Priority Areas 'New Development of Rare Earth Complexes' (No. 07230101). The authors are also indebted to Dr C. Kabuto for the discussion of the steric structure of (I) which was initially made for compound (II).

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: TA1110). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis[bis(triphenylphosphoranylidene)ammonium] Pentachlorofluoroosmate(IV) Hydrate, $[(C_6H_5)_3P{=}N{=}P(C_6H_5)_3]_2[OsFCl_5].H_2O$

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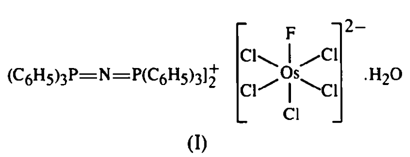
Abstract

The structure of the title compound, $(C_{36}H_{30}NP_2)_2[OsCl_5F].H_2O$, has been determined by single-crystal X-ray diffraction at 208 K. The octahedral $[OsCl_5F]^{2-}$ complex anions are completely ordered in an A_2B-type lattice. The monoclinic unit cell contains four formula units, each with a solvent water molecule linked to the F atom by a hydrogen bond [O···F 2.737 (8) Å]. The Os—F bond [1.960 (4) Å] is weakened by *trans* influence and, correspondingly, the Os—Cl1 bond [2.307 (2) Å] *trans* to Os—F is strengthened compared with the average Os—Cl bond length along the Cl—Os—Cl axes of 2.339 Å.

Comment

We are currently studying the spectroscopic properties of inorganic octahedral complexes. The present work was performed in order to confirm the results obtained from vibrational and NMR spectroscopy for the heteroleptic series $[MCl_{6-n}F_n]^{2-}$ (M = Os, Ir, Pt; n = 1–5; Preetz & Ruf, 1986; Alyoubi, Greenslade, Foster & Preetz, 1990; Bruhn, Drews, Meynhardt & Preetz, 1995). Until recently, structure analyses of mixed-halogeno complexes have failed because of total or partial disorder in the packing of the complexes in the anion sublattice. Using the twofold organic cation *N,N'*-methylenedipyridinium (Brüdgam & Hartl, 1986), which forms *AB*-type salts, ordered structures of all compounds of the series $[OsCl_{6-n}F_n]^{2-}$ (n = 2–5) became accessible, with each Os—F and Os—Cl bond clearly distinguishable (Bruhn & Preetz, 1994*a*,*b*, 1995*a*,*b*, 1996). Unfortunately, the *N,N'*-methylenedipyridinium salt of $[OsCl_5F]^{2-}$ crystallizes in space group $P\bar{1}$, with the central ion located on the inversion center causing disorder in the F—Os—Cl axis. This problem is overcome by crystallizing the mixed-ligand complex with bis(triphenylphosphoranylidene)ammonium as cation to give a completely ordered A_2B-type lattice. In the course of our work on mixed F/Cl complexes of Os^{IV}, we isolated the title complex, (I), by ion-exchange chromatography.



$$[(C_6H_5)_3P{=}N{=}P(C_6H_5)_3]_2^+\ [OsCl_5F]^{2-}.H_2O$$

(I)

The title compound crystallizes in space group $P2_1/c$, with all atoms residing in general positions. The Os atom has an octahedral coordination consisting of one F and five Cl ligands. These ligands form one asymmetric F—Os—Cl1 axis and the symmetric Cl2—Os—Cl3 and Cl4—Os—Cl5 axes. According to the *trans* influence (Preetz, Peters & Bublitz, 1996), the Os—F bond [1.960 (4) Å] is lengthened and the Os—Cl1 bond [2.307 (2) Å] shortened compared with the Os—F and Os—Cl distances in *trans*-$[OsCl_4F_2]^{2-}$ (1.926 and 2.340 Å, respectively) (Bruhn & Preetz, 1996). A comparison of the Os—Cl1 distance with the averaged value of the symmetric Cl—Os—Cl axes of 2.339 Å reveals a shortening of 0.032 Å (1.4%). The F—Os—Cl angles are 2–3° less than the expected value of 90°, which is a result of the larger atomic radius of chlorine compared with fluorine. A view of (I) with the atom labelling is shown in Fig. 1.

