

Dichloro(η^6 -*p*-cymene)[η^1 -(2,4,6-tri-*tert*-butylphenyl)phosphine- κP]osmium(II)Bruno Therrien^{a*} and Anthony K. Burrell^b^aInstitut de Chimie, Université de Neuchâtel, Case postale 2, CH-2007 Neuchâtel, Switzerland, and ^bActinide, Catalysis and Separations Chemistry, C-SIC, Mail Stop J514, Los Alamos National Laboratory, Los Alamos, NM 87545, USA

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Key indicators

Single-crystal X-ray study

T = 150 K

Mean $\sigma(C-C)$ = 0.007 Å

Disorder in main residue

R factor = 0.031

wR factor = 0.081

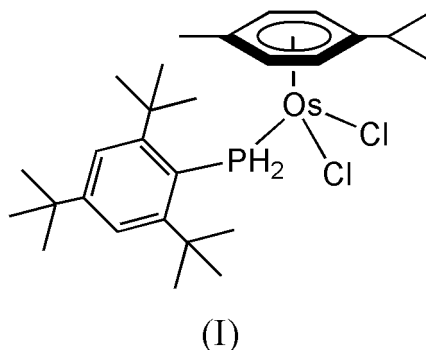
Data-to-parameter ratio = 19.3

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

In the title compound, [Os(C₁₀H₁₄)Cl₂(C₁₈H₃₁P)], the presence of bulky substituents imposes a structural distortion on the (2,4,6-tri-*tert*-butylphenyl)phosphine ligand, which is bowed.

Comment

The title complex, (I), has previously been synthesized and used as a starting material to form terminal phosphinidene complexes, but it has not been structurally characterized (Termaten *et al.*, 2003). We report here a modified synthesis and the single-crystal structure analysis of (I), a sterically hindered complex. The compound was prepared quantitatively from [OsCl₂(*p*-cymene)]₂ (Kiel *et al.*, 1990) and (2,4,6-tri-*tert*-butylphenyl)PH₂ (Cowley *et al.*, 1990), following the procedure described for the synthesis of [RuCl₂(η^1 -PH₂Cy)(η^6 -*p*-cymene)] (Van der Maelen Uría *et al.*, 1994).



The *p*-cymene ring can be considered planar [the maximum deviation of atoms C2–C7 from the mean plane is 0.009 (3) Å]. The isopropyl group is bent towards the Os atom [the deviation from the plane is 0.100 (8) Å for C8], while the methyl is bent away [the deviation from the ring plane is 0.056 (7) Å for C1]. The distance between the centroid of the *p*-cymene ring and the Os atom is 1.701 (12) Å. The molecular structure and the atomic numbering scheme are presented in Fig. 1. Selected bond lengths and angles are given in Table 1.

The presence of *tert*-butyl groups on the arene substituent of the phosphine generates a significant distortion of the ligand. To minimize the interaction with the chloro and *p*-cymene ligands, the phosphine ligand is bowed. Atoms P and C25 are, respectively, 0.985 (5) and 0.278 (8) Å above the C11–C16 aromatic plane [the maximum deviation of atoms C11–C16 from the mean plane is 0.069 (3) Å]. The two other *tert*-butyl groups, involving atoms C17 and C21, are bent away from the Os atom, by, respectively, 0.425 (7) and 0.458 (7) Å from the C11–C16 plane.

Experimental

To a benzene solution of $[\text{OsCl}_2(p\text{-cymene})]_2$ (154 mg, 0.22 mmol) were added 2 equivalents of (2,4,6-tri-*tert*-butylphenyl)phosphine (144 mg, 0.44 mmol). The solution was stirred overnight and the volatile substances were removed under vacuum. The solid was washed with cold diethyl ether to give (I) quantitatively. Crystals suitable for X-ray diffraction were grown by slow diffusion of pentane into a dichloromethane solution. $^1\text{H NMR}$ (CDCl_3): δ 7.55 (s, 2H), 6.56 (d, 2H, $^1J_{\text{PH}} = 392$ Hz), 5.37 (d, 2H), 5.07 (d, 2H), 2.63 (sept, 1H), 2.06 (s, 3H), 1.54 (s, 18H), 1.36 (s, 9H), 1.24 (d, 6H); $^{31}\text{P NMR}$ (CDCl_3): δ -55.7.

