C3*B*	0.7345 (6)	0.3999 (10)	0.8736 (5)	0.080 (2)
C2*B*	0.6162 (5)	0.4455 (6)	0.7907 (4)	0.0611 (12)
C1*B*	0.5443 (4)	0.3799 (5)	0.6922 (3)	0.0471 (10)
C11	0.3029 (10)	0.3680 (9)	0.0956 (5)	0.101 (3)
Cl1*A*†	0.2617 (11)	0.2225 (7)	0.0948 (9)	0.090 (2)
Cl1*B*†	0.2736 (18)	0.2336 (14)	0.0806 (12)	0.171 (6)
Cl2*A*†	0.4236 (17)	0.411 (3)	0.0359 (10)	0.225 (12)
Cl2*B*†	0.4335 (18)	0.379 (2)	0.0047 (8)	0.146 (5)
Cl3	0.1442 (4)	0.4410 (3)	0.0357 (3)	0.1446 (12)

† Site occupancy = 0.50.

Table 2. *Selected geometric parameters (Å, °)*

Mo1—O1*A*	1.703 (4)	C7*A*—N1*A*	1.492 (6)
Mo1—O1*B*	1.714 (3)	N1*A*—C1N*A*	1.470 (6)
Mo1—O2*B*	1.946 (3)	N1*A*—C8*A*	1.508 (5)
Mo1—O2*A*	1.945 (3)	C8*A*—C8*B*	1.529 (5)
Mo1—N1*A*	2.400 (4)	C8*B*—N1*B*	1.498 (5)
Mo1—N1*B*	2.413 (4)	N1*B*—C7*B*	1.496 (5)
O2*A*—C1*A*	1.338 (5)	N1*B*—C1N*B*	1.496 (5)
O2*B*—C1*B*	1.349 (6)		
O1*A*—Mo1—O1*B*	107.5 (2)	C1N*A*—N1*A*—C8*A*	113.2 (3)
O1*A*—Mo1—O2*B*	94.5 (2)	C7*A*—N1*A*—C8*A*	110.3 (4)
O1*B*—Mo1—O2*B*	96.9 (2)	C1N*A*—N1*A*—Mo1	107.1 (3)
O1*A*—Mo1—O2*A*	97.7 (2)	C7*A*—N1*A*—Mo1	108.5 (3)
O1*B*—Mo1—O2*A*	94.3 (2)	C8*A*—N1*A*—Mo1	111.9 (2)
O2*B*—Mo1—O2*A*	160.11 (15)	N1*A*—C8*A*—C8*B*	110.2 (3)
O1*A*—Mo1—N1*A*	88.3 (2)	N1*A*—C8*A*—C9*A*	113.0 (3)
O1*B*—Mo1—N1*A*	163.9 (2)	C8*B*—C8*A*—C9*A*	110.6 (3)
O2*B*—Mo1—N1*A*	84.25 (13)	N1*B*—C8*B*—C8*A*	110.1 (3)
O2*A*—Mo1—N1*A*	80.50 (13)	N1*B*—C8*B*—C9*B*	113.1 (4)
O1*A*—Mo1—N1*B*	161.3 (2)	C8*A*—C8*B*—C9*B*	111.6 (4)
O1*B*—Mo1—N1*B*	90.8 (2)	C7*B*—N1*B*—C1N*B*	106.1 (3)
O2*B*—Mo1—N1*B*	79.32 (13)	C7*B*—N1*B*—C8*B*	110.9 (3)
O2*A*—Mo1—N1*B*	84.15 (15)	C1N*B*—N1*B*—C8*B*	112.8 (3)
N1*A*—Mo1—N1*B*	73.55 (12)	C7*B*—N1*B*—Mo1	107.5 (3)
C1*A*—O2*A*—Mo1	140.3 (3)	C1N*B*—N1*B*—Mo1	108.9 (3)
C1*B*—O2*B*—Mo1	139.3 (3)	C8*B*—N1*B*—Mo1	110.5 (2)
N1*A*—C7*A*—C6*A*	115.6 (4)	N1*B*—C7*B*—C6*B*	114.5 (4)
C1N*A*—N1*A*—C7*A*	105.7 (4)		

Data collection: *XSCANS* (Siemens, 1994). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1300). Services for accessing these data are described at the back of the journal.

