

O32—Co1—O34 ⁱ	84.54 (17)	Co1—O34—N31	88.2 (3)
O32—Co1—N11 ⁱ	113.04 (18)	Co1—N11—C12	117.5 (4)
O34—Co1—N11	156.13 (16)	Co1—N11—C16	125.1 (4)
O32 ⁱ —Co1—O34	84.54 (17)	C12—N11—C16	117.4 (5)
O34—Co1—O34 ⁱ	85.78 (17)	O32—N31—O34	115.1 (4)
O34—Co1—N11 ⁱ	89.93 (17)	O33—N31—O34	123.1 (5)
O32 ⁱ —Co1—N11	113.04 (18)	O32—N31—O33	121.8 (4)
O34 ⁱ —Co1—N11	89.93 (17)	N11—C12—C13	122.2 (6)
N11—Co1—N11 ⁱ	102.88 (18)	N11—C16—C17	116.8 (5)
O32 ⁱ —Co1—O34 ⁱ	58.63 (16)	N11—C16—C15	122.9 (5)

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.

Reflections were measured with a scan angle of $\Delta\omega = (0.80 + 0.35\tan\theta)^\circ$, and horizontal and vertical apertures of 3.00 and 4.00 mm, respectively. Only 27% of the intensity data were above the $2.5\sigma(I)$ level in the $\theta = 25^\circ$ region. Due to the irregular shape of the crystal (which was cut from a larger aggregate), no satisfactory description of the crystal could be obtained for use in a numerical absorption correction procedure. An empirical absorption correction was therefore applied. The thiophene ring proved to be disordered over two orientations. The site-occupation factor of the major disorder component refined to 0.580 (7). Mild restraints were applied to the bond lengths and 1,3 distances in both disorder components. All non-H atoms were refined with anisotropic displacement parameters, except for the disordered C atoms, which were refined isotropically. The displacement parameters of the C atoms in both disorder components were constrained to the same value. The relatively high *R* values are related to the weak scattering that is most probably a consequence of the disorder in the thiophene ring.

Data collection: locally modified *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *SET4* (de Boer & Duisenberg, 1984). Data reduction: *HELENA* (Spek, 1993). Program(s) used to solve structure: *DIRDIF92* (Beurskens *et al.*, 1992). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *PLATON* (Spek, 1990), *PLUTON* (Spek, 1995a). Software used to prepare material for publication: *PLATON*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: CF1076). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37; Version 5.10 of October 1995.
Barrow, M. J., Milburn, G. H. W., Zeng, Z., Sarkar, A. & Talwar, S. (1994). *Acta Cryst. C50*, 650–652.

Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., Garcia-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1992). *The DIRDIF Program System*. Technical Report. Crystallography Laboratory, University of Nijmegen, The Netherlands.

Boer, J. L. de & Duisenberg, A. J. M. (1984). *Acta Cryst. A40*, C-410.
Enraf-Nonius (1989). *CAD-4 Software*. Version 5. Enraf-Nonius, Delft, The Netherlands.

Giordano, T. J. & Rasmussen, P. G. (1975). *Inorg. Chem.* **14**, 1628–1634.

Han, R. & Parkin, G. (1991). *J. Am. Chem. Soc.* **113**, 9707–9708.

Neenan, T. X. & Driessen, W. L. (1996). *Acta Cryst. C52*, 59–61.

Neenan, T. X., Driessen, W. L., Haasnoot, J. G. & Reedijk, J. (1996). *Inorg. Chim. Acta*. In the press.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Spek, A. L. (1990). *Acta Cryst. A46*, C-34.

Spek, A. L. (1993). *HELENA. Program for Data Reduction*. University of Utrecht, The Netherlands.

Spek, A. L. (1995a). *PLUTON. Molecular Graphics Program*. Version of July 1995. University of Utrecht, The Netherlands.

Spek, A. L. (1995b). *Collected Abstracts of the Annual Meeting of the American Crystallographic Association*, 1995, Montreal, p. 33.

