while in the the third the metal is in a general position. This seems to indicate that for these tetrakis complexes the metal's site symmetry is not the deciding factor in the choice of the coordination polyhedron.

Data collection and reduction were performed at The University of Jordan, and structure determination and refinement at Fordham University, Bronx, NY, to which we are grateful. We are also grateful to D. D. Cavalluzzo for her help in the final stages of refinement.

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Structure of 1,2,2,2,3,4,4,4-Octacarbonyl-1,2;3,4-di-μ-hydrido-1,3-bis(tricyclohexylphosphine)-*tetrahedro*-diplatinumdiosmium(4*Pt*–*Os*)(*Os*–*Os*)

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Abstract. $[Os_2Pt_2(\mu\text{-}H)_2(CO)_8\{P(C_6H_{11})_3\}_2]$, $M_r = 1557{\cdot}6$, monoclinic, $P2_1/a$, $a = 19{\cdot}713\,(2)$, $b = 10{\cdot}482\,(4)$, $c = 24{\cdot}146\,(6)$ Å, $\beta = 92{\cdot}25\,(1)°$, $V = 4985\,(2)$ Å^3, $Z = 4$, $D_x = 2{\cdot}08$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 108{\cdot}5$ cm^{-1}, $F(000) = 2936$, $T = 298$ K, $R = 0{\cdot}036$ for 4515 observed reflections. The dihedral butterfly angle of the metal framework is $84{\cdot}2\,(1)°$, and the Pt(1)···Pt(2) distance is $3{\cdot}230\,(1)$ Å, compared with corresponding magnitudes of $82{\cdot}9°$ and $3{\cdot}206\,(1)$ Å found in the related triphenylphosphine derivative.

Introduction. The dihedral 'butterfly' angle in several tetraruthenium clusters has been found to vary widely (Carty, MacLaughlin, Wagner & Taylor, 1982), and for $[AuFe_3(\mu_3\text{-}HC{=}NBu^t)(CO)_9(PPh_3)]$ this angle has values of $110{\cdot}9$ and $132{\cdot}1°$ for the two molecules within the same asymmetric unit (Bruce & Nicholson, 1983). Molecular-dynamical calculations on $[Fe_4(\mu_4\text{-}C)(CO)_{12}]^{2-}$ also suggest that the deformation of this angle is a soft mode (Bogdan, Horwitz & Shriver, 1986). The title butterfly cluster complex (1) has been examined structurally to compare parameters with those of the previously determined triphenylphosphine analogue (2) (Farrugia, Howard, Mitrprachachon, Stone & Woodward, 1981).

Experimental. Thin yellow plates from hexane solution: crystal dimensions $0{\cdot}30 \times 0{\cdot}29 \times 0{\cdot}016$ mm; systematic absences: $h = 2n + 1$ in $h0l$; $k = 2n + 1$ in $0k0$; 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, $11 < \theta < 13°$; intensities measured to $\theta = 25{\cdot}0°$ over range of hkl 0 to 23, 0 to 12, −28 to +28; $11,2,\bar{2}$, 402, $20\bar{2}$ measured every 2 h with nonlinear decay corrected by two linear sections, corresponding to 8% decay over 150 h data collection; 9573 reflections measured, 8757 independent data with 4515 having $I > 2{\cdot}0\sigma(I)$ considered observed and used in structure determination and refinement; R_{int} before absorption correction 0·20, after correction 0·097; corrected for Lorentz/polarization, absorption [Gaussian grid (Coppens, 1970) using 512 grid points; range of transmission factors on F^2, 0·061 to 0·829]; solved by direct methods (*MITHRIL*; Gilmore, 1984) and subsequent full-matrix least squares; isotropic thermal parameters for cyclohexyl-ring C atoms, anisotropic for remaining 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 ($U = 0{\cdot}05$ Å^2) thermal parameters; $w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o)]^{-1}$; max.

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(Δ/σ) 0·041, av. 0·003; $(\Delta\rho)_{max}$ +1·06, $(\Delta\rho)_{min}$ −1·63 e Å^{-3} in vicinity of heavy metal atoms; R = 0·036, wR = 0·047; $R(wR)$ = 0·120(0·053) for all data; S = 1·34; 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).

