Fig. 2. Unit-cell packing diagram for $[P(CH_3)(C_6H_5)_3]_2[\{IPd-[CC(=O)(OCH_3)]_4\}_2]$ as viewed with the z axis vertical, the x axis to the right and the y axis to the left.

Related literature. Two related mononuclear structures of palladium metallocycles which contain methyl carboxylate substituents have been reported.

$[Pd\{CC(=O)(OCH_3)\}_4]$ [norbornadiene] was reported by Brown, Itoh, Suzuki, Hirai & Ibers (1978) and $[Pd\{CC(=O)(OCH_3)\}_4]$ $[C_5H_4PPh_3]$ was reported by Pierpont, Downs, Itoh, Nishiyama & Ishii (1976).

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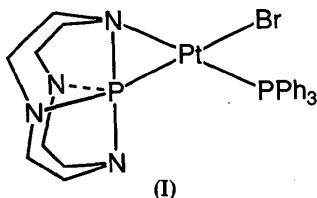
Bromo(η²-cyclenphosphoranido)(triphenylphosphino)platinum(II)

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Abstract. $[PtBr(C_8H_{16}N_4P)(C_{18}H_{15}P)]$, $M_r = 736.5$, monoclinic, $P2_1/n$, $a = 11.651$ (5), $b = 14.301$ (5), $c = 15.527$ (7) Å, $\beta = 93.81$ (4)°, $V = 2581$ (2) Å³, $Z = 4$, $D_x = 1.90$ g cm⁻³, $\lambda(Mo\ K\alpha) = 0.71073$ Å, $\mu(Mo\ K\alpha) = 71.70$ cm⁻¹, $F(000) = 1432$, $T = 295$ K, final $R = 0.043$ for 3399 observed reflections. The cyclenphosphoranido ligand coordinates to the Pt atom through P and one of its N atoms with Pt—P and Pt—N bonds of 2.230 (3) and 2.107 (8) Å, respectively. A Br atom and a triphenylphosphine group complete the distorted square-planar geometry about Pt, with Pt—Br and Pt—P distances of 2.523 (2) and 2.225 (3) Å, respectively.

Experimental. The title compound (I) was prepared by the reaction of the corresponding Pt—Cl complex with bromobutane, accompanied by elimination of chlorobutane.

(I)

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Crystals of the title compound are colorless polyhedra. Unit-cell parameters by least-squares fit of 25 reflections in the range $10 < 2\theta < 25°$. Crystal dimensions $0.20 \times 0.30 \times 0.10$ mm, space group $P2_1/n$ from systematic absences ($0k0$, k odd; $h0l$, $h + l$ odd); automatic Nicolet $R3m/V$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan mode, 4952 reflections measured, 4566 independent reflections in the range $3 < 2\theta < 50°$, $R_{int} = 0.014$, hkl range h $-13\rightarrow13$, k $0\rightarrow17$, l $0\rightarrow18$, 3399 observed reflections with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics; three standard reflections remeasured after every 100 reflections did not show any significant change ($\sim 2\%$) in intensity during data collection; Lorentz–polarization correction, absorption correction based on ψ scans (max., min. transmission = 0.9940, 0.4964), no extinction corrections. Structure solved by direct methods (*SHELXTL-Plus*, Sheldrick, 1988) and subsequent difference Fourier methods. H atoms located in difference Fourier maps, H atoms included in the refinement with geometric constraints in a riding model with isotropic temperature factors fixed at 0.05 Å². Final full-matrix least-squares refinement using *SHELXTL-Plus*, all non-H atoms anisotropic, converged to $R = 0.043$, $wR = 0.050$; $w = 1/[\sigma^2(F_o) + 0.0048(F_o)^2]$, $\sum w(|F_o| - |F_c|)^2$ minimized, 307 parameters, $S = 0.94$, $(\Delta/\sigma)_{max} = 0.01$,
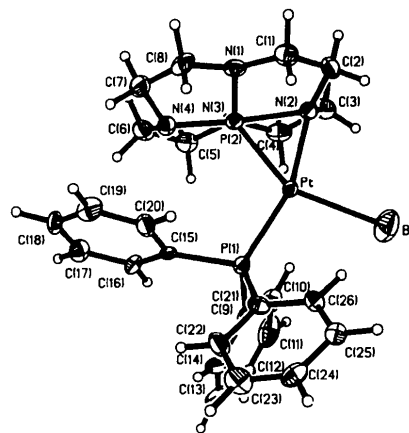
Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($\text{Å}^2 \times 10^3$)

