (7)
C112—C111—C116	119.3 (7)	C224—C223—C222	120.4 (8)
C112—C111—P1	117.5 (6)	C223—C224—C225	120.4 (8)
C116—C111—P1	123.3 (6)	C226—C225—C224	119.8 (8)
C113—C112—C111	120.8 (8)	C221—C226—C225	121.0 (8)
C112—C113—C114	119.9 (8)	C236—C231—C232	118.5 (6)
C115—C114—C113	119.8 (8)	C236—C231—P2	120.3 (5)
C114—C115—C116	120.9 (9)	C232—C231—P2	121.2 (5)
C111—C116—C115	119.3 (8)	C233—C232—C231	120.0 (7)
C122—C121—C126	118.5 (7)	C234—C233—C232	120.9 (7)
C122—C121—P1	122.5 (6)	C233—C234—C235	120.1 (7)
C126—C121—P1	119.0 (5)	C234—C235—C236	119.6 (7)
C121—C122—C123	119.7 (8)	C235—C236—C231	120.8 (7)
C124—C123—C122	121.1 (8)	C1—O1—C4	105.8 (13)
C125—C124—C123	119.0 (8)	O1—C1—C2	111.5 (13)
C124—C125—C126	120.8 (8)	C3—C2—C1	101.9 (15)
C125—C126—C121	120.9 (8)	C4—C3—C2	107.9 (14)
C132—C131—C136	118.7 (6)	C3—C4—O1	111.1 (15)
C132—C131—P1	120.1 (5)		

trans-dichlorobis-(triphenylphosphine)palladium(II)

Crystal data

$[PdCl_2(C_{18}H_{15}P)_2]\cdot 2CHCl_3$	$Z = 1$
$M_r = 940.58$	$F_{000} = 472$
Triclinic, $P\bar{1}$	$D_x = 1.567$ Mg m^{-3}
$a = 7.880$ (2) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 11.201$ (2) Å	Cell parameters from 25 reflections
$c = 12.043$ (2) Å	$\theta = 3.7$–7.7º
$\alpha = 89.19$ (3)º	$\mu = 1.11$ mm^{-1}
$\beta = 76.76$ (3)º	$T = 223$ (2) K
$\gamma = 74.65$ (3)º	Chunk, yellow
$V = 996.6$ (4) Å^3	0.40 × 0.40 × 0.40 mm

Data collection

Rigaku AFC5-S diffractometer	$R_{int} = 0.022$
Radiation source: normal-focus sealed tube	$\theta_{max} = 23.9$º
Monochromator: graphite	$\theta_{min} = 2.5$º
$T = 223(2)$ K	$h = -8\rightarrow 8$
ω–scans	$k = -12\rightarrow 0$
Absorption correction: ψ scans	$l = -13\rightarrow 13$
$T_{min} = 0.504$, $T_{max} = 0.641$	3 standard reflections

2828 measured reflections
2647 independent reflections
2420 reflections with $I > 2\sigma(I)$
every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.140$
$S = 1.03$
2647 reflections
223 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
Calculated $w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 5.4021P]$ where $P = (F_o^2 + 2F_c^2)/3$?
$\Delta\rho_{max} = 1.81$ e Å^{-3}
$\Delta\rho_{min} = -1.41$ e Å^{-3}
Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement on F^2 for ALL reflections except for 0 with very negative F^2 or flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating _refine_ls_R_factor_obs *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R–factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Pd1	0.0000	0.0000	0.0000	0.0276 (2)
Cl1	−0.0721 (2)	0.17130 (15)	−0.10408 (13)	0.0438 (4)
P1	−0.0572 (2)	−0.12980 (14)	−0.12970 (12)	0.0281 (4)
C11	−0.1786 (8)	−0.2372 (6)	−0.0592 (5)	0.0313 (13)
C12	−0.3318 (9)	−0.1896 (7)	0.0280 (6)	0.046 (2)
H12	−0.3658 (9)	−0.1050 (7)	0.0503 (6)	0.080*
C13	−0.4336 (10)	−0.2650 (8)	0.0815 (6)	0.054 (2)
H13	−0.5395 (10)	−0.2314 (8)	0.1381 (6)	0.080*
C14	−0.3807 (11)	−0.3893 (8)	0.0523 (6)	0.058 (2)
H14	−0.4489 (11)	−0.4413 (8)	0.0903 (6)	0.080*
C15	−0.2286 (11)	−0.4384 (7)	−0.0321 (6)	0.053 (2)
H15	−0.1930 (11)	−0.5238 (7)	−0.0519 (6)	0.080*
C16	−0.1275 (9)	−0.3626 (6)	−0.0882 (5)	0.040 (2)
H16	−0.0237 (9)	−0.3964 (6)	−0.1463 (5)	0.080*