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Dichloro(η^6-hexamethylbenzene)(pyridine-*N*)ruthenium

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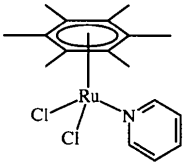
Abstract

The title compound, $[RuCl_2(C_{12}H_{18})(C_5H_5N)]$, crystallizes with two discrete independent molecules per asymmetric unit. In each molecule, the Ru atom displays a 'piano-stool' configuration.

Comment

The title compound was prepared during investigations into the synthesis of terminal imido complexes of ruthenium (Burrell & Steedman, 1995). The ruthenium starting materials for the synthesis of imido complexes are the arene–ruthenium chloride dimers, $[RuCl_2(\eta^6\text{-arene})]_2$, which are poorly soluble in the solvents necessary for the preparation of imido complexes. The pyridine adducts, $[RuCl_2(py)(\eta^6\text{-arene})]$, have been employed in reactions where the dimeric complex gives poor results (Müller, Qiao, Siewing & Westphal, 1993). In an attempt to improve the yields of our imido complexes, we prepared the pyridine (py) adduct $[\mathrm{RuCl_2(py)}(\eta^6\text{-}\mathrm{C_6Me_6})]$, (I).



(I)

Compound (I) crystallizes from benzene in the space group $P2_1/c$ with two independent molecules in the asymmetric unit. The two independent molecules show no significant differences in their structural parameters. However, it should be noted that the Ru—Cl distances on each molecule are statistically different, *e.g.* Ru1—Cl11 2.416 (2) and Ru1—Cl12 2.4361 (14) Å. Three related structures have been reported, namely, the pyridine adduct $[\mathrm{RuCl_2(py)}(\eta^6\text{-}\mathrm{C_6H_3Me_3})]$ (Carter, Davies, Fawcett & Russell, 1993), and $[\mathrm{RuCl_2(py)}(\eta^6\text{-}\mathrm{MeC_6H_4CHMe_2})]$ and $[\mathrm{RuCl_2(py)}(\eta^6\text{-}\mathrm{MeC_{16}H_{16}})]$ (Aronson, Elsegood, Steed & Tocher, 1991). All of these pyridine adducts display similar structural parameters, with all Ru—N distances in the range 2.120 (4)–2.160 (6) Å.

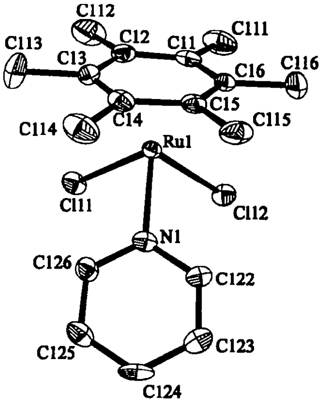


Fig. 1. *ZORTEP* (Zsolnai, 1994*b*) drawing of molecule 1 of (I) showing displacement ellipsoids drawn at the 50% probability level.

Experimental

The title compound was synthesized by the previously described procedure (Müller, Qiao, Siewing & Westphal, 1993; Bennett & Smith, 1974). Crystals were obtained by the slow evaporation of a benzene solution.

Crystal data

$[\mathrm{RuCl_2(C_{12}H_{18})(C_5H_5N)}]$
$M_r = 413.34$
Monoclinic
$P2_1/c$
$a = 14.674\ (6)$ Å
$b = 16.365\ (8)$ Å
$c = 15.233\ (6)$ Å
$\beta = 111.39\ (3)^\circ$
$V = 3406\ (3)$ Å^3
$Z = 8$
$D_x = 1.612$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8$–18°
$\mu = 1.227$ mm^{-1}
$T = 173\ (2)$ K
Block
0.60 × 0.30 × 0.30 mm
Yellow–orange

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.479$, $T_{\max} = 0.692$
6189 measured reflections
5847 independent reflections

4359 reflections with $I > 2\sigma(I)$
$R_{\mathrm{int}} = 0.030$
$\theta_{\max} = 24.97^\circ$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 19$
$l = -17 \rightarrow 16$
3 standard reflections every 100 reflections intensity decay: <2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.095$
$S = 1.049$
5847 reflections
379 parameters
Only H-atom coordinates refined

$w = 1/[\sigma^2(F_o^2) + (0.0371P)^2 + 6.3792P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.017$
$\Delta\rho_{\max} = 0.501$ e Å^{-3}
$\Delta\rho_{\min} = -1.031$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Ru1—N1	2.122 (4)	Ru2—N2	2.120 (4)
Ru1—Cl11	2.416 (2)	Ru2—Cl21	2.407 (2)
Ru1—Cl12	2.4361 (14)	Ru2—Cl22	2.4358 (14)
N1—Ru1—Cl11	86.16 (10)	N2—Ru2—Cl21	86.75 (10)
N1—Ru1—Cl12	86.12 (10)	N2—Ru2—Cl22	84.22 (10)
Cl11—Ru1—Cl12	89.98 (5)	Cl21—Ru2—Cl22	88.45 (5)

A 1 mm collimator was used for this data collection. A one-electron hole is located 0.82 Å from Ru2.