Discussion. 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. 1, with a stereoview of the unit-cell packing in Fig. 2. Complex (1) exhibits the

Table 1. *Final positional parameters (fractional coordinates) with e.s.d.'s in parentheses, and equivalent isotropic thermal parameters,* U_{eq}(Å^2)

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

	x	y	z	U_{eq}
Os(1)	0·80559 (3)	0·43141 (7)	0·30918 (3)	0·035
Os(2)	0·76679 (3)	0·28780 (7)	0·21679 (3)	0·040
Pt(1)	0·88610 (3)	0·43748 (7)	0·22059 (3)	0·034
Pt(2)	0·85744 (3)	0·18155 (7)	0·29039 (3)	0·035
P(1)	0·9435 (2)	0·4583 (4)	0·1390 (2)	0·035
P(2)	0·9244 (2)	0·1013 (4)	0·3637 (2)	0·034
O(1)	0·9697 (11)	0·6114 (22)	0·2901 (8)	0·145
O(2)	0·8601 (10)	−0·0428 (17)	0·2143 (8)	0·114
O(3)	0·6903 (7)	0·3642 (15)	0·3824 (7)	0·089
O(4)	0·8813 (7)	0·5743 (15)	0·4024 (7)	0·084
O(5)	0·7478 (7)	0·6725 (16)	0·2557 (7)	0·085
O(6)	0·6560 (8)	0·1683 (17)	0·2813 (7)	0·104
O(7)	0·6741 (8)	0·4893 (18)	0·1633 (8)	0·110
O(8)	0·7638 (10)	0·1061 (18)	0·1199 (7)	0·111
C(1)	0·9356 (11)	0·5390 (23)	0·2668 (8)	0·068
C(2)	0·8572 (11)	0·0471 (19)	0·2441 (8)	0·062
C(3)	0·7315 (10)	0·3874 (20)	0·3509 (6)	0·053
C(4)	0·8544 (9)	0·5195 (18)	0·3644 (8)	0·047
C(5)	0·7676 (9)	0·5795 (22)	0·2746 (8)	0·055
C(6)	0·6998 (10)	0·2110 (20)	0·2582 (7)	0·055
C(7)	0·7098 (10)	0·4092 (23)	0·1838 (8)	0·062
C(8)	0·7640 (10)	0·1761 (22)	0·1561 (10)	0·066
C(111)	1·0188 (7)	0·5614 (16)	0·1451 (7)	0·038 (4)
C(112)	1·0474 (9)	0·6240 (20)	0·0961 (8)	0·059 (5)
C(113)	1·1021 (9)	0·7212 (19)	0·1107 (8)	0·055 (5)
C(114)	1·1595 (9)	0·6587 (19)	0·1411 (8)	0·057 (5)
C(115)	1·1338 (9)	0·5974 (19)	0·1946 (8)	0·057 (5)
C(116)	1·0772 (8)	0·5005 (16)	0·1813 (7)	0·038 (4)
C(121)	0·8910 (8)	0·5361 (16)	0·0848 (7)	0·044 (4)
C(122)	0·8628 (9)	0·6624 (18)	0·1072 (8)	0·053 (5)
C(123)	0·8211 (10)	0·7299 (22)	0·0579 (9)	0·074 (6)
C(124)	0·7654 (10)	0·6473 (21)	0·0363 (9)	0·070 (6)
C(125)	0·7933 (10)	0·5225 (21)	0·0150 (9)	0·071 (6)
C(126)	0·8327 (9)	0·4527 (18)	0·0587 (8)	0·052 (5)
C(131)	0·9658 (8)	0·2995 (17)	0·1121 (7)	0·043 (4)
C(132)	0·9968 (8)	0·2115 (18)	0·1542 (8)	0·050 (5)
C(133)	1·0017 (9)	0·0738 (19)	0·1288 (8)	0·058 (5)
C(134)	1·0407 (11)	0·0752 (24)	0·0755 (10)	0·084 (7)
C(135)	1·0091 (10)	0·1679 (22)	0·0351 (9)	0·073 (6)
C(136)	1·0050 (9)	0·2998 (19)	0·0567 (8)	0·060 (5)
C(211)	0·8700 (8)	0·0214 (16)	0·4147 (7)	0·036 (4)
C(212)	0·8200 (9)	0·1135 (19)	0·4388 (8)	0·058 (5)
C(213)	0·7770 (11)	0·0446 (22)	0·4841 (9)	0·075 (6)
C(214)	0·7397 (11)	−0·0655 (24)	0·4549 (10)	0·083 (7)
C(215)	0·7884 (10)	−0·1607 (20)	0·4284 (9)	0·066 (6)
C(216)	0·8329 (10)	−0·0915 (20)	0·3870 (9)	0·065 (6)
C(221)	0·9850 (7)	−0·0270 (15)	0·3465 (7)	0·034 (4)
C(222)	1·0260 (9)	−0·0837 (19)	0·3936 (8)	0·053 (5)
C(223)	1·0649 (9)	−0·2035 (19)	0·3758 (8)	0·061 (5)
C(224)	1·1076 (9)	−0·1743 (20)	0·3277 (8)	0·062 (5)
C(225)	1·0685 (10)	−0·1116 (20)	0·2793 (9)	0·064 (6)
C(226)	1·0291 (9)	0·0068 (19)	0·2973 (8)	0·056 (5)
C(231)	0·9693 (7)	0·2298 (16)	0·4042 (7)	0·038 (4)
C(232)	1·0012 (9)	0·1945 (19)	0·4599 (8)	0·057 (5)
C(233)	1·0267 (9)	0·3155 (19)	0·4916 (8)	0·057 (5)
C(234)	1·0770 (11)	0·3833 (22)	0·4558 (10)	0·075 (6)
C(235)	1·0477 (9)	0·4168 (19)	0·3993 (8)	0·059 (5)
C(236)	1·0193 (8)	0·2996 (17)	0·3676 (7)	0·049 (5)
H(1)	0·84270	0·29360	0·34750	0·050
H(2)	0·83060	0·35250	0·17000	0·050