Acta Cryst. (1996). **C52**, 2193–2196

Bis[1,2-bis(diphenylphosphino)ethane-*P,P'*]-chloroosmium(II) Hexafluorophosphate Dichloromethane Solvate

ALAN J. LOUGH,* ROBERT H. MORRIS* AND MARCEL SCHLAF

Department of Chemistry, University of Toronto, Toronto, Ontario, Canada M5S 1A1. E-mail: alough@alchemy.chem.utoronto.ca and rmorris@alchemy.chem.utoronto.ca

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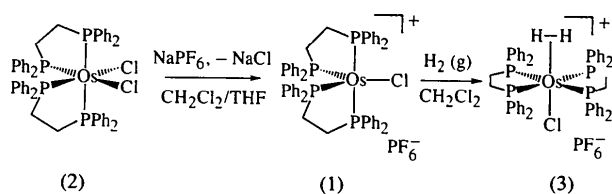
Abstract

The cation molecule of the title compound, $[\text{OsCl}(\text{C}_2\text{H}_2\text{P}_2)_2]\text{PF}_6 \cdot 1.5\text{CH}_2\text{Cl}_2$, has a distorted trigonal-bipyramidal structure. Despite strong steric repulsion between the PPh_2 groups, the Os—P bonds in the equatorial plane are short [2.2416(12) and 2.2587(13) Å] and the P—Os—P angle is only $94.24(5)^\circ$.

Comment

The title compound, (1), was synthesized in order to provide the starting material in the preparation of the dihydrogen complex *trans*- $[\text{Os}(\text{H}_2)\text{Cl}(\text{dppe})_2]\text{PF}_6$, (3) [where dppe is 1,2-bis(diphenylphosphino)ethane; see scheme below]. The X-ray and neutron diffraction studies of (3) are discussed elsewhere (Maltby *et al.*, 1996). An X-ray study of the five-coordinate compound

(1) was of interest in order to help understand its reaction with hydrogen to give the octahedral six-coordinate compound (3).



The cation of compound (1) has a distorted trigonal-bipyramidal structure (Fig. 1) which is comparable to that of the Ru^{II} analogue, (4), reported recently (Chin, Lough, Morris, Schweitzer & D'Agostino, 1994; Polam & Porter, 1993). The major distortion from trigonal-bipyramidal coordination is the 'Y'-shaped geometry of the equatorial plane. The equatorial angles P1—Os—Cl and P3—Os—Cl are 128.15 (4) and 137.60 (4)°, respectively, and in compound (4) the Cl—Ru—P_{eq} angles are 129.2 (1) and 135.8 (1)° (where P_{eq} is an equatorial P atom). The Os—Cl distance of 2.3760 (11) Å in compound (1) is slightly shorter than the Ru—Cl distance of 2.395 (2) Å in (4), perhaps because of more *p*(Cl) to *d*(M) π-bonding for *M* = Os versus Ru. These metal-chlorine distances are significantly shorter than the corresponding distances in the six-coordinate dichloro complexes *trans*-[Os(dppe)₂Cl₂] (Levason, Champness & Webster, 1993), with Os—Cl 2.434 (1) Å, and *trans*-[Ru(dppe)₂Cl₂] (Lobana, Singh & Tiekink, 1990), with Ru—Cl 2.436 (1). A sample of 61 independent Os^{II}—Cl distances taken from 26 Cambridge Structural Database (Allen, Kennard & Taylor, 1983) entries ranged from 2.289 to 2.511 Å, with a mean value of 2.405 (6) Å.

In compound (1), the Os—P_{eq} distances Os—P1 and Os—P2 of 2.2416 (12) and 2.2587 (13) Å, respectively, are significantly shorter than the Os—P_{ax} distances (where P_{ax} is an axial P atom) of 2.3640 (12) Å for Os—P2 and 2.3634 (12) Å for Os—P4, and they are also shorter than the Os—P distances found in the six-coordinate dichloroosmium compound mentioned above, which range from 2.348 (1) to 2.372 (1) Å. This suggests that the P_{eq} atoms in compound (1) are acting as powerful σ donors. Steric repulsions in the molecule cause angles at the *sp*³-hybridized P_{eq} atoms to deviate from 109.5 to 125.5 (2)° for Os—P3—C35 and 124.4 (2)° for Os—P1—C9. Hence, one might expect the Os—P bonds in the equatorial plane of compound (1) to be longer and the P1—Os—P3 angle of 94.24 (5)° to be wider because of the steric strain imposed by the bulky PPh₂ groups. A recent theoretical study (Rachidi, Eisenstein & Jean, 1990) explains why the atoms at the top of the 'Y' equatorial plane should be good σ donors when the bottom of the 'Y' is a π donor, chloride in this case. Table 3 shows the relationship between *M*—P_{eq} distances and P_{eq}—*M*—P_{eq} angles for four related trigonal-bipyramidal complexes. In all cases, the P—*M*—P angles are small and the *M*—P_{eq} distances are short.