	x	y	z	U_{eq}*
Pt	−2000 (1)	1971 (1)	8192 (1)	28 (1)
Br	75 (1)	2376 (2)	8591 (1)	89 (1)
P(1)	−2834 (2)	2307 (2)	9402 (2)	29 (1)
P(2)	−3255 (2)	1278 (2)	7250 (2)	29 (1)
N(1)	−3625 (8)	1947 (6)	6401 (6)	41 (3)
N(2)	−1737 (7)	1441 (6)	6954 (5)	35 (3)
N(3)	−2980 (8)	123 (6)	7288 (6)	37 (3)
N(4)	−4646 (7)	1098 (6)	7440 (6)	36 (3)
C(1)	−2782 (11)	2577 (9)	6109 (8)	51 (4)
C(2)	−1662 (11)	2095 (9)	6235 (8)	52 (4)
C(3)	−1111 (10)	558 (9)	6933 (8)	51 (4)
C(4)	−1813 (11)	−151 (8)	7430 (8)	49 (4)
C(5)	−3819 (11)	−383 (9)	7774 (8)	50 (4)
C(6)	−4938 (12)	119 (10)	7609 (9)	63 (5)
C(7)	−5446 (10)	1571 (10)	6847 (8)	54 (4)
C(8)	−4792 (9)	2313 (9)	6424 (8)	48 (4)
C(9)	−2329 (9)	1551 (8)	10311 (7)	37 (3)
C(10)	−1579 (10)	814 (9)	10152 (8)	51 (4)
C(11)	−1308 (12)	193 (12)	10778 (12)	79 (6)
C(12)	−1713 (13)	264 (12)	11598 (11)	73 (6)
C(13)	−2446 (15)	1022 (13)	11754 (8)	82 (7)
C(14)	−2766 (12)	1626 (10)	11131 (7)	54 (4)
C(15)	−4371 (8)	2134 (7)	9341 (6)	27 (3)
C(16)	−4897 (8)	1376 (7)	9668 (7)	34 (3)
C(17)	−6055 (10)	1225 (8)	9547 (8)	45 (4)
C(18)	−6761 (9)	1882 (8)	9057 (8)	44 (4)
C(19)	−6220 (11)	2645 (10)	8717 (8)	52 (4)
C(20)	−5074 (9)	2778 (9)	8865 (8)	43 (4)
C(21)	−2691 (9)	3487 (7)	9815 (6)	33 (3)
C(22)	−3460 (11)	3888 (9)	10367 (8)	54 (4)
C(23)	−3228 (12)	4808 (8)	10696 (9)	56 (5)
C(24)	−2341 (11)	5281 (9)	10460 (8)	50 (4)
C(25)	−1570 (9)	4944 (7)	9897 (7)	38 (3)
C(26)	−1770 (9)	4037 (8)	9601 (7)	35 (3)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Fig. 1. Thermal-ellipsoid drawing (*SHELXTL-Plus*) of the molecule. Thermal ellipsoids scaled to enclose 30% probability. H atoms are represented as spheres of arbitrary radii.

$\Delta\rho_{max,min} = 1\cdot03$, $-3\cdot48$ e Å^{-3} in the final difference Fourier map. Atomic scattering factors for C, H, N, O, P and Pt, and the real and imaginary parts of the dispersion correction for P and Pt were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 1,* selected bond lengths and angles are given in Table 2. The identification of the atoms and the configuration are shown in the thermal-ellipsoid drawing (*SHELXTL-Plus*) of Fig. 1.

Related literature. Previous syntheses and structural studies of transition-metal complexes with tetraaza-η^2-phosphoranido ligands include (η^2-cyclenphosphoranido)ML_n, where $ML_n = \text{Mo}(\eta^5\text{-C}_5\text{H}_5)(\text{CO})_2$ (Lattman, Chopra, Cowley & Arif, 1986), $\text{Co}(\text{CO})_3$ (de Meester, Lattman & Chu, 1987) and $\text{Pt}(\text{Cl})\text{PPh}_3$ (Lattman, Burns, Chopra, Cowley & Arif, 1987). Also, the (η^2-cyclamphosphoranido)$M(\eta^5\text{-C}_5\text{H}_5)$-$(\text{CO})_2$ ($M = \text{Mo}$, W) complexes have been reported (Dupart, Grand, Pace & Riess, 1982; Dupart, Grand & Riess, 1986).