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

Os(1)–Os(2)	2·774 (1)	Os(1)–Pt(1)	2·714 (1)
Os(1)–Pt(2)	2·854 (1)	Os(2)–Pt(1)	2·826 (1)
Os(2)–Pt(2)	2·710 (1)	Pt(1)–P(1)	2·321 (5)
Pt(1)–C(1)	1·80 (3)	Pt(2)–P(2)	2·324 (5)
Pt(2)–C(2)	1·80 (2)	Os(1)–C(4)	1·86 (2)
Os(1)–C(3)	1·86 (2)	Os(1)–C(5)	1·90 (3)
Os(2)–C(6)	1·87 (2)	Os(2)–C(7)	1·86 (3)
Os(2)–C(8)	1·87 (3)	Pt(1)···Pt(2)	3·230 (1)
Pt(1)–Os(1)–Os(2)	62·0 (1)	Pt(2)–Os(1)–Os(2)	57·5 (1)
Pt(1)–Os(2)–Os(1)	58·0 (1)	Pt(2)–Os(2)–Os(1)	62·7 (1)
Os(1)–Pt(1)–Os(2)	60·1 (1)	Os(1)–Pt(2)–Os(2)	59·7 (1)
Pt(1)–Os(1)–Pt(2)	70·9 (1)	Pt(1)–Os(2)–Pt(2)	71·4 (1)
Os(1)–Pt(1)–P(1)	172·4 (2)	Os(2)–Pt(2)–P(2)	171·4 (2)
H(2)–Pt(1)–C(1)	172·8 (8)	H(1)–Pt(2)–C(2)	165·4 (7)
Pt(1)–Os(1)–C(3)	157·9 (6)	Pt(2)–Os(2)–C(7)	159·3 (7)
H(1)–Os(1)–C(5)	175·9 (7)	H(2)–Os(2)–C(6)	174·2 (6)
Os(2)–Os(1)–C(4)	164·8 (6)	Os(1)–Os(2)–C(8)	165·1 (6)
Pt(1)–C(1)–O(1)	171 (2)	Pt(2)–C(2)–O(2)	177 (2)
Os(1)–C(3)–O(3)	172 (2)	Os(2)–C(6)–O(6)	176 (2)
Os(1)–C(4)–O(4)	175 (2)	Os(2)–C(7)–O(7)	178 (2)
Os(1)–C(5)–O(5)	176 (2)	Os(2)–C(8)–O(8)	178 (2)

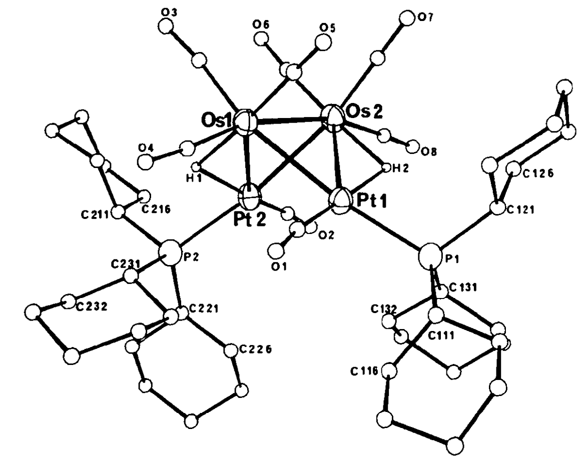

Fig. 1. Molecular structure of complex (1).