Experimental

The title compound was prepared by dissolving *cis*-OsCl₂(dppe)₂ [(2); prepared according to the method of Chatt & Hayter (1961)] and NaPF₆ in a mixture of tetrahydrofuran and CH₂Cl₂. The reaction mixture was stirred at room temperature for 48 h resulting in a dark brown solution which was evaporated to dryness and redissolved in CH₂Cl₂. The dark brown solution was filtered through Celite/cotton wool to remove the excess NaPF₆ and also the NaCl formed in the reaction. Dark red crystals suitable for X-ray diffraction analysis were obtained within 5 d by slow diffusion of diethyl ether into a CH₂Cl₂ solution of the title compound at room temperature.

Crystal data

[OsCl(C₂₆H₂₄P₂)₂]₂PF₆·
1.5CH₂Cl₂
*M*_r = 1294.79
Monoclinic
*P*2₁/*c*
a = 12.298 (1) Å
b = 15.685 (2) Å
c = 27.851 (4) Å
β = 100.51 (1)°
V = 5282.2 (11) Å³
Z = 4
*D*_x = 1.628 Mg m⁻³
*D*_m not measured

Mo *K*α radiation
λ = 0.71073 Å
Cell parameters from 38
reflections
θ = 5.13–15.09°
μ = 2.826 mm⁻¹
T = 150 (2) K
Fragment cut from needle
0.36 × 0.29 × 0.26 mm
Dark red

Data collection

Siemens *P*4 diffractometer
ω scans

7904 observed reflections
[*I* > 2σ(*I*)]

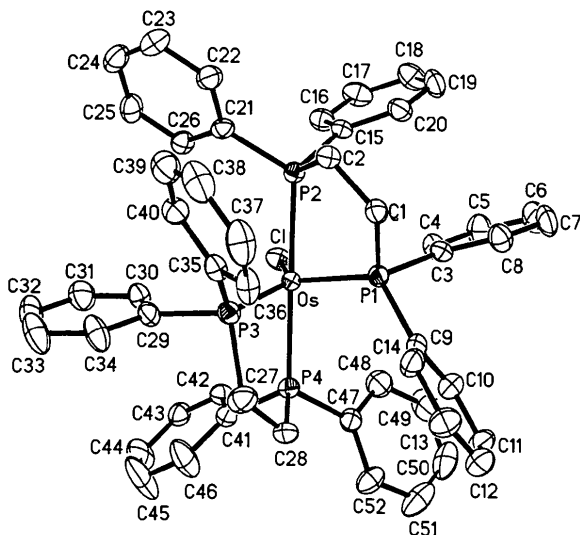


Fig. 1. View of the title molecule with the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