* Lists of anisotripic temperature factors, bond lengths, bond angles, torsion angles, hydrogen parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51995 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Selected bond lengths* (Å) *and bond angles* (°)

Pt—Br	2·523 (2)	Pt—P(1)	2·225 (3)
Pt—P(2)	2·230 (3)	Pt—N(2)	2·107 (8)
P(1)—C(9)	1·842 (11)	P(1)—C(15)	1·804 (10)
P(1)—C(21)	1·809 (11)	P(2)—N(1)	1·662 (9)
P(2)—N(2)	1·871 (9)	P(2)—N(3)	1·683 (9)
P(2)—N(4)	1·687 (9)	N(1)—C(1)	1·428 (16)
N(1)—C(8)	1·460 (15)	N(2)—C(2)	1·463 (15)
N(2)—C(3)	1·460 (15)	N(3)—C(4)	1·419 (15)
N(3)—C(5)	1·464 (16)	N(4)—C(6)	1·468 (17)
N(4)—C(7)	1·434 (15)	C(1)—C(2)	1·476 (18)
C(3)—C(4)	1·542 (18)	C(5)—C(6)	1·496 (18)
C(7)—C(8)	1·484 (18)		
Br—Pt—P(1)	102·0 (1)	Br—Pt—P(2)	147·0 (1)
P(1)—Pt—P(2)	110·3 (1)	Br—Pt—N(2)	96·4 (2)
P(1)—Pt—N(2)	161·2 (2)	P(2)—Pt—N(2)	51·0 (2)
Pt—P(1)—C(9)	112·6 (4)	Pt—P(1)—C(15)	114·4 (3)
C(9)—P(1)—C(15)	103·1 (5)	Pt—P(1)—C(21)	117·8 (4)
C(9)—P(1)—C(21)	105·0 (5)	C(15)—P(1)—C(21)	102·3 (5)
Pt—P(2)—N(1)	112·9 (3)	Pt—P(2)—N(2)	61·1 (3)
N(1)—P(2)—N(2)	86·2 (4)	Pt—P(2)—N(3)	107·3 (3)
N(1)—P(2)—N(3)	129·2 (5)	N(2)—P(2)—N(3)	87·1 (4)
Pt—P(2)—N(4)	123·2 (3)	N(1)—P(2)—N(4)	91·4 (5)
N(2)—P(2)—N(4)	175·7 (4)	N(3)—P(2)—N(4)	91·6 (4)
P(2)—N(1)—C(1)	117·8 (8)	P(2)—N(1)—C(8)	112·3 (8)
C(1)—N(1)—C(8)	116·3 (9)	Pt—N(2)—P(2)	67·9 (3)
Pt—N(2)—C(2)	119·0 (7)	P(2)—N(2)—C(2)	111·8 (7)
Pt—N(2)—C(3)	115·8 (7)	P(2)—N(2)—C(3)	112·4 (7)
C(2)—N(2)—C(3)	118·7 (9)	P(2)—N(3)—C(4)	117·0 (7)
P(2)—N(3)—C(5)	111·8 (7)	C(4)—N(3)—C(5)	116·7 (9)
P(2)—N(4)—C(6)	114·4 (8)	P(2)—N(4)—C(7)	114·0 (7)
C(6)—N(4)—C(7)	114·6 (9)	N(1)—C(1)—C(2)	106·5 (10)
N(2)—C(2)—C(1)	107·6 (10)	N(2)—C(3)—C(4)	105·9 (10)
N(3)—C(4)—C(3)	106·0 (9)	N(3)—C(5)—C(6)	106·2 (10)
N(4)—C(6)—C(5)	106·2 (10)	N(4)—C(7)—C(8)	106·9 (9)
N(1)—C(8)—C(7)	105·1 (10)		

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A Pentamethylcyclopentadienyl, Chloro, Oxo, Ether Complex of Ytterbium(III)

