

actinide elements, the structure of the uranyl complex $\{UO_2(OH_2)[(OPPh)_2Pd(S_2CNET_2)]_2\}$ has been determined (Veitch, Blake, Allan & Schröder, 1987).

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Structure of 1,2,2,2,3,3,3,4,4,4-Decacarbonyl-1,2,3,4-di- μ -hydrido-1,3-bis(tricyclohexylphosphine)-cyclo-platinumtriosmium(2 Pt–Os)(3 Os–Os)–Toluene (1/1)

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Abstract. $[Os_3Pt(\mu-H)_2(CO)_{10}\{P(C_6H_{11})_3\}_2].C_7H_8$, $M_r = 1700.8$, triclinic, $P\bar{1}$, $a = 11.427$ (2), $b = 14.842$ (3), $c = 17.815$ (2) Å, $\alpha = 93.32$ (1), $\beta = 99.60$ (1), $\gamma = 104.24$ (1)°, $V = 2872.0$ (8) Å³, $Z = 2$, $D_x = 1.97$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71069$ Å, $\mu = 91.8$ cm⁻¹, $F(000) = 1620$, $T = 298$ K, $R = 0.024$ for 7123 observed reflections. The metal atom skeleton is of the 'butterfly' form, with a non-bonding Pt...Os(1) distance of 3.506 (1) Å, and a butterfly angle, defined by the torsion angle Os(1)–Os(2)–Os(3)–Pt, of 87.5 (1)°. The two hydride ligands are *cis* to the phosphine groups, lying along Os–Os and Os–Pt edges.

Experimental. Yellow prisms from toluene solution: crystal dimensions *ca* 0.3 × 0.4 × 0.3 mm; Enraf–Nonius CAD-4F diffractometer; graphite monochromator; $\theta/2\theta$ scan mode; cell parameters refined by least-squares method on basis of 25 independent θ values, $11 \leq \theta \leq 13^\circ$; intensities measured to $\theta = 25^\circ$ over range of *hkl* 0 to 13, –17 to +17, –21 to +21; $\bar{3}\bar{6}3$, $\bar{3}2\bar{7}$, $\bar{6}26$ measured every 2 h, linear decay correction applied corresponding to 8% decay over 169 h data collection, 10 643 reflections measured, 10 008 independent data with 7123 having $I \geq 3.0\sigma(I)$ considered observed and used in structure determination and refinement; R_{int} 0.028 before and 0.027 after absorption correction; corrected for Lorentz/polarization, absorption (*DIFABS*; Stuart & Walker, 1983);

range of transmission factors on *F*, 0.823 to 1.238; solved by direct methods (*MITHRIL*; Gilmore, 1984) and subsequent full-matrix least squares; anisotropic thermal parameters for non-H atoms; H atoms included at fixed calculated positions [cyclohexyl C–H = 1.0 Å: hydrides (*HYDEX*; Orpen, 1980) Os–H, Pt–H *ca* 1.85 Å] with fixed isotropic ($U = 0.05$ Å²) thermal parameters; solvent molecule of toluene disordered over two sites (50:50), the benzene ring treated as rigid hexagon C–C = 1.395 Å: H-atom contributions from solvent not included; $w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o)]^{-1}$; max. Δ/σ 0.29, ave. 0.025; $(\Delta\rho)_{max} + 0.72$, $(\Delta\rho)_{min} - 0.74$ e Å⁻³ in vicinity of heavy metal atoms; $R = 0.024$, $wR = 0.028$; $R(wR) = 0.051$ (0.030) for all data; $S = 1.27$; atomic scattering factors including anomalous terms from *International Tables for X-ray Crystallography* (1974); calculations carried out on a Gould-SEL 32/27 minicomputer using the *GX* suite of programs (Mallinson & Muir, 1985). Final positional parameters are given in Table 1, with selected bond distances and angles in Table 2.* The molecular structure and atomic labelling scheme are shown in Fig.

* Lists of structure factors, anisotropic thermal parameters, calculated hydrogen positional parameters, and a complete listing of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44915 (59 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final positional parameters (fractional coordinates) with *e.s.d.*'s in parentheses, and equivalent isotropic thermal parameters, U_{eq} (Å²)