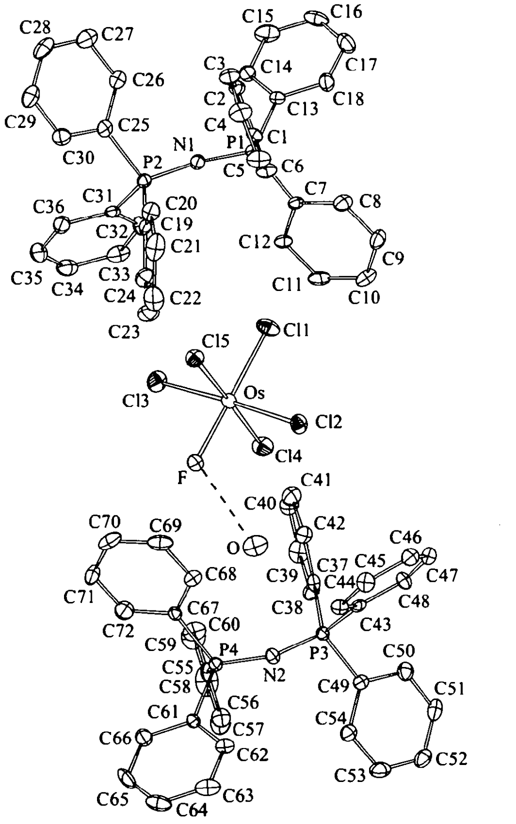


Fig. 1. A view of an asymmetric unit of (I) with the atomic labeling scheme. Ellipsoids represent 30% probability and H atoms have been omitted for clarity.

The unit cell contains two crystallographically independent cations. The P—N—P angles are 136.8 (4) and 139.1 (5)°. Additionally, the unit cell contains one water molecule per formula unit. This water molecule is linked by a weak hydrogen bond to the F atom of the complex anion, with an F···O distance of 2.737 (8) Å.

Experimental

The title complex was prepared by reaction of $(^nBu_4N)_2$-$[OsCl_6]$ with BrF_3 in a frigene/dichloromethane solution at 273 K, yielding a mixture of different partly-fluorinated complexes which were separated by ion-exchange chromatography on diethylaminoethylcellulose (Preetz, Ruf & Tensfeldt, 1984). Crystals of the title compound were obtained by treatment of the tetramethylammomium complex salt with bis(triphenylphosphine)ammonium chloride in *N*,*N*-dimethylformamide and recrystallization was from *N*,*N*-dimethylformamide/ether solution.

Crystal data

$(C_{36}H_{30}NP_2)_2[OsCl_5F].H_2O$
$M_r = 1481.653$
Monoclinic
$P2_1/c$
$a = 21.374\ (6)$ Å
$b = 13.072\ (8)$ Å
$c = 23.771\ (12)$ Å
$\beta = 105.11\ (3)°$
$V = 6412\ (5)$ Å^3
$Z = 4$
$D_x = 1.536$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 1.77–10.10°$
$\mu = 2.349$ mm^{-1}
$T = 208\ (2)$ K
Prismatic
0.3 × 0.2 × 0.2 mm
Orange

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
$T_{min} = 0.56$, $T_{max} = 0.63$
11 596 measured reflections
11 272 independent reflections

6705 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.0351$
$\theta_{max} = 24.99°$
$h = 0 \rightarrow 25$
$k = 0 \rightarrow 15$
$l = -28 \rightarrow 27$
3 standard reflections frequency: 240 min intensity decay: 0.5%