Crystal data

$[\text{Os}(\text{C}_{10}\text{H}_{14})\text{Cl}_2(\text{C}_{18}\text{H}_{31}\text{P})]$
 $M_r = 673.71$
 Monoclinic, $C2/c$
 $a = 15.3347$ (2) Å
 $b = 13.0280$ (2) Å
 $c = 31.6125$ (4) Å
 $\beta = 102.678$ (1)°
 $V = 6161.58$ (15) Å³
 $Z = 8$

$D_x = 1.453$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 6588 reflections
 $\theta = 1.3\text{--}26.7^\circ$
 $\mu = 4.38$ mm⁻¹
 $T = 150$ (2) K
 Plate, orange
 $0.28 \times 0.20 \times 0.03$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (Blessing, 1995)
 $T_{\min} = 0.374$, $T_{\max} = 0.880$
 18 106 measured reflections

6510 independent reflections
 5767 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$
 $\theta_{\text{max}} = 26.7^\circ$
 $h = -19 \rightarrow 19$
 $k = -16 \rightarrow 10$
 $l = -37 \rightarrow 40$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.081$
 $S = 1.19$
 6510 reflections
 337 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0385P)^2 + 8.2375P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.023$
 $\Delta\rho_{\text{max}} = 1.01$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.05$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Os—C6	2.167 (4)	Os—C3	2.243 (4)
Os—C7	2.201 (4)	Os—P	2.3498 (10)
Os—C2	2.221 (4)	Os—Cl1	2.4103 (11)
Os—C5	2.223 (4)	Os—Cl2	2.4168 (10)
Os—C4	2.229 (4)		
P—Os—Cl1	83.91 (4)	Cl1—Os—Cl2	85.41 (4)
P—Os—Cl2	82.97 (4)	Cl1—P—Os	114.57 (13)

The positions of H atoms attached to P atoms were refined, with $U_{\text{iso}}(\text{H})$ values fixed at $1.2 U_{\text{eq}}(\text{P})$. Other H atoms were treated as riding, with C—H distances in the range 0.93–0.96 Å and $U_{\text{iso}}(\text{H})$ values of $1.2 U_{\text{eq}}(\text{C})$ or $1.5 U_{\text{eq}}(\text{C}_{\text{methyl}})$. One of the *tert*-butyl groups

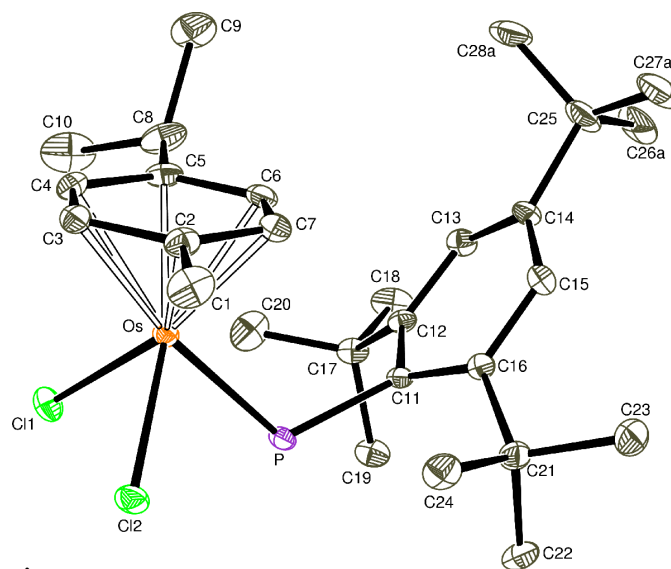


Figure 1

The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity, as has the minor disorder component of a *tert*-butyl group.

appears to be disordered over two positions (C26–C28). The occupancy factors were initially allowed to refine freely; as they were nearly in the ratio 0.7/0.3, the occupancy was then fixed at exactly 0.7/0.3.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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