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	U_{eq}
Os(1)	0.44475 (2)	0.19828 (2)	0.79135 (2)	0.037
Os(2)	0.19596 (2)	0.19858 (2)	0.71846 (2)	0.036
Os(3)	0.35387 (2)	0.37178 (2)	0.77100 (1)	0.034
Pt	0.35310 (2)	0.29999 (2)	0.62614 (1)	0.034
P(1)	0.64090 (15)	0.22421 (11)	0.87428 (9)	0.039
P(2)	0.31968 (14)	0.23408 (11)	0.49912 (9)	0.035
O(1)	0.5362 (6)	0.4794 (4)	0.6232 (4)	0.090
O(2)	0.3203 (5)	0.1694 (4)	0.9307 (3)	0.085
O(3)	0.5978 (5)	0.2216 (4)	0.6628 (3)	0.077
O(4)	0.3728 (6)	-0.0102 (3)	0.7437 (4)	0.087
O(5)	0.0757 (5)	0.1841 (4)	0.8581 (3)	0.082
O(6)	0.0975 (5)	-0.0084 (3)	0.6613 (4)	0.077
O(7)	-0.0196 (5)	0.2676 (4)	0.6448 (3)	0.074
O(8)	0.2936 (5)	0.3936 (4)	0.9295 (3)	0.072
O(9)	0.5619 (5)	0.5504 (3)	0.7950 (3)	0.066
O(10)	0.1653 (5)	0.4665 (4)	0.6969 (4)	0.078
C(1)	0.4647 (7)	0.4115 (5)	0.6266 (4)	0.055
C(2)	0.3635 (7)	0.1804 (5)	0.8773 (5)	0.059
C(3)	0.5330 (7)	0.2157 (5)	0.7057 (4)	0.054
C(4)	0.4014 (7)	0.0683 (5)	0.7628 (5)	0.066
C(5)	0.1278 (6)	0.1902 (5)	0.8073 (4)	0.052
C(6)	0.1366 (6)	0.0685 (5)	0.6822 (4)	0.049
C(7)	0.0635 (7)	0.2406 (5)	0.6713 (4)	0.050
C(8)	0.3171 (6)	0.3844 (4)	0.8700 (4)	0.048
C(9)	0.4860 (6)	0.4816 (4)	0.7859 (4)	0.045
C(10)	0.2341 (7)	0.4284 (4)	0.7232 (4)	0.049
C(11)	0.6330 (6)	0.2582 (4)	0.9748 (4)	0.048
C(12)	0.6018 (7)	0.3516 (5)	0.9870 (4)	0.055
C(13)	0.5625 (8)	0.3613 (6)	1.0638 (5)	0.075
C(14)	0.6645 (8)	0.3539 (6)	1.1287 (4)	0.079
C(15)	0.7033 (8)	0.2665 (6)	1.1168 (4)	0.075
C(16)	0.7401 (7)	0.2553 (6)	1.0388 (4)	0.062
C(121)	0.6982 (6)	0.1181 (4)	0.8807 (4)	0.048
C(122)	0.6145 (8)	0.0403 (5)	0.9148 (4)	0.062
C(123)	0.6577 (9)	-0.0464 (5)	0.9186 (5)	0.081
C(124)	0.6799 (10)	-0.0802 (6)	0.8394 (6)	0.098
C(125)	0.7677 (10)	-0.0069 (6)	0.8108 (5)	0.089
C(126)	0.7240 (7)	0.0811 (5)	0.8042 (4)	0.061
C(131)	0.7669 (6)	0.3107 (5)	0.8441 (4)	0.050
C(132)	0.7371 (7)	0.3987 (5)	0.8189 (4)	0.061
C(133)	0.8381 (8)	0.4582 (5)	0.7836 (5)	0.078
C(134)	0.9593 (8)	0.4828 (7)	0.8352 (7)	0.097
C(135)	0.9905 (8)	0.3973 (8)	0.8638 (7)	0.111
C(136)	0.8921 (7)	0.3351 (6)	0.8990 (5)	0.068
C(211)	0.1969 (6)	0.2755 (4)	0.4415 (4)	0.040
C(212)	0.2265 (7)	0.3816 (5)	0.4509 (5)	0.065
C(213)	0.1270 (9)	0.4176 (6)	0.4032 (5)	0.086
C(214)	0.0033 (8)	0.3775 (6)	0.4254 (5)	0.086
C(215)	-0.0287 (7)	0.2735 (6)	0.4164 (6)	0.079
C(216)	0.0708 (7)	0.2352 (5)	0.4625 (4)	0.058
C(221)	0.4496 (6)	0.2588 (6)	0.4476 (4)	0.060
C(222)	0.5637 (8)	0.3222 (8)	0.4851 (5)	0.092
C(223)	0.6718 (6)	0.3332 (5)	0.4463 (4)	0.059
C(224)	0.6421 (8)	0.3276 (7)	0.3618 (5)	0.090
C(225)	0.5293 (8)	0.2674 (7)	0.3226 (5)	0.081
C(226)	0.4207 (6)	0.2573 (5)	0.3619 (4)	0.053
C(231)	0.2638 (6)	0.1049 (4)	0.4887 (4)	0.041
C(232)	0.2288 (6)	0.0575 (4)	0.4077 (4)	0.049
C(233)	0.1783 (7)	-0.0483 (4)	0.4067 (4)	0.058
C(234)	0.2690 (7)	-0.0909 (4)	0.4541 (5)	0.066
C(235)	0.3011 (8)	-0.0451 (5)	0.5344 (5)	0.066
C(236)	0.3539 (7)	0.0609 (5)	0.5379 (4)	0.057
C(S11)	0.1788 (15)	0.2941 (10)	0.1137 (6)	0.087 (6)
C(S12)	0.1167 (12)	0.2327 (8)	0.0491 (5)	0.090 (6)
C(S13)	0.0222 (18)	0.1553 (12)	0.0556 (7)	0.055 (5)
C(S14)	-0.0101 (12)	0.1393 (8)	0.1267 (6)	0.066 (4)
C(S15)	0.0520 (16)	0.2007 (10)	0.1913 (6)	0.092 (6)
C(S16)	0.146 (2)	0.278 (1)	0.185 (1)	0.090 (6)
C(S17)	-0.1119 (18)	0.0592 (11)	0.1313 (7)	0.11 (1)
C(S21)	-0.0843 (16)	0.0759 (11)	0.1498 (8)	0.14 (1)
C(S22)	-0.061 (3)	0.063 (2)	0.076 (1)	0.119 (8)
C(S23)	0.020 (3)	0.133 (1)	0.048 (1)	0.13 (1)
C(S24)	0.0785 (13)	0.2168 (9)	0.0936 (8)	0.137 (9)
C(S25)	0.056 (3)	0.230 (1)	0.167 (1)	0.126 (9)
C(S26)	-0.026 (2)	0.160 (1)	0.196 (1)	0.14 (1)
C(S27)	0.163 (2)	0.295 (1)	0.079 (1)	0.16 (1)
H(1)	0.48210	0.32580	0.81650	0.050
H(2)	0.23120	0.19120	0.62080	0.050