Refinement

Refinement on F^2
$R(F) = 0.0479$
$wR(F^2) = 0.1060$
$S = 1.162$
11 268 reflections
782 parameters
Water H atoms were refined with bond-length restraints, other H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0135P)^2 + 22.6947P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.178$ e Å^{-3}
$\Delta\rho_{min} = -0.728$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.00034 (2)
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 (Å²)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Os	0.759170 (15)	0.50441 (3)	0.009470 (13)	0.02621 (9)
Cl1	0.77988 (10)	0.5021 (2)	0.10968 (8)	0.0444 (5)
Cl2	0.65692 (10)	0.5785 (2)	−0.00170 (9)	0.0398 (5)
Cl3	0.86010 (11)	0.4313 (2)	0.01531 (11)	0.0482 (6)
Cl4	0.71226 (12)	0.3415 (2)	0.00371 (10)	0.0450 (6)
Cl5	0.80401 (10)	0.6684 (2)	0.01136 (9)	0.0382 (5)
F	0.7421 (2)	0.5060 (4)	−0.0756 (2)	0.0372 (10)
N1	0.9262 (3)	0.9011 (5)	0.1904 (3)	0.028 (2)
P1	0.87204 (11)	0.9464 (2)	0.21798 (10)	0.0228 (5)
P2	0.96090 (10)	0.9283 (2)	0.14088 (9)	0.0240 (5)
C1	0.8618 (4)	1.0822 (6)	0.2098 (4)	0.026 (2)
C2	0.9094 (4)	1.1449 (6)	0.2445 (3)	0.029 (2)
C3	0.9095 (5)	1.2485 (6)	0.2336 (4)	0.041 (2)
C4	0.8627 (5)	1.2923 (6)	0.1891 (4)	0.044 (2)
C5	0.8147 (5)	1.2303 (7)	0.1554 (4)	0.042 (3)
C6	0.8135 (4)	1.1267 (7)	0.1654 (4)	0.037 (2)
C7	0.7946 (4)	0.8882 (6)	0.1885 (3)	0.025 (2)
C8	0.7402 (4)	0.9285 (6)	0.2010 (4)	0.034 (2)
C9	0.6812 (4)	0.8790 (7)	0.1825 (4)	0.040 (2)
C10	0.6764 (4)	0.7892 (7)	0.1520 (4)	0.041 (2)
C11	0.7290 (4)	0.7491 (7)	0.1378 (4)	0.040 (2)
C12	0.7884 (4)	0.7988 (6)	0.1560 (4)	0.032 (2)
C13	0.8948 (4)	0.9172 (6)	0.2944 (3)	0.024 (2)
C14	0.9528 (4)	0.8682 (7)	0.3190 (3)	0.032 (2)
C15	0.9699 (5)	0.8445 (7)	0.3773 (4)	0.045 (3)
C16	0.9280 (5)	0.8660 (7)	0.4109 (4)	0.045 (2)
C17	0.8696 (5)	0.9137 (8)	0.3873 (4)	0.047 (3)
C18	0.8539 (4)	0.9393 (7)	0.3289 (4)	0.038 (2)
C19	0.9118 (3)	0.9896 (7)	0.0769 (3)	0.028 (2)
C20	0.9027 (4)	1.0958 (7)	0.0757 (4)	0.037 (2)
C21	0.8625 (5)	1.1427 (8)	0.0283 (5)	0.051 (3)
C22	0.8311 (5)	1.0849 (10)	−0.0190 (5)	0.061 (3)
C23	0.8400 (4)	0.9809 (10)	−0.0183 (4)	0.061 (3)
C24	0.8798 (4)	0.9331 (8)	0.0295 (4)	0.044 (2)
C25	1.0312 (3)	1.0092 (7)	0.1680 (3)	0.028 (2)
C26	1.0564 (4)	1.0196 (6)	0.2275 (4)	0.031 (2)
C27	1.1125 (4)	1.0779 (7)	0.2491 (4)	0.044 (2)
C28	1.1414 (4)	1.1257 (7)	0.2111 (5)	0.049 (3)