Absorption correction:	$R_{\text{int}} = 0.0291$	C37	0.0738 (5)	0.0855 (4)	-0.1068 (2)	0.0462 (15)
empirical (<i>SHELXA90</i> ;	$\theta_{\text{max}} = 26.01^\circ$	C38	0.1529 (5)	0.0692 (4)	-0.1348 (2)	0.046 (2)
Sheldrick, 1990)	$h = 0 \rightarrow 14$	C39	0.1265 (4)	0.0225 (4)	-0.1774 (2)	0.0391 (13)
$T_{\text{min}} = 0.6611$, $T_{\text{max}} =$	$k = 0 \rightarrow 19$	C40	0.0205 (4)	-0.0078 (3)	-0.1916 (2)	0.0290 (11)
0.8207	$l = -34 \rightarrow 33$	C41	-0.4973 (4)	-0.1055 (3)	-0.2359 (2)	0.0279 (11)
10 774 measured reflections	3 standard reflections	C42	-0.5310 (4)	-0.1770 (3)	-0.2646 (2)	0.0291 (10)
10 284 independent reflections	monitored every 97 reflections	C43	-0.5971 (4)	-0.1687 (3)	-0.3104 (2)	0.0322 (11)
	intensity decay: <2%	C44	-0.6287 (6)	-0.0895 (4)	-0.3279 (2)	0.054 (2)
		C45	-0.5961 (8)	-0.0184 (4)	-0.3004 (2)	0.079 (3)
		C46	-0.5288 (6)	-0.0258 (4)	-0.2543 (2)	0.057 (2)
		C47	-0.5097 (4)	-0.1748 (3)	-0.1429 (2)	0.0282 (10)
		C48	-0.5046 (5)	-0.2629 (4)	-0.1379 (2)	0.0446 (14)
		C49	-0.5734 (5)	-0.3050 (4)	-0.1112 (2)	0.052 (2)
		C50	-0.6460 (5)	-0.2608 (5)	-0.0895 (2)	0.052 (2)
		C51	-0.6544 (4)	-0.1739 (5)	-0.0954 (2)	0.050 (2)
		C52	-0.5872 (4)	-0.1302 (4)	-0.1218 (2)	0.0366 (13)
		P5	-0.19826 (15)	-0.74099 (10)	-0.04815 (5)	0.0449 (4)
		F1	-0.2782 (4)	-0.6615 (3)	-0.0585 (2)	0.091 (2)
		F2	-0.1183 (3)	-0.8222 (3)	-0.03769 (13)	0.0655 (11)
		F3†	-0.1467 (14)	-0.7032 (7)	0.0020 (4)	0.092 (5)
		F4†	-0.1067 (9)	-0.7051 (7)	-0.0749 (5)	0.093 (4)
		F5†	-0.2938 (11)	-0.7847 (5)	-0.0236 (4)	0.073 (3)
		F6†	-0.2563 (9)	-0.7853 (7)	-0.0994 (3)	0.064 (3)
		F3*†	-0.1052 (16)	-0.6738 (17)	-0.0172 (12)	0.090 (8)
		F4*†	-0.168 (4)	-0.7037 (18)	-0.0952 (10)	0.133 (15)
		F5*†	-0.223 (3)	-0.7682 (11)	0.0021 (10)	0.082 (11)
		F6*†	-0.278 (2)	-0.8007 (15)	-0.0756 (14)	0.094 (12)
		C25†	-0.4861 (15)	-0.5785 (10)	-0.0036 (4)	0.076 (5)
		C13†	-0.4632 (5)	-0.4935 (4)	0.0364 (2)	0.111 (2)
		C14†	-0.5299 (4)	-0.5398 (4)	-0.0638 (2)	0.0880 (14)
		C15†	-0.4579 (10)	-0.2622 (10)	-0.4000 (4)	0.079 (4)
		C11†	-0.4101 (2)	-0.3174 (2)	-0.34524 (13)	0.0727 (7)
		C12†	-0.3472 (4)	-0.2382 (2)	-0.43007 (14)	0.0876 (12)
		C15*†	-0.488 (3)	-0.238 (3)	-0.4186 (11)	0.079 (13)
		C11*†	-0.4402 (11)	-0.3157 (11)	-0.3777 (5)	0.135 (6)
		C12*†	-0.4000 (13)	-0.1937 (11)	-0.4510 (6)	0.134 (6)

RefinementRefinement on F^2 $R(F) = 0.0340$ $wR(F^2) = 0.0797$ $S = 1.042$

10 279 reflections

706 parameters

H atoms riding (C—H = 0.96 Å)

 $w = 1/[\sigma^2(F_o^2) + (0.0253P)^2 + 9.8932P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = -0.034$ $\Delta\rho_{\text{max}} = 0.724 \text{ e } \text{Å}^{-3}$ $\Delta\rho_{\text{min}} = -0.824 \text{ e } \text{Å}^{-3}$

