ics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

References

Antti, C. J. & Lundberg, B. K. S. (1971). *Acta Chem. Scand.* **25**, 1758–1766.
Fargher, R. G. & Pyman, L. I. (1919). *J. Chem. Soc.* **115**, 217–260.
Hester, C. A., Baughman, R. G. & Collier, H. L. (1997). *Polyhedron*, **16**, 2893–2895.
Misra, T. K., Das, D., Sinha, C., Ghosh, P. & Pal, C. K. (1998). *Inorg. Chem.* **37**, 1672–1678.
Sheldrick, G. M. (1996). *SADABS. Program for Empirical Absorption Correction of Area Detector Data*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL. Structure Determination Programs*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT. Area Detector Control and Integration Software*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

Dibromo[propane-1,3-diylbis(diphenylphosphine)-*P*,*P*′]palladium(II)

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Abstract

In the title compound, *cis*-[PdBr₂{Ph₂P(CH₂)₃PPh₂}], the Pd atom is coordinated to the diphosphine ligand through the P atoms and to two bromo ligands in a slightly distorted square-planar arrangement. The displacements of the P atoms from the PdBr₂ plane are

0.066 and −0.349 Å, a consequence of the steric strain caused by the eclipsing of bonds in the alkyl chain of the phosphine.

Comment

It is well known that palladium(II) frequently forms square-planar complexes in which the metal centre is coordinated to two P atoms of tertiary phosphine ligands and to two mutually *cis* halide ligands. Many crystal structures have been reported in which the halogen is chlorine, but to the best of our knowledge, only three structures of this type are known with bromine (Wilson *et al.*, 1996; Peters *et al.*, 1984).

The crystal structure of the title complex, (I), consists of equivalent discrete *cis*-[PdBr₂{Ph₂P(CH₂)₃PPh₂}] molecules. The coordination around palladium is

(I)
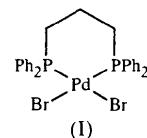
slightly distorted square planar, with the most noticeable distortion in the P2—Pd1—Br2 angle of 88.04 (5)°. The remaining angles around palladium are closer to the ideal value of 90° for a square-planar geometry and the sum of the angles about the metal centre is 360.28°. The planar coordination is slightly distorted, with displacements of the P atoms from the PdBr₂ plane of 0.066 (P1) and −0.349 Å (P2). This is probably a consequence of the steric strain caused by the appreciable eclipsing of bonds in the alkyl chain of the phosphine required if both P atoms are to bond to the same Pd atom. The strain is also indicated in the bond angles in the alkyl chain,
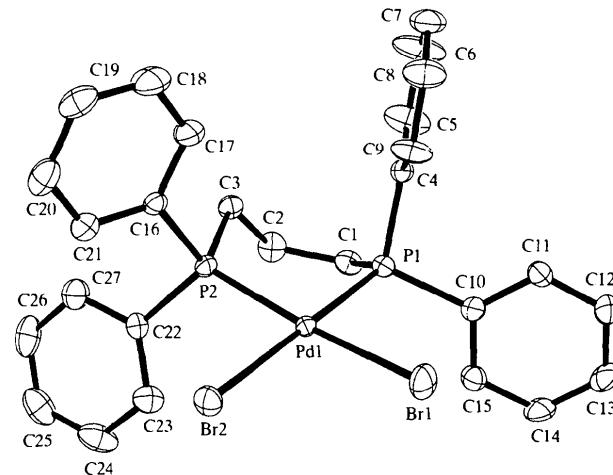

Fig. 1. View of the title compound with 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

which show large deviations from ideal values (Serffen & Palenik, 1976). The mean deviation of atoms P1, P2, Pd1, Br1 and Br2 from their mean plane is 0.0993 Å. The Pd1—P1 and Pd2—P2 bond lengths of 2.2557 (16) and 2.2535 (16) Å, respectively, are similar to the values found in related palladium(II) complexes (Singh et al., 1995; Vila et al., 1996). The Pd—Br bond lengths of 2.4744 (9) and 2.4767 (9) Å are similar to those found in other complexes with Br trans to P (Wilson et al., 1996; Peters et al., 1984; Butler et al., 1997; Marcoux et al., 1997), but longer than those found when Br is trans to N or Br (Smeets et al., 1997; Casares et al., 1997), consistent with the strong trans influence of phosphines.