C29	1.1165 (4)	1.1182 (7)	0.1525 (5)	0.046 (3)
C30	1.0604 (4)	1.0585 (7)	0.1307 (4)	0.036 (2)
C31	0.9864 (3)	0.8092 (6)	0.1156 (3)	0.024 (2)
C32	0.9598 (4)	0.7171 (6)	0.1290 (3)	0.031 (2)
C33	0.9731 (4)	0.6275 (7)	0.1052 (4)	0.043 (2)
C34	1.0141 (4)	0.6265 (7)	0.0683 (4)	0.044 (2)
C35	1.0411 (4)	0.7166 (8)	0.0555 (4)	0.046 (3)
C36	1.0267 (4)	0.8079 (7)	0.0790 (4)	0.038 (2)
N2	0.5676 (3)	0.0871 (5)	−0.1829 (3)	0.0254 (15)
P3	0.54007 (9)	0.0568 (2)	−0.12957 (9)	0.0231 (4)
P4	0.62522 (10)	0.0517 (2)	−0.20895 (9)	0.0226 (5)
C37	0.5983 (3)	0.0101 (7)	−0.0662 (3)	0.024 (2)
C38	0.6093 (4)	−0.0948 (7)	−0.0579 (4)	0.035 (2)
C39	0.6605 (5)	−0.1269 (8)	−0.0106 (4)	0.049 (3)
C40	0.6973 (5)	−0.0551 (10)	0.0250 (4)	0.047 (3)
C41	0.6870 (5)	0.0464 (9)	0.0154 (5)	0.052 (3)
C42	0.6369 (4)	0.0800 (7)	−0.0301 (4)	0.033 (2)
C43	0.5081 (3)	0.1716 (6)	−0.1059 (3)	0.025 (2)
C44	0.5121 (4)	0.2631 (6)	−0.1330 (3)	0.027 (2)
C45	0.4928 (4)	0.3527 (6)	−0.1113 (4)	0.034 (2)
C46	0.4683 (4)	0.3490 (6)	−0.0624 (4)	0.031 (2)
C47	0.4638 (4)	0.2576 (7)	−0.0361 (4)	0.034 (2)
C48	0.4828 (4)	0.1682 (6)	−0.0570 (3)	0.030 (2)
C49	0.4744 (4)	−0.0335 (6)	−0.1516 (3)	0.026 (2)
C50	0.4448 (4)	−0.0783 (6)	−0.1120 (4)	0.034 (2)
C51	0.3923 (4)	−0.1413 (7)	−0.1316 (4)	0.042 (2)
C52	0.3670 (4)	−0.1581 (7)	−0.1894 (4)	0.045 (2)
C53	0.3941 (4)	−0.1158 (7)	−0.2295 (4)	0.046 (2)
C54	0.4479 (4)	−0.0519 (6)	−0.2115 (4)	0.033 (2)
C55	0.6481 (4)	−0.0809 (6)	−0.1952 (4)	0.025 (2)
C56	0.6088 (5)	−0.1561 (7)	−0.2273 (4)	0.043 (2)
C57	0.6206 (5)	−0.2566 (7)	−0.2120 (5)	0.058 (3)
C58	0.6723 (6)	−0.2851 (8)	−0.1665 (5)	0.065 (3)
C59	0.7113 (6)	−0.2106 (8)	−0.1353 (5)	0.058 (3)
C60	0.6987 (5)	−0.1087 (7)	−0.1492 (4)	0.044 (2)
C61	0.5984 (4)	0.0717 (6)	−0.2868 (3)	0.023 (2)
C62	0.5388 (4)	0.1154 (6)	−0.3107 (3)	0.030 (2)
C63	0.5194 (5)	0.1338 (7)	−0.3695 (4)	0.043 (3)
C64	0.5584 (5)	0.1148 (7)	−0.4046 (4)	0.047 (3)
C65	0.6188 (5)	0.0722 (7)	−0.3806 (4)	0.045 (3)
C66	0.6389 (4)	0.0494 (7)	−0.3221 (4)	0.039 (2)
C67	0.6967 (4)	0.1277 (6)	−0.1829 (3)	0.022 (2)
C68	0.6942 (5)	0.2176 (7)	−0.1528 (4)	0.037 (2)
C69	0.7473 (5)	0.2816 (7)	−0.1376 (4)	0.042 (2)
C70	0.8029 (4)	0.2568 (7)	−0.1524 (4)	0.046 (2)
C71	0.8070 (4)	0.1673 (8)	−0.1805 (4)	0.044 (2)
C72	0.7550 (4)	0.1031 (7)	−0.1961 (4)	0.041 (2)
O	0.6178 (4)	0.4662 (7)	−0.1384 (3)	0.084 (3)

