

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

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

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Abstract

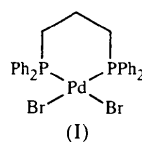
In the title compound, *cis*-[PdBr₂{Ph₂P(CH₂)₃PPh₂}], the Pd atom is coordinated to the diphosphine ligand through the P atoms and to two bromo ligands in a slightly distorted square-planar arrangement. The displacements of the P atoms from the PdBr₂ plane are

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



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,

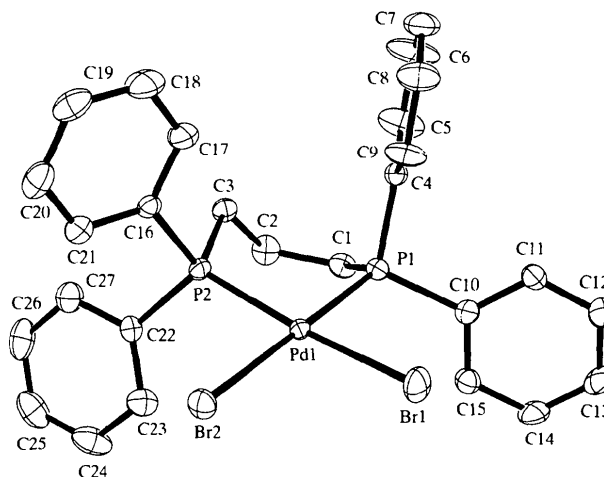


Fig. 1. View of the title compound with 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

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 PdCl₂ (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

[PdBr₂(C₂₇H₂₆P₂)]

M_r = 678.64

Triclinic

P $\bar{1}$

a = 8.6829 (13) Å

b = 10.7510 (16) Å

c = 14.420 (2) Å

α = 88.617 (13)°

β = 80.841 (11)°

γ = 73.689 (11)°

V = 1275.1 (3) Å³

Z = 2

D_x = 1.768 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 7.5–18.0°

μ = 4.001 mm⁻¹

T = 293 (2) K

Prism

0.25 × 0.20 × 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 > 2σ(*I*)

R_{int} = 0.039

$\theta_{\max}$ = 26.3°

h = 0 → 10

k = -12 → 13

l = -17 → 17

2 standard reflections

frequency: 120 min

intensity decay: <1%

Refinement

Refinement on *F*²

R [*F*² > 2σ(*F*²)] = 0.039

wR(*F*²) = 0.095

$w = 1/[\sigma^2(F_o^2) + (0.0378P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

(Δ/σ)_{max} = 0.001

S = 0.985

5173 reflections

289 parameters

H-atom parameters constrained

$\Delta\rho_{\max} = 0.564 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -1.100 \text{ e } \text{\AA}^{-3}$

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, 1997*a*). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*b*). Molecular graphics: *SHELXTL* (Sheldrick, 1995).

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