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Abstract. 2,3,4-μ_3-Chloro-1,2;1,2;2,3;2,4;3,4;3,5;4,5-hepta-μ-chloro-2,5-bis(diethyl ether)-3,4,5-μ_3-oxo-1,1,3,4,5-pentakis(η^5-pentamethylcyclopentadienyl)-pentaytterbium(III), [Yb$_5$O(C$_4$H$_{10}$O)$_2$(C$_{10}$H$_{15}$)$_5$Cl$_8$], $M_r = 1989\cdot23$, monoclinic, $P2_1/m$, $a = 17\cdot007$ (3), $b = 16\cdot485$ (3), $c = 13\cdot592$ (3) Å, $\beta = 111\cdot39$ (2)°, $V = 3548\cdot2$ Å^3, $Z = 2$, $D_x = 1\cdot86$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 68\cdot49$ cm^{-1}, $F(000) = 1906$, $T = 296$ K, $R = 0\cdot056$ for 4061 [$F^2 > 2\sigma(F^2)$] of 6532 total unique data. The structure consists of a single large cluster in which the five Yb atoms are linked together by Cl and O bridges. The π-bonding cyclopentadienyl rings and the O atoms of the ether ligands are bonded to the Yb atoms on the periphery of the cluster.

Experimental. The title complex (1) was isolated from the reaction of [Yb(C$_5$Me$_5$)$_2$(OEt$_2$)] with YbCl$_3$ containing some YbOCl as an impurity. A blue air-sensitive crystal, $0\cdot15 \times 0\cdot27 \times 0\cdot33$ mm, was sealed inside a quartz capillary in an argon-filled drybox. X-ray diffraction intensities (θ–2θ scans) were obtained using a modified Picker FACS-I automatic diffractometer equipped with a graphite monochromator. Cell dimensions from 27 reflections, $20 < 2\theta < 26°$; analytical absorption correction, range $2\cdot0$ to $3\cdot1$; max. $\sin\theta/\lambda = 0\cdot60$ Å^{-1}, h -20 to 20, k 0 to 19, l -16 to 16; three standard reflections, $2\cdot2$, $2\cdot1$, $2\cdot3\%$ variation in standards' intensities from average, intensities adjusted isotropically; 13 177 data, 6532 unique [including 4061, $F^2 > 2\sigma(F^2)$], $R_{int} = 0\cdot045$; structure solved by Patterson and Fourier methods; refined on F, 281 parameters; H atoms not included; distance restraints on one ether [O(2)—C(29) 1·45 (1), C(29)—C(30) 1·54 (1) Å] and two atoms of a methyl Cp ring [C(23)—C(23')

1·41 (1), C(23)—C(28) 1·54 (1) Å]; anisotropic thermal parameters on 28 atoms (4 Yb, 5 Cl, 2 O and 17 C atoms), and isotropic on 16 atoms (1 O and 15 C atoms); $R = 0\cdot099$ (all data), $R = 0\cdot056$ [$F^2 > 2\sigma(F^2)$ data], $wR = 0\cdot072$, $S = 1\cdot44$; $w = 4F^2[\sigma^2(F^2) + (0\cdot035F^2)^2]^{-1}$; max. (shift/$\sigma$) = 0·06; extinction correction $F_{obs}(1 + 3\cdot4 \times 10^{-8}I)$, max. correction 2·7%; max. and min. of ΔF synthesis 2·3 and $-1\cdot9$ e Å^{-3}; atomic f for neutral Yb, Cl, O and C atoms from *International Tables for X-ray Crystallography* (1974); local unpublished programs and *ORTEP* (Johnson, 1965).

(1)
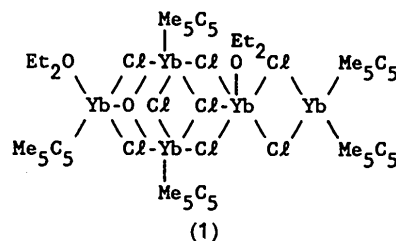
Atomic parameters are listed in Table 1,* and distances and angles are listed in Table 2. Fig. 1 shows a schematic diagram of the structure.

Related literature. Comparable distances for bridging Yb—Cl are 2·595 and 2·756 Å for Li[YbCl$_2$-(C$_5$Me$_5$)$_2$][Et$_2$O] and Yb(C$_5$Me$_5$)$_2$(μ-Cl)$_2$AlCl$_2$ respectively (Watson, Whitney & Harlow, 1981).