Extinction correction: none

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
Os	-0.235336 (14)	-0.181395 (11)	-0.170932 (6)	0.01917 (5)
C1	-0.30626 (9)	-0.29993 (7)	-0.21993 (4)	0.0300 (3)
P1	-0.17897 (9)	-0.17684 (8)	-0.08976 (4)	0.0220 (2)
P2	-0.05459 (10)	-0.23705 (7)	-0.16437 (4)	0.0206 (2)
P3	-0.19586 (10)	-0.04298 (8)	-0.18213 (5)	0.0248 (3)
P4	-0.41316 (10)	-0.12145 (8)	-0.17523 (4)	0.0242 (3)
C1	-0.0296 (4)	-0.1553 (3)	-0.0731 (2)	0.0256 (10)
C2	0.0342 (4)	-0.1832 (3)	-0.1130 (2)	0.0241 (9)
C3	-0.1948 (4)	-0.2812 (3)	-0.0616 (2)	0.0253 (10)
C4	-0.2491 (4)	-0.3491 (3)	-0.0871 (2)	0.0306 (11)
C5	-0.2592 (5)	-0.4259 (4)	-0.0644 (2)	0.0415 (13)
C6	-0.2164 (5)	-0.4364 (4)	-0.0153 (2)	0.0437 (14)
C7	-0.1624 (5)	-0.3695 (4)	0.0107 (2)	0.0417 (14)
C8	-0.1511 (4)	-0.2926 (3)	-0.0120 (2)	0.0322 (12)
C9	-0.2411 (4)	-0.1097 (3)	-0.0485 (2)	0.0266 (10)
C10	-0.3382 (4)	-0.1383 (3)	-0.0348 (2)	0.0310 (11)
C11	-0.3907 (4)	-0.0903 (4)	-0.0043 (2)	0.0388 (13)
C12	-0.3469 (5)	-0.0137 (4)	0.0134 (2)	0.0422 (14)
C13	-0.2507 (5)	0.0159 (3)	0.0003 (2)	0.0388 (13)
C14	-0.1978 (4)	-0.0317 (3)	-0.0309 (2)	0.0310 (11)
C15	-0.0385 (4)	-0.3503 (3)	-0.1498 (2)	0.0216 (9)
C16	-0.0739 (4)	-0.4109 (3)	-0.1861 (2)	0.0294 (11)
C17	-0.0704 (4)	-0.4973 (3)	-0.1745 (2)	0.0381 (13)
C18	-0.0319 (5)	-0.5239 (3)	-0.1274 (2)	0.0423 (14)
C19	0.0049 (5)	-0.4650 (3)	-0.0912 (2)	0.0374 (13)
C20	0.0025 (4)	-0.3778 (3)	-0.1021 (2)	0.0299 (11)
C21	0.0147 (4)	-0.2273 (3)	-0.2166 (2)	0.0235 (10)
C22	0.1276 (4)	-0.2084 (3)	-0.2097 (2)	0.0302 (11)
C23	0.1805 (5)	-0.2044 (3)	-0.2496 (2)	0.0384 (13)
C24	0.1232 (5)	-0.2200 (3)	-0.2959 (2)	0.0371 (13)
C25	0.0113 (5)	-0.2395 (3)	-0.3027 (2)	0.0342 (12)
C26	-0.0428 (4)	-0.2423 (3)	-0.2632 (2)	0.0273 (10)
C27	-0.2941 (4)	0.0308 (3)	-0.1610 (2)	0.0376 (13)
C28	-0.3946 (4)	-0.0150 (3)	-0.1479 (2)	0.0294 (11)
C29	-0.2189 (4)	-0.0229 (3)	-0.2481 (2)	0.0284 (11)
C30	-0.2527 (4)	-0.0880 (3)	-0.2813 (2)	0.0325 (12)
C31	-0.2663 (5)	-0.0726 (4)	-0.3309 (2)	0.0441 (13)
C32	-0.2483 (5)	0.0076 (4)	-0.3478 (2)	0.048 (2)
C33	-0.2168 (5)	0.0730 (4)	-0.3153 (2)	0.051 (2)
C34	-0.2012 (4)	0.0575 (3)	-0.2655 (2)	0.0393 (14)
C35	-0.0602 (4)	0.0069 (3)	-0.1636 (2)	0.0260 (10)
C36	-0.0329 (5)	0.0566 (3)	-0.1211 (2)	0.0366 (13)

† Partial occupancies (see below).