Data collection: *CAD-4/PC Software* (Enraf–Nonius, 1994). Cell refinement: *CAD-4/PC Software*. Data reduction: *XCAD-4B* (Harms, 1994). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XPMA* (Zsolnai, 1994*a*) and *ZORTEP* (Zsolnai, 1994*b*). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1142). Services for accessing these data are described at the back of the journal.

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Di-μ-bromo-bis{[(2,4,6-tris-*tert*-butylphenyl)phosphanediylmethyl-*P*]phenyl-C^2}-dipalladium

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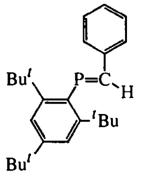
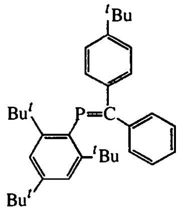
Abstract

The crystal structure of the title compound, $[Pd_2Br_2(C_{25}H_{34}P)_2]$, a new binuclear phospha-alkene compound containing a trivalent P atom shows a centrosymmetric dimeric arrangement. The Pd_2Br_2 core is planar and adopts an irregular diamond shape. The coordination of the Pd atom is square planar. No stacking interactions were observed in the molecular packing.

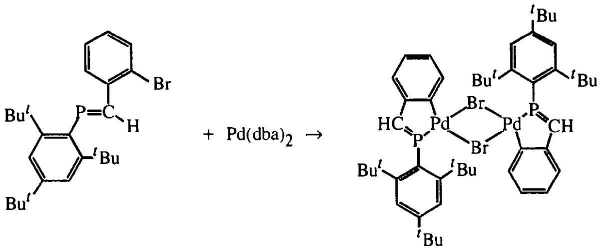
Comment

There is an increasing interest in the chemistry of compounds containing a low coordinated trivalent P atom (Jouaiti, Geoffroy, Terron & Bernardinelli, 1992, 1995; Jouaiti, Geoffroy & Bernardinelli, 1996). A recent communication (Kawanami, Toyota & Yoshifuji, 1996) on the preparation of a novel binuclear complex of a phospha-alkene ligand, $[PdClL']_2$, has prompted us to report another path leading to an analogous complex, $[PdBrL]_2$, and the corresponding crystal structure.


L


L'

Whereas Kawanami *et al.* (1996) obtained $[PdClL']_2$ by reacting *L'* with a Pd^{II} complex, we were able to form $[PdBrL]_2$ by reacting (1) with bis(dibenzylideneacetone)palladium, $[Pd(dba)_2]$, a Pd^0 reagent well known for facilitating oxidative addition on the carbon–halogen bond (Albert, Barro & Granell, 1991).


(1) $[PdBrL]_2$

While the crystallographic analysis of the chlorine complex was unsuccessful in the absence of both a *tert*-butyl group on the metallated phenyl ring and a phenyl group bound to the phospha-alkene C atom (Kawanami *et al.*, 1996), we succeeded in solving the structure of the bromine compound bearing no substituent in these positions. It should be noted that, as shown by ^{31}P NMR, $[PdBrL]_2$ in the presence of a phosphine as Ph_3P or $ArPH_2$ (Ar = 2,4,6-tris-*tert*-butylphenyl) gives rise to the mononuclear complexes $Ph_3PPd(L)Br$ or $Ar(H_2)PPd(L)Br$, respectively.

In $[PdBrL]_2$, the phospha-alkene molecule chelates the palladium(II) ion through orthometallation and coordination to the P atom. Additional coordination of the metal to two Br atoms generates a centrosymmetric dimer. The $PdBr_2$ core has an irregular diamond shape with slightly different metal–bromine bond lengths. This structure is similar to that reported for the chlorine compound. The increase of the palladium–halogen bond lengths and the absence of a phenyl ring on the phospha-alkene C atom does not affect the conformation of the five-membered ring containing the Pd atom. This five-membered ring is planar (maximum deviation of 0.08 Å for the C2 atom) and almost parallel (5.2°) to the Pd_2Br_2 plane. The coordination of the Pd atom is