1, with a stereoview in Fig. 2 illustrating the orientation of the cyclohexyl groups.

Related literature. The triphenylphosphine analogue of the title complex has been structurally characterized (Farrugia, Howard, Mitprachachon, Stone & Woodward, 1981). The ligand disposition is very similar, with a non-bonded Pt—Os distance of 3.530 (2) Å, and a butterfly angle of 88.4°.

Table 2. Selected bond lengths (Å) and bond angles (°), with *e.s.d.*'s in parentheses

Os(1)—Pt	3.506 (1)	Os(1)—Os(2)	2.923 (1)
Os(2)—Pt	2.853 (1)	Os(1)—Os(3)	3.023 (1)
Os(3)—Pt	2.732 (1)	Os(2)—Os(3)	2.760 (1)
Os(1)—P(1)	2.400 (2)	Pt—P(2)	2.344 (2)
Pt—C(1)	1.822 (8)	Os(1)—C(2)	1.917 (9)
Os(1)—C(3)	1.960 (8)	Os(1)—C(4)	1.886 (8)
Os(2)—C(5)	1.875 (8)	Os(2)—C(6)	1.916 (7)
Os(2)—C(7)	1.868 (8)	Os(3)—C(8)	1.889 (8)
Os(3)—C(9)	1.901 (7)	Os(3)—C(10)	1.889 (8)
Mean carbonyl C—O 1.14 (1)			
Os(3)—Pt—P(2)	171.2 (1)	H(2)—Pt—C(1)	175.3 (3)
H(1)—Os(1)—C(4)	177.8 (3)	C(3)—Os(1)—C(2)	178.0 (4)
Os(2)—Os(1)—P(1)	166.0 (1)	Os(3)—Os(2)—C(6)	161.2 (2)
H(2)—Os(2)—C(5)	168.0 (3)	Os(1)—Os(2)—C(7)	160.8 (3)
H(1)—Os(3)—C(10)	174.8 (3)	Pt—Os(3)—C(8)	161.6 (2)
Os(2)—Os(3)—C(9)	164.0 (3)	Os(2)—Os(1)—Os(3)	55.3 (1)
Os(1)—Os(2)—Os(3)	64.2 (1)	Os(2)—Os(3)—Os(1)	60.5 (1)
Os(1)—Os(2)—Pt	74.8 (1)	Os(3)—Os(2)—Pt	58.2 (1)
Os(1)—Os(3)—Pt	74.9 (1)	Os(2)—Os(3)—Pt	62.6 (1)
Os(2)—Pt—Os(3)	59.2 (1)	Pt—C(1)—O(1)	176.2 (7)
Os(1)—C(3)—O(3)	169.7 (7)		
Mean remaining Os—C—O 176.8 (7)			

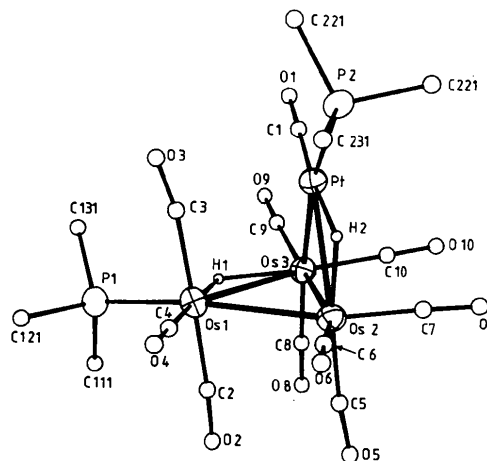


Fig. 1. Molecular structure and atomic labelling with cyclohexyl groups omitted for clarity.