Table 2. Selected geometric parameters (Å , $^\circ$)

Os—P1	2.2416 (12)	P2—C2	1.839 (4)
Os—P3	2.2587 (13)	P3—C35	1.830 (5)
Os—P4	2.3634 (12)	P3—C29	1.835 (5)
Os—P2	2.3640 (12)	P3—C27	1.846 (5)
Os—C1	2.3760 (11)	P4—C47	1.820 (5)
P1—C9	1.827 (5)	P4—C41	1.830 (5)
P1—C3	1.840 (5)	P4—C28	1.832 (5)
P1—C1	1.842 (5)	C1—C2	1.537 (6)
P2—C21	1.820 (5)	C27—C28	1.530 (7)
P2—C15	1.824 (5)		
P1—Os—P3	94.24 (5)	C27—P3—Os	112.9 (2)
P1—Os—P4	98.71 (4)	C47—P4—C41	102.1 (2)
P3—Os—P4	80.26 (4)	C47—P4—C28	104.9 (2)
P1—Os—P2	79.65 (4)	C41—P4—C28	105.4 (2)
P3—Os—P2	98.21 (4)	C47—P4—Os	118.2 (2)
P4—Os—P2	177.71 (4)	C41—P4—Os	117.6 (2)
P1—Os—C1	128.15 (4)	C28—P4—Os	107.3 (2)
P3—Os—C1	137.60 (4)	C2—C1—P1	112.5 (3)
P4—Os—C1	92.46 (4)	C1—C2—P2	112.6 (3)
P2—Os—C1	89.82 (4)	C4—C3—P1	123.0 (4)
C9—P1—C3	98.9 (2)	C8—C3—P1	118.9 (4)
C9—P1—C1	104.4 (2)	C14—C9—P1	123.6 (4)
C3—P1—C1	103.5 (2)	C10—C9—P1	117.7 (4)
C9—P1—Os	124.4 (2)	C20—C15—P2	121.2 (4)
C3—P1—Os	111.4 (2)	C16—C15—P2	119.6 (3)
C1—P1—Os	111.8 (2)	C26—C21—P2	120.2 (4)
C21—P2—C15	102.2 (2)	C22—C21—P2	120.4 (4)
C21—P2—C2	106.5 (2)	C28—C27—P3	112.7 (3)
C15—P2—C2	104.4 (2)	C27—C28—P4	111.9 (3)
C21—P2—Os	118.6 (2)	C34—C29—P3	120.2 (4)
C15—P2—Os	116.02 (15)	C30—C29—P3	120.8 (4)
C2—P2—Os	107.87 (15)	C40—C35—P3	118.5 (4)
C35—P3—C29	100.1 (2)	C36—C35—P3	122.7 (4)
C35—P3—C27	105.0 (2)	C46—C41—P4	122.7 (4)
C29—P3—C27	102.8 (2)	C42—C41—P4	118.6 (4)
C35—P3—Os	125.5 (2)	C48—C47—P4	119.1 (4)
C29—P3—Os	107.8 (2)	C52—C47—P4	122.3 (4)

Table 3. Selected bond lengths (Å) and angles (°) for some distorted trigonal-bipyramidal complexes of the type [MCl(dppe)₂]ⁿ⁺ (n = 0, 1), with Y-shaped equatorial planes

Complex	M—P _{ax}	M—P _{eq}	M—Cl	P _{eq} —M—P _{eq}
[RuCl(dppe) ₂] ⁺ ^a	2.371 (2) 2.372 (2)	2.243 (2) 2.238 (2)	2.395 (2)	95.0 (1)
[OsCl(dppe) ₂] ⁺ ^b	2.3634 (12) 2.3640 (12)	2.2416 (12) 2.2587 (13)	2.3760 (11)	94.24 (5)
TcCl(dppe) ₂ ^c	2.376 (2) 2.359 (2)	2.240 (2) 2.255 (2)	2.432 (2)	93.9 (1)
ReCl(dppe) ₂ ^d	2.366 (1) 2.380 (1)	2.269 (1) 2.256 (1)	2.403 (1)	93.7

References: (a) Chin, Lough, Morris, Schweitzer & D'Agostino (1994); (b) this work; (c) Burrell, Bryan & Kubas (1994); (d) Hughes, Pombeiro, Pickett & Richards (1983).

The hexafluorophosphate anion is rotationally disordered over two sites about the axial F—P—F axis, with occupancies of 0.68 (2)/0.32(2). In total, there are 1.5 CH₂Cl₂ molecules for every cation in three independent sites, with occupancies of 0.5, 0.75 and 0.25 for the C2S, C1S and C1S* molecules, respectively.