C21	0.1526 (8)	−0.2231 (5)	−0.2205 (5)	0.0297 (13)
C22	0.3079 (9)	−0.2590 (6)	−0.1793 (5)	0.040 (2)
H22	0.3044 (9)	−0.2358 (6)	−0.1039 (5)	0.080*
C23	0.4686 (9)	−0.3289 (7)	−0.2486 (6)	0.050 (2)
H23	0.5727 (9)	−0.3543 (7)	−0.2194 (6)	0.080*
C24	0.4766 (9)	−0.3613 (6)	−0.3596 (6)	0.047 (2)
H24	0.5865 (9)	−0.4071 (6)	−0.4069 (6)	0.080*
C25	0.3236 (10)	−0.3266 (7)	−0.4010 (6)	0.051 (2)
H25	0.3290 (10)	−0.3486 (7)	−0.4771 (6)	0.080*
C26	0.1610 (9)	−0.2595 (6)	−0.3321 (5)	0.041 (2)
H26	0.0561 (9)	−0.2385 (6)	−0.3607 (5)	0.080*
C31	−0.1896 (8)	−0.0554 (5)	−0.2300 (5)	0.0304 (13)
C32	−0.3613 (8)	−0.0650 (6)	−0.2265 (5)	0.0388 (15)
H32	−0.4134 (8)	−0.1129 (6)	−0.1711 (5)	0.080*
C33	−0.4590 (10)	−0.0056 (7)	−0.3029 (7)	0.053 (2)
H33	−0.5764 (10)	−0.0128 (7)	−0.2988 (7)	0.080*
C34	−0.3828 (10)	0.0644 (7)	−0.3852 (6)	0.049 (2)
H34	−0.4483 (10)	0.1051 (7)	−0.4373 (6)	0.080*
C35	−0.2119 (9)	0.0740 (6)	−0.3907 (5)	0.043 (2)
H35	−0.1595 (9)	0.1209 (6)	−0.4471 (5)	0.080*
C36	−0.1157 (9)	0.0153 (6)	−0.3137 (5)	0.0371 (15)
H36	0.0013 (9)	0.0233 (6)	−0.3178 (5)	0.080*
Cl11	−0.1256 (5)	−0.5939 (4)	−0.3365 (4)	0.152 (2)
Cl12	0.1755 (3)	−0.7829 (2)	−0.4636 (2)	0.0683 (6)
Cl13	0.2364 (5)	−0.6022 (3)	−0.3243 (3)	0.1211 (12)
C1	0.0880 (10)	−0.6846 (7)	−0.3415 (6)	0.055 (2)
H1	0.0777 (10)	−0.7386 (7)	−0.2758 (6)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.0368 (4)	0.0308 (4)	0.0161 (3)	−0.0107 (3)	−0.0062 (3)	−0.0005 (2)
Cl1	0.0685 (11)	0.0396 (9)	0.0289 (8)	−0.0175 (8)	−0.0192 (8)	0.0071 (7)
P1	0.0344 (8)	0.0316 (8)	0.0188 (8)	−0.0097 (7)	−0.0059 (6)	−0.0021 (6)
C11	0.038 (3)	0.037 (3)	0.022 (3)	−0.013 (3)	−0.009 (3)	0.003 (3)
C12	0.045 (4)	0.049 (4)	0.039 (4)	−0.012 (3)	−0.001 (3)	−0.005 (3)
C13	0.047 (4)	0.073 (6)	0.042 (4)	−0.026 (4)	0.001 (3)	0.006 (4)
C14	0.071 (5)	0.076 (6)	0.046 (4)	−0.051 (5)	−0.016 (4)	0.022 (4)
C15	0.070 (5)	0.044 (4)	0.056 (5)	−0.025 (4)	−0.022 (4)	0.007 (4)
C16	0.047 (4)	0.038 (4)	0.038 (4)	−0.015 (3)	−0.010 (3)	0.000 (3)
C21	0.036 (3)	0.030 (3)	0.022 (3)	−0.010 (3)	−0.004 (2)	0.000 (2)
C22	0.041 (4)	0.049 (4)	0.029 (3)	−0.012 (3)	−0.006 (3)	−0.005 (3)
C23	0.037 (4)	0.054 (4)	0.056 (5)	−0.005 (3)	−0.012 (3)	−0.003 (4)
C24	0.043 (4)	0.046 (4)	0.041 (4)	−0.003 (3)	0.005 (3)	0.000 (3)
C25	0.065 (5)	0.049 (4)	0.025 (3)	−0.003 (4)	−0.001 (3)	−0.001 (3)
C26	0.048 (4)	0.045 (4)	0.026 (3)	−0.001 (3)	−0.011 (3)	−0.006 (3)
C31	0.041 (3)	0.030 (3)	0.021 (3)	−0.008 (3)	−0.011 (3)	−0.006 (2)
C32	0.039 (4)	0.043 (4)	0.034 (3)	−0.011 (3)	−0.008 (3)	−0.002 (3)