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

Os—F	1.960 (4)	Os—Cl4	2.342 (3)
Os—Cl1	2.307 (2)	Os—Cl5	2.344 (3)
Os—Cl3	2.330 (2)	F—O	2.737 (8)
Os—Cl2	2.341 (2)		
F—Os—Cl1	179.63 (14)	Cl2—Os—Cl4	89.81 (9)
F—Os—Cl3	89.20 (15)	F—Os—Cl5	88.6 (2)
Cl1—Os—Cl3	90.44 (9)	Cl1—Os—Cl5	91.41 (9)
F—Os—Cl2	87.80 (14)	Cl3—Os—Cl5	90.36 (9)
Cl1—Os—Cl2	92.56 (8)	Cl2—Os—Cl5	89.37 (9)
Cl3—Os—Cl2	176.99 (9)	Cl4—Os—Cl5	177.75 (8)
F—Os—Cl4	89.3 (2)	P1—N1—P2	139.1 (5)
Cl1—Os—Cl4	90.72 (9)	P3—N2—P4	136.8 (4)
Cl3—Os—Cl4	90.35 (9)		

The major residual features in the ΔF synthesis lie close to the metal atom.

Data collection: *CAD*-4-*PC* (Enraf–Nonius, 1994). Cell refinement: *CAD*-4-*PC*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1142). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). C**53**, 66–67

[1,2-Bis(diphenylphosphino)ethane-*P*,*P*′]-bis(α-toluenethiolato-*S*)palladium(II), $[Pd\{Ph_2P(CH_2)_2PPh_2\}(SCH_2Ph)_2]$

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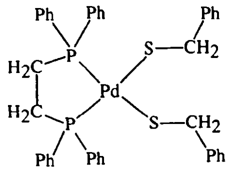
Abstract

The title compound, $[Pd(C_7H_7S)_2(C_{26}H_{24}P_2)]$, is a mononuclear palladium(II) complex. The molecule possesses a crystallographic twofold axis and the Pd atom is four-coordinated by two phosphine P atoms and two S atoms from $PhCH_2S^-$ ligands, and has a distorted square-planar geometry. The Pd—S and Pd—P distances are 2.360 (2) and 2.277 (2) Å, respectively.

Comment

Transition metal compounds with mixed sulfur and phosphine ligands have attracted much attention due to their relevance and importance to a wide variety of chemical and industrial systems. Of the nickel group metals, many nickel compounds with such mixed ligands have been reported. Surprisingly few palladium compounds, such as $[Pd_2(SC_6F_5)_2(PPh_3)_2]$ (Fenn & Segrott, 1972), have been structurally characterized. We have recently reported the palladium compounds $[Pd(SCH_2CH_2SCH_2CH_2S)(PPh_3)_2]$ and $[Pd_2(PPh_3)_2(HOC_6H_4S)_2Cl_2]$ (Cao, Hong, Jiang, Xie & Liu, 1996), $[Pd_2(PPh_3)_2(SC_2H_4S)_2]$ (Cao, Hong, Jiang & Liu, 1995) and $[Pd\{Ph_2P(CH_2)_3PPh_2\}(SC_3H_6S)].CH_3CN$ (Su, Hong, Zhou, Xue, Liu & Mak, 1996). We report here the crystal structure of a mononuclear complex, $[Pd\{Ph_2P(CH_2)_2PPh_2\}(SCH_2Ph)_2]$, (I).



(I)

The title compound is a mononuclear palladium(II) complex, where the Pd atom is four-coordinated by two phosphine P atoms and two S atoms from two $PhCH_2S^-$ ligands, and has a distorted square-planar geometry. The molecule possesses a crystallographic twofold axis passing through the Pd atoms and the midpoint of C(1)—C(1^i) [symmetry code: (i) $1-x$, $-y$, z]. The Pd(1), P(1), P(1^i), S(1) and S(1^i) atoms are in a plane with displacements of 0.0000, −0.087, 0.085, 0.073 and −0.070 Å, respectively. The Pd—S and Pd—P distances are 2.360 (2) and 2.277 (2) Å, respectively. The structure of the title compound is depicted in Fig. 1.

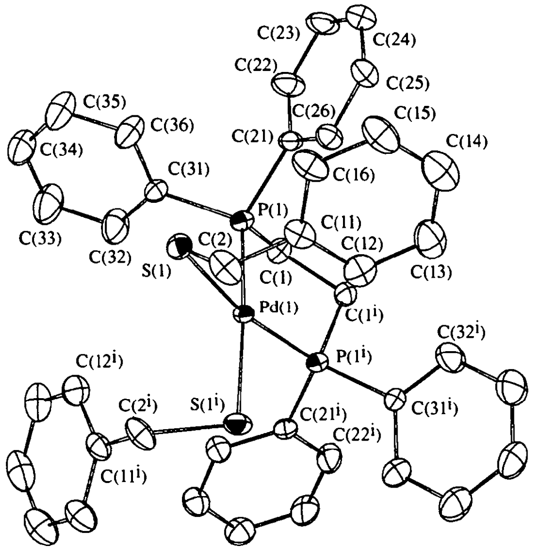


Fig. 1. The structure of $[Pd\{Ph_2P(CH_2)_2PPh_2\}(SCH_2S)_2]$ with displacement ellipsoids at the 30% probability level.

Experimental

The title compound was obtained from the reaction of $PdCl_2$, $NaSCH_2Ph$ and $Ph_2P(CH_2)_2PPh_2$ (molar ratio 1:2:1) in MeOH, and recrystallized from CH_3CN solution.

Crystal data

$[Pd(C_7H_7S)_2(C_{26}H_{24}P_2)]$
$M_r = 751.23$
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å