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# Structure of *trans*-Dichlorobis[1,2-bis-(diphenylphosphino)ethane]osmium(II) Dichloromethane Solvate

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## Abstract

The structure consists of discrete *trans* octahedral molecules with Os—Cl = 2.434 (1) Å and Os—P = 2.372 (1) and 2.348 (1) Å.

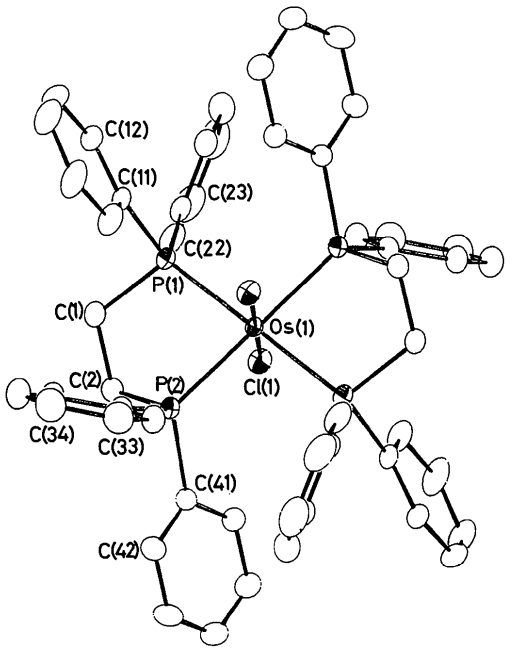


Fig. 1. View of $[OsCl_2(C_{26}H_{24}P_2)_2].CH_2Cl_2$ showing the atom-labelling scheme. H atoms are excluded and the thermal ellipsoids are drawn at the 30% probability level.

## Comment

Crystals of the title compound were obtained during studies of the effects of ligand set and metal stereochemistry on the relative stabilities of osmium(II), -(III) and -(IV) complexes (Champness, Levason, Pletcher, Spicer & Webster, 1992). The structure consists of discrete *trans* octahedral molecules with the Os atom located on a centre of symmetry. The geometry is very similar to that of *trans*-$[Ru(Ph_2PCH_2CH_2PPh_2)_2Cl_2]$ (Lobana, Singh & Tiekink, 1990), for which Ru—Cl = 2.436 (1), Ru—P = 2.389 (1), 2.369 (1) Å and P—Ru—P = 82.1 (1)°. Comparison may also be made with an Os complex containing a four-membered chelate ring, $[Os\{H_2C{=}C(PPh_2)_2\}_2Cl_2]$, for which Os—Cl = 2.431 (1), Os—P = 2.343 (1), 2.330 (1) Å and P—Os—P = 72.74 (3)° (Cotton, Diebold & Matusz, 1987).

## Experimental

*Crystal data*

$[OsCl_2(C_{26}H_{24}P_2)_2].CH_2Cl_2$
$M_r$ = 1142.9
Triclinic
$P\bar{1}$
$a$ = 10.065 (2) Å
$b$ = 10.437 (2) Å
$c$ = 12.974 (4) Å
$\alpha$ = 68.22 (2)°
$\beta$ = 70.69 (2)°
$\gamma$ = 88.31 (2)°
$V$ = 1187.6 (6) Å$^3$
$Z$ = 1
$D_x$ = 1.597 Mg m$^{-3}$
$D_m$ = 1.57 (2) Mg m$^{-3}$
Density measured by flotation in $CCl_4$-$C_6H_{14}$
Mo $K\alpha$ radiation
$\lambda$ = 0.71069 Å
Cell parameters from 24 reflections
$\theta$ = 13.0 - 17.3°
$\mu$ = 3.08 mm$^{-1}$
$T$ = 293 (3) K
Air-stable blocks
0.40 × 0.22 × 0.18 mm
Pale yellow

*Data collection*

Enraf-Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical
$T_{min}$ = 0.91, $T_{max}$ = 1.00
4947 measured reflections
4666 independent reflections
4644 observed reflections [$F > 3\sigma(F)$]
$R_{int}$ = 0.011
$\theta_{max}$ = 26.0°
$h$ = 0 → 12
$k$ = −12 → 12
$l$ = −15 → 15
1 standard reflection
frequency: 60 min
intensity variation: 3.5%

*Refinement*

Refinement on $F$
Final $R$ = 0.0231
$wR$ = 0.0313
$S$ = 1.27
4639 reflections
278 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.0004F^2]$
$(\Delta/\sigma)_{max}$ = 0.2
$\Delta\rho_{max}$ = 1.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.02 e Å$^{-3}$
Atomic scattering factors from *SHELX*76 (Sheldrick, 1976) (C, H, P, Cl) and *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1) (Os)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å$^2$)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

| | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| Os1 | 0.0000 | 0.0000 | 0.0000 | 0.0238 (2) |
| Cl1 | 0.2577 (1) | 0.0244 (1) | −0.0760 (1) | 0.0360 (6) |
| P1 | 0.0343 (1) | 0.1507 (1) | 0.0901 (1) | 0.0287 (6) |
| P2 | 0.0212 (1) | 0.2115 (1) | −0.1584 (1) | 0.0311 (6) |
| C1 | 0.1064 (4) | 0.3268 (3) | −0.0266 (3) | 0.0413 (28) |
| C2 | 0.1454 (3) | 0.3310 (3) | −0.1521 (3) | 0.0385 (26) |
| C11 | −0.1125 (3) | 0.2036 (3) | 0.1900 (3) | 0.0336 (25) |
| C12 | −0.1016 (4) | 0.2347 (3) | 0.2828 (3) | 0.0440 (30) |
| C13 | −0.2149 (5) | 0.2809 (4) | 0.3503 (3) | 0.0594 (39) |
| C14 | −0.3387 (4) | 0.2980 (4) | 0.3250 (4) | 0.0651 (44) |
| C15 | −0.3484 (4) | 0.2736 (4) | 0.2307 (4) | 0.0631 (46) |
| C16 | −0.2361 (4) | 0.2270 (4) | 0.1628 (4) | 0.0500 (36) |
| C21 | 0.1623 (3) | 0.1060 (3) | 0.1676 (3) | 0.0359 (27) |
| C22 | 0.3053 (4) | 0.1476 (4) | 0.1047 (3) | 0.0488 (36) |
| C23 | 0.4060 (4) | 0.1031 (5) | 0.1581 (5) | 0.0665 (52) |
| C24 | 0.3626 (5) | 0.0161 (5) | 0.2765 (5) | 0.0764 (64) |
| C25 | 0.2211 (5) | −0.0228 (4) | 0.3406 (4) | 0.0656 (48) |
| C26 | 0.1194 (4) | 0.0200 (3) | 0.2866 (3) | 0.0456 (33) |
| C31 | −0.1373 (3) | 0.3014 (3) | −0.1578 (3) | 0.0375 (26) |
| C32 | −0.2537 (4) | 0.2338 (4) | −0.1589 (3) | 0.0469 (32) |
| C33 | −0.3776 (4) | 0.2947 (4) | −0.1558 (4) | 0.0589 (40) |
| C34 | −0.3882 (5) | 0.4238 (5) | −0.1510 (4) | 0.0674 (46) |
| C35 | −0.2740 (5) | 0.4928 (4) | −0.1498 (4) | 0.0633 (43) |
| C36 | −0.1485 (4) | 0.4336 (4) | −0.1543 (3) | 0.0498 (34) |
| C41 | 0.0996 (4) | 0.2277 (3) | −0.3123 (3) | 0.0383 (26) |
| C42 | 0.0510 (5) | 0.3154 (4) | −0.4010 (3) | 0.0564 (36) |
| C43 | 0.1111 (6) | 0.3214 (5) | −0.5156 (3) | 0.0721 (45) |
| C44 | 0.2175 (5) | 0.2423 (4) | −0.5438 (3) | 0.0661 (40) |
| C45 | 0.2666 (4) | 0.1557 (4) | −0.4574 (3) | 0.0542 (35) |
| C46 | 0.2079 (4) | 0.1486 (3) | −0.3423 (3) | 0.0434 (29) |
| Cl2*S*† | 0.5340 (4) | 0.3950 (4) | 0.5889 (2) | 0.1906 (51) |
| C3*S*‡ | 0.4164 | 0.4751 | 0.5144 | 0.0600 |

† Site occupancy refined to 0.928 (6).
‡ Site occupancy refined to 0.464 (3).

Table 2. *Geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| Os1—Cl1 | 2.434 (1) | P1—C21 | 1.832 (3) |
| Os1—P1 | 2.372 (1) | P2—C2 | 1.833 (3) |
| Os1—P2 | 2.348 (1) | P2—C31 | 1.826 (3) |
| P1—C1 | 1.867 (3) | P2—C41 | 1.831 (3) |
| P1—C11 | 1.839 (3) | | |
| Cl1—Os1—P1 | 83.1 (1) | C1—P1—C11 | 97.7 (1) |
| Cl1—Os1—P2 | 85.2 (1) | C1—P1—C21 | 104.3 (1) |
| P1—Os1—P2 | 81.9 (1) | C11—P1—C21 | 103.3 (1) |
| Os1—P1—C1 | 109.0 (1) | C2—P2—C31 | 104.8 (1) |
| Os1—P1—C11 | 123.1 (1) | C2—P2—C41 | 101.2 (1) |
| Os1—P1—C21 | 116.5 (1) | C31—P2—C41 | 102.8 (1) |
| Os1—P2—C2 | 106.4 (1) | P1—C1—C2 | 113.4 (2) |
| Os1—P2—C31 | 117.7 (1) | P2—C2—C1 | 110.6 (2) |
| Os1—P2—C41 | 121.7 (1) | | |
| P1—C1—C2—P2 | 35.4 (3) | | |

Crystals were grown by vapour diffusion of $Et_2O$ into a $CH_2Cl_2$ solution of the compound and mounted in glass capillaries. Five reflections thought to be suffering from extinction were excluded. For the disordered solvent molecule ($CH_2Cl_2$), isotropic C3*S* was not refined and no H atoms were included. The remaining H-atom positions were determined from electron-density maps but not refined. Data collection: *Enraf–Nonius Structure Determination Package* (Frenz, 1985). Cell refinement: *Enraf–Nonius Structure Determination Package*. Data reduction: *Enraf–Nonius Structure Determination Package*. Program(s) used to solve structure: *SHELX*76 (Sheldrick, 1976). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71319 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1057]

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# Structures of Two Encapsulated Cobalt(III) Complexes Containing *cyclo*-Triphosphate

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## Abstract

The conformational and packing properties of two encapsulated cobalt(III) complexes containing *cyclo*-triphosphate have been investigated. Compound (I),