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trans-Tetrachlorobis(triphenylphosphine)osmium(IV)

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The structure of the title compound, $[OsCl_4(C_{18}H_{15}P)_2]$, is centrosymmetric. The geometry of the Os^{IV} center is octahedral with the P atoms occupying mutually *trans*-positions.

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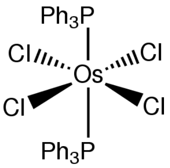
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.008 Å
R factor = 0.032
wR factor = 0.078
Data-to-parameter ratio = 19.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Osmium(IV) complexes of the type *trans*-$[Os(PR_3)_2X_4]$ (X = Cl or Br) have been characterized spectroscopically (Salmon & Walton, 1978; Cipriano *et al.*, 1990), but only the $Os^{IV}(PMe_2Ph)_2Cl_4$ species has been structurally characterized (Aslanov *et al.*, 1970). In our preparation of $OsCl_2(PPh_3)_3$ from the reaction of K_2OsCl_6 with PPh_3 according to the literature method of Hoffmann & Caulton (1975), the title compound, (I), was also formed and subsequently characterized crystallographically.



(I)

The structure of (I) (Fig. 1) is centrosymmetric. The geometry around the Os^{IV} center is octahedral with two P atoms in mutually *trans* positions. The Os$\cdots$Cl distances [mean 2.313 (6) Å] are experimentally equivalent to those reported for the $Os^{IV}(PMe_2Ph)_2Cl_4$ structure [mean 2.319 (3) Å; Aslanov *et al.*, 1970], but significantly shorter than those for $[Os^{III}(PEt_3)_2Cl_4]^-$ [mean 2.380 (2) Å; Kim *et al.*, 1984], as expected.

Experimental

The title compound was obtained as a by-product when $Os(PPh_3)_3Cl_2$ was prepared according to the literature method (Hoffmann & Caulton, 1975). A mixture of K_2OsCl_6 and PPh_3 (1:7) in 2-propanol and H_2O (2:1) was refluxed for 4 h to give a green precipitate, which was collected and extracted with benzene. A small amount of benzene insoluble orange-red solid was collected by filtration and recrystallized from a CH_2Cl_2 solution layered with hexane to afford orange–red crystals suitable for X-ray structure analysis.

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Crystal data

$[OsCl_4(C_{18}H_{15}P)_2]$
$M_r = 856.54$
Monoclinic, $P2_1/n$
$a = 9.3364$ (9) Å
$b = 20.948$ (2) Å
$c = 9.6251$ (9) Å
$\beta = 117.425$ (2)°
$V = 1670.9$ (3) Å^3
$Z = 2$
$D_x = 1.702$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3136 reflections
$\theta = 1.0$–27.5°
$\mu = 4.26$ mm^{-1}
$T = 294$ (2) K
Prism, orange–red
0.20 × 0.16 × 0.10 mm

Data collection

Bruker CCD area-detector diffractometer
φ and ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.483$, $T_{\max} = 0.676$
11 100 measured reflections
3844 independent reflections
2867 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.044$
$\theta_{\max} = 27.6°$
$h = -6 \rightarrow 12$
$k = -25 \rightarrow 27$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.078$
$S = 0.98$
3844 reflections
194 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0409P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.98$ e Å^{-3}
$\Delta\rho_{\min} = -0.60$ e Å^{-3}

The C-bound H atoms were placed in their geometrically calculated positions and included in the final refinement in the riding model approximation.

Data collection: Bruker *SMART* (Bruker, 1999); cell refinement: Bruker *SMART*; data reduction: Bruker *SHELXTL-NT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: Bruker *SHELXTL-NT*; software used to prepare material for publication: Bruker *SHELXTL-NT*.

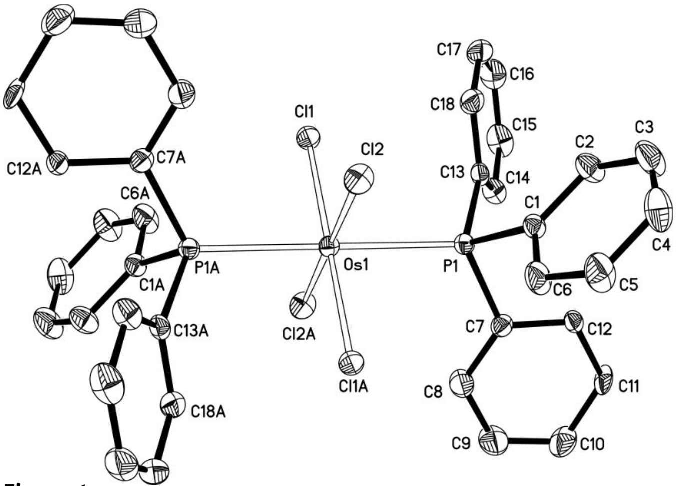

Figure 1
Molecular structure of (I) with displacement ellipsoids shown at the 30% probability level (*SHELXTL-NT*; Bruker, 1999). Symmetry operation for atoms labeled with A: $-x$, $1 - y$, $-z$.

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