Experimental

To a vigorously stirred emulsion of PdCl$_2$ (0.2 g, 1.13 mmol) dissolved in a saturated aqueous solution of potassium bromide (10 ml) and dichloromethane (10 ml), propane-1,3-diylbis(diphenylphosphine) (0.46 g, 1.11 mmol) was added. The mixture was stirred overnight and the organic phase removed using a separatory funnel, dried over anhydrous sodium sulfate and gravity filtered. The solvent was removed in vacuo. The resulting pale-yellow solid was recrystallized from dichloromethane/hexane to give pale-yellow crystals of the title compound (yield 75%).

Crystal data
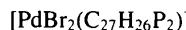
[PdBr$_2$(C$_{27}$H$_{26}$P$_2$)]
$M_r = 678.64$
Triclinic
$P\bar{1}$
$a = 8.6829\,(13)$ Å
$b = 10.7510\,(16)$ Å
$c = 14.420\,(2)$ Å
$\alpha = 88.617\,(13)°$
$\beta = 80.841\,(11)°$
$\gamma = 73.689\,(11)°$
$V = 1275.1\,(3)$ Å^3
$Z = 2$
$D_x = 1.768$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.5–18.0°$
$\mu = 4.001$ mm^{-1}
$T = 293\,(2)$ K
Prism
$0.25 \times 0.20 \times 0.20$ mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction:
  ψ scan (North et al., 1968)
  $T_{min} = 0.337$, $T_{max} = 0.449$
5526 measured reflections
5173 independent reflections

2965 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 26.3°$
$h = 0 \rightarrow 10$
$k = -12 \rightarrow 13$
$l = -17 \rightarrow 17$
2 standard reflections
  frequency: 120 min
  intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.095$
$S = 0.985$
5173 reflections
289 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0378P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.564$ e Å^{-3}
$\Delta\rho_{min} = -1.100$ e Å^{-3}
Extinction correction: none
Scattering factors from International Tables for Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Pd1—P2	2.2535 (16)	Pd1—Br1	2.4744 (9)
Pd1—P1	2.2557 (16)	Pd1—Br2	2.4767 (9)
P2—Pd1—P1	90.80 (6)	C2—C1—P1	118.3 (4)
P1—Pd1—Br1	91.22 (5)	C1—C2—C3	116.6 (5)
P2—Pd1—Br2	88.04 (5)	C2—C3—P2	111.3 (4)
Br1—Pd1—Br2	90.22 (3)		

Data collection: CAD-4 Software (Enraf–Nonius, 1989). Cell refinement: CAD-4 Software. Data reduction: CAD-4 SDP (Frenz, 1978). Program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a). Program(s) used to refine structure: SHELXL97 (Sheldrick, 1997b). Molecular graphics: SHELXTL (Sheldrick, 1995).

References

Butler, I. R., Hobson, L. J., Coles, S. J., Hursthouse, M. B. & Malik, K. M. (1997). J. Organomet. Chem. 540, 27–32.

Casares, J. A., Espinet, P., Martinez-Ilarduya, J. M. & Lin, Y. (1997). Organometallics, 16, 770–780.

Enraf–Nonius (1989). CAD-4 Software. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Frenz, B. A. (1978). The Enraf–Nonius CAD-4 SDP – a Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution. Computing in Crystallography, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft University Press.

Marcoux, J. F., Wagaw, S. & Buchwald, S. L. (1997). J. Org. Chem. 62, 1568–1574.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). Acta Cryst. A24, 351–359.

Peters, K., Peters, E. M., von Schnering, H. G. & Abicht, H. P. (1984). Z. Kristallogr. 168, 149–151.

Serffen, W. L. & Palenik, G. J. (1976). Inorg. Chem. 15, 2432–2438.

Sheldrick, G. M. (1995). SHELXTL. Structure Determination Programs. Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997a). SHELXS97. Program for the Solution of Crystal Structures. University of Göttingen, Germany.

Sheldrick, G. M. (1997b). SHELXL97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany.

Singh, S., Jha, N. K., Narula, P. & Singh, T. P. (1995). Acta Cryst. C51, 593–595.

Smeets, W. J. J., Spek, A. L., Hoare, J. L., Canty, A. J., Hovestad, N. & van Koten, G. (1997). Acta Cryst. C53, 1045–1047.

Vila, J. M., Gayoso, M., López-Torres, M., Fernández, J. J., Fernández, A., Ortigueira, J. M., Bailey, N. A. & Adams, H. (1996). J. Organomet. Chem. 511, 129–138.

Wilson, W. L., Fischer, J., Wasylishen, R. E., Eichele, K., Catalano, V. J., Frederick, J. H. & Nelson, J. H. (1996). Inorg. Chem. 35, 1486–1496.