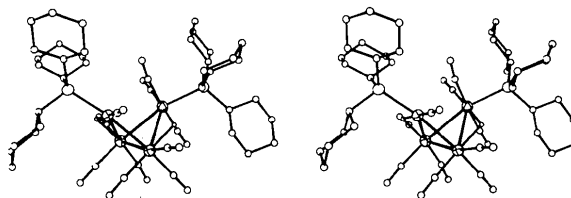


Fig. 2. Stereoview illustrating disposition of cyclohexyl groups.

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Structure of a Binuclear [(1*R*,2*R*)-*N,N'*-Disalicylidene-1,2-cyclohexanediamine]titanium(IV) Complex

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Abstract. Dichloro[7,7'-(cyclohexylenedinitrilo)di-*o*-cresolato]- μ -oxo-dititanium(IV)-acetone (1/2), [Ti₂(C₂₀H₂₀N₂O₂)₂Cl₂O].2C₃H₆O, $M_r = 939.6$, monoclinic, $C2$, $a = 22.189$ (6), $b = 9.616$ (2), $c = 23.452$ (6) Å, $\beta = 115.21$ (2)°, $V = 4527$ (2) Å³, $Z = 4$, $D_m(C_6H_6/CCl_4) = 1.44$ (3), $D_x = 1.38$ Mg m⁻³, Mo $K\alpha$, $\lambda = 0.71073$ Å, $\mu = 0.511$ mm⁻¹, $F(000) = 1963.7$, $T = 299$ (1) K, final $R = 0.045$ for 2421 observed unique reflections. This compound is a binuclear complex and has a pseudo center of symmetry at the bridging O(0) atom except for the (*R,R*)-1,2-cyclohexanediamine (*R,R*-chxn) moieties in the Schiff base. The Ti–O(0)–Ti angle is 168.3 (3)° and Ti–O(0) distances are 1.798 (6) and 1.814 (6) Å. The Ti atoms are coordinated octahedrally by two N and three O atoms and a Cl⁻ ion with O(0) and Cl⁻ *trans*.

Experimental. An orange crystal of dimensions 0.2 × 0.2 × 0.6 mm, crystallized from acetone and dichloromethane solution, sealed in a capillary tube together with mother liquor. Rigaku AFC-5 four-circle diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. Laue group $2/m$; cell parameters refined by least squares for 20 2θ values ($20 < 2\theta < 24^\circ$); intensity measurement performed to $2\theta = 45^\circ$ ($h - 23 \rightarrow 23$, $k - 10 \rightarrow 0$, $l 0 \rightarrow 25$), $\theta - 2\theta$ scan. Variation of five standard reflections, $0.99 \leq \sum(|F_o|/|F_o|_{\text{initial}})/5 \leq 1.01$. 3340 reflections measured, 2494 reflections

observed with $|F_o| > 3\sigma(|F_o|)$, 2421 unique ($R_{\text{int}} = 0.01$), absorption correction ($0.880 < A < 0.921$). The systematic absences, hkl with $h + k$ odd, show that the space group is $C2$, Cm or $C2/m$. The space group was assumed to be $C2$ because this compound has an optically active ligand and the crystal structure should not have mirror symmetry. Structure was solved by direct methods using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Absolute structure was assigned with reference to the known absolute configuration of *R,R*-chxn moieties. Two independent acetone molecules and all H atoms except for the acetone H atoms were revealed by difference synthesis. Non-H atoms were refined anisotropically and H atoms isotropically by block-diagonal least squares using *UNICSIII* computation program system (Sakurai & Kobayashi, 1979). $\sum w||F_o| - |F_c||^2$ minimized, $w^{-1} = \sigma^2(|F_o|) + (0.015|F_o|)^2$, final $R = 0.045$, $wR = 0.063$, $S = 2.66$ for 2421 unique reflections.* Reflection/parameter ratio 3.4, $\Delta/\sigma < 0.2$, $-0.25 \leq \Delta\rho \leq 0.83$ e Å⁻³. Complex neutral-atom scattering factors from *International Tables for X-ray Crystal-*

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and bond lengths and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44881 (37 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.