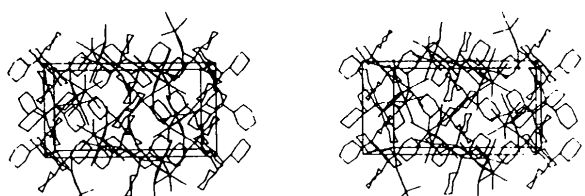

Fig. 2. Stereoview of the unit-cell packing of (1).

* 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 44746 (47 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

same idealized C_2 symmetry as the PPh_3 analogue (2) (Farrugia *et al.*, 1981). Apart from the hydride-bridged metal–metal vectors the Os atoms have approximate octahedral coordination, and the Pt atoms square-planar geometry (Table 2). Though the differences between the dihedral butterfly angle Pt(1)—Os(1)—Os(2)—Pt(2) = 84·2 (1)° and the non-bonding Pt(1)···Pt(2) separation = 3·230 (1) Å compared with corresponding parameters in (2) [82·9° and 3·206 (1) Å] are statistically significant, they are only marginal. The Os_2Pt_2 framework is evidently rather insensitive to the nature of the phosphine ligand. The slightly greater butterfly angle in (1) compared with (2) may be a result of steric repulsion between the larger cyclohexylphosphine ligands [Tolman cone angles 170 *vs* 145° for PCy_3 and PPh_3 respectively (Tolman, 1977)], or a consequence of the greater electron-releasing ability of PCy_3 [electronic parameters 2056·4 and 2068·9 cm^{-1} for PCy_3 *vs* PPh_3 (Tolman, 1977)]. Formal electron density at the cluster arising from ligand donation has been suggested as important in determining the butterfly angle in cluster complexes (Carty *et al.*, 1982).

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Structure of Dichlorodiquinolinecobalt(II): Isomorphism with the Analogous Co, Ni and Zn Halogenides

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Abstract. $[CoCl_2(C_9H_7N)_2]$, $M_r = 388·16$, triclinic, $P\bar{1}$, $a = 8·789$ (1), $b = 9·554$ (1), $c = 11·026$ (1) Å, $\alpha = 80·26$ (1), $\beta = 72·19$ (1), $\gamma = 71·83$ (1)°, $V = 835$ (1) Å^3, $Z = 2$, $F(000) = 394$, $D_m = 1·53$ (2) Mg m^{-3} (by flotation), $D_x = 1·544$ Mg m^{-3}, μ(Mo $K\alpha$) = 1·347 mm^{-1}, λ(Mo $K\alpha$) = 0·71069 Å, $T = 293$ (1) K. The structure was solved by Patterson and Fourier methods and refined to $R = 0·059$ for 4302 contributing reflections. The Co atom is tetrahedrally surrounded by two halogen and two N atoms [the mean Co—Cl and Co—N distances are 2·244 (4) and 2·05 (2) Å], the coordination tetrahedron being slightly distorted.

Introduction. The structure determination of the title compound was undertaken as part of studies of MQ_2X_2 complexes (M denotes a divalent metal of the transition series, Q = quinoline, X = halogen element Cl, Br, I or CNS group).

Experimental. Prepared by refluxing cobaltous chloride and quinoline in a 1:2 mole ratio (Brown, Nuttal & Sharp, 1964); dark blue prismatic crystals obtained from ethanol. Cell dimensions determined by least-squares refinement of θ angles of 60 strong reflections in the range $10 < \theta < 12°$, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, ω–2θ scan mode, 2θ scan width (0·7 + 0·3tanθ)°, aperture (2·4 + 0·9tanθ) mm, maximum scan time 40 s, $2\theta_{max}$ 60°, the hemisphere with index range $0 \le h \le 12$, $-13 \le k \le 13$ and $-15 \le l \le 15$ measured, crystal size 0·2 × 0·2 × 0·4 mm, three standard reflections (25$\bar{1}$, 440, 435) checked every 140 reflections measured; no significant decay; orientation

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