Data collection: XSCANS (Siemens, 1994). Cell refinement: XSCANS. Data reduction: SHELXTL/PC (Sheldrick, 1994). Program(s) used to solve structure: SHELXTL/PC. Program(s) used to refine structure: SHELXTL/PC. Molecular graphics: SHELXTL/PC. Software used to prepare material for publication: SHELXTL/PC.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1187). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Allen, F. H., Kennard, O. & Taylor, R. (1983). *Acc. Chem. Res.* **16**, 146–153.
- Burrell, A. K., Bryan, J. C. & Kubas, G. J. (1994). *J. Am. Chem. Soc.* **116**, 1575–1576.
- Chatt, J. & Hayter, R. G. (1961). *J. Chem. Soc.* pp. 2605–2611.
- Chin, B., Lough, A. J., Morris, R. H., Schweitzer, C. T. & D'Agostino, C. (1994). *Inorg. Chem.* **33**, 6278–6288.
- Hughes, D. L., Pombeiro, A. J. L., Pickett, C. J. & Richards, R. L. (1983). *J. Organomet. Chem.* **248**, C26–C28.
- Levason, W., Champness, N. R. & Webster, M. (1993). *Acta Cryst.* **C49**, 1884–1885.
- Lobana, T. S., Singh, R. & Tiekink, E. R. T. (1990). *J. Coord. Chem.* **21**, 255–229.
- Maltby, P. A., Schlaf, M., Steinbeck, M., Lough, A. J., Morris, R. H., Klooster, W. T., Koetzle, T. F. & Srivastava, R. C. (1996). *J. Am. Chem. Soc.* In the press.
- Polam, J. R. & Porter, L. C. (1993). *J. Coord. Chem.* **29**, 109–119.
- Rachidi, I. E., Eisenstein, O. & Jean, Y. (1990). *New J. Chem.* **14**, 671–677.
- Sheldrick, G. M. (1990). *SHELXA90. Program for Absorption Correction*. University of Göttingen, Germany.
- Sheldrick, G. M. (1994). *SHELXTL/PC*. Version 5.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Siemens (1994). *XSCANS Users Manual*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1996). **C52**, 2196–2198

1-(R)-{2-[1-(R)-(N,N-Dimethylamino)ethyl]-1,1'-(S,S)-biferrocen-2'-yl}ethyl-N,N-dimethylammonium Trichloro(dimethyl sulfoxide-S)platinate(II)

BRIAN H. ROBINSON, JIM SIMPSON AND DAVID J. WILSON

Department of Chemistry, University of Otago, PO Box 56, Dunedin, New Zealand. E-mail: jsimpson@alkali.otago.ac.nz

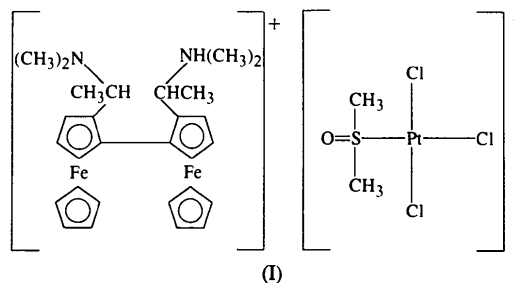
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Abstract

The structure of the title compound, [Fe₂(C₅H₅)₂-(C₁₈H₂₇N₂)] [PtCl₃(C₂H₆OS)], reveals an unusual twisting of the ferrocenyl moieties in the monoprotonated bis(amine) with respect to the interferrocenyl C—C bond. This deformation can be ascribed to hydrogen bonding between the ammonium proton and the unprotonated amine residue. The absolute configuration at each of the chiral C atoms is shown to be R, while the metallocene chiralities are S.

Comment

As part of our continuing investigations into the role of ferrocenyl (Ranatunge-Bandarage, Robinson & Simpson, 1994) and biferrocenylamines (Spescha, Duffy, Robinson & Simpson, 1994) as ligands for platinum with potential for enhanced antitumour activity, we attempted to substitute the complex [PtCl₂(dmsO)₂] (dmsO is dimethyl sulfoxide) with 2,2'-bis[1-(N,N-dimethylamino)ethyl]-1,1'-biferrocene. The reaction resulted in protonation of one of the amino groups. The resulting cation was isolated as the trichloro(dimethyl sulfoxide)platinate(II) salt, (I), and is the subject of this report.



The most unusual feature of the biferrocenyl moiety [Fig. 1(a) and Table 2] is the considerable twist of the cyclopentadiene rings of the fulvalene bridge unit. The angle between the C1—C5 and C11—C15 ring planes is 103.2 (8)°, and the separation of the N atoms of the di-