C33	0.041 (4)	0.063 (5)	0.057 (5)	−0.005 (4)	−0.022 (4)	−0.005 (4)
C34	0.055 (5)	0.052 (4)	0.038 (4)	0.001 (4)	−0.022 (3)	−0.001 (3)
C35	0.055 (4)	0.046 (4)	0.026 (3)	−0.008 (3)	−0.013 (3)	0.002 (3)
C36	0.046 (4)	0.037 (4)	0.028 (3)	−0.008 (3)	−0.009 (3)	−0.004 (3)
Cl11	0.113 (2)	0.151 (3)	0.154 (3)	0.062 (2)	−0.064 (2)	−0.076 (3)
Cl12	0.0605 (12)	0.0749 (14)	0.0674 (13)	−0.0184 (10)	−0.0086 (10)	−0.0278 (11)
Cl13	0.123 (2)	0.142 (3)	0.106 (2)	−0.062 (2)	−0.008 (2)	−0.065 (2)
C1	0.065 (5)	0.054 (5)	0.038 (4)	−0.012 (4)	−0.001 (4)	−0.002 (3)

Geometric parameters (Å, °)

Pd1—Cl1^{i}	2.293 (2)	C21—C26	1.393 (8)
Pd1—Cl1	2.293 (2)	C22—C23	1.387 (9)
Pd1—P1^{i}	2.343 (2)	C23—C24	1.373 (10)
Pd1—P1	2.343 (2)	C24—C25	1.369 (10)
P1—C11	1.814 (6)	C25—C26	1.384 (10)
P1—C21	1.819 (6)	C31—C32	1.376 (9)
P1—C31	1.824 (6)	C31—C36	1.391 (9)
C11—C16	1.380 (9)	C32—C33	1.384 (10)
C11—C12	1.392 (9)	C33—C34	1.383 (11)
C12—C13	1.370 (10)	C34—C35	1.366 (10)
C13—C14	1.370 (11)	C35—C36	1.381 (9)
C14—C15	1.371 (11)	Cl11—C1	1.709 (8)
C15—C16	1.381 (10)	Cl12—C1	1.741 (7)
C21—C22	1.385 (9)	Cl13—C1	1.718 (8)
Cl1^{i}—Pd1—Cl1	180.0	C22—C21—C26	118.7 (6)
Cl1^{i}—Pd1—P1^{i}	92.88 (6)	C22—C21—P1	120.2 (4)
Cl1—Pd1—P1^{i}	87.12 (6)	C26—C21—P1	121.1 (5)
Cl1^{i}—Pd1—P1	87.12 (6)	C21—C22—C23	120.4 (6)
Cl1—Pd1—P1	92.88 (6)	C24—C23—C22	120.5 (6)
P1^{i}—Pd1—P1	180.0	C25—C24—C23	119.6 (6)
C11—P1—C21	106.7 (3)	C24—C25—C26	120.7 (6)
C11—P1—C31	104.3 (3)	C25—C26—C21	120.1 (6)
C21—P1—C31	104.2 (3)	C32—C31—C36	117.9 (6)
C11—P1—Pd1	112.5 (2)	C32—C31—P1	123.1 (5)
C21—P1—Pd1	111.1 (2)	C36—C31—P1	119.1 (5)
C31—P1—Pd1	117.2 (2)	C31—C32—C33	121.5 (6)
C16—C11—C12	118.8 (6)	C34—C33—C32	119.6 (7)
C16—C11—P1	123.4 (5)	C35—C34—C33	119.8 (6)
C12—C11—P1	117.8 (5)	C34—C35—C36	120.3 (6)
C13—C12—C11	120.7 (7)	C35—C36—C31	121.0 (6)
C14—C13—C12	119.9 (7)	Cl11—C1—Cl13	113.6 (5)
C13—C14—C15	120.3 (7)	Cl11—C1—Cl12	112.5 (4)
C14—C15—C16	120.1 (7)	Cl13—C1—Cl12	111.0 (4)
C11—C16—C15	120.2 (6)		

Symmetry codes: (i) −*x*, −*y*, −*z*.