Table 2. *Bond distances* (Å) *and angles* (°) *for* $[Al(CH_3)]_2[C_{10}H_{20}N_4][Al(CH_3)_3]_2$

Al(1)—N(1)	1·948 (2)	Al(1)—N(2)	1·903 (2)
Al(1)—C(6)	1·929 (3)	Al(1)—N(1*a*)	1·947 (2)
Al(2)—N(2)	2·037 (2)	Al(2)—C(7)	1·990 (3)
Al(2)—C(8)	1·978 (3)	Al(2)—C(9)	1·978 (3)
N(1)—C(1)	1·505 (3)	N(1)—C(5*a*)	1·498 (2)
N(2)—C(3)	1·505 (3)	N(2)—C(4)	1·504 (3)
C(1)—C(2)	1·507 (3)	C(2)—C(3)	1·515 (3)
C(4)—C(5)	1·502 (3)		
N(1)—Al(1)—N(2)	102·3 (1)	N(1)—Al(1)—C(6)	117·0 (1)
N(2)—Al(1)—C(6)	124·1 (1)	N(1)—Al(1)—N(1*a*)	90·3 (1)
N(2)—Al(1)—N(1*a*)	92·8 (1)	C(6)—Al(1)—N(1*a*)	123·0 (1)
N(2)—Al(2)—C(7)	106·3 (1)	N(2)—Al(2)—C(8)	106·0 (1)
C(7)—Al(2)—C(8)	112·8 (1)	N(2)—Al(2)—C(9)	105·1 (1)
C(7)—Al(2)—C(9)	113·3 (1)	C(8)—Al(2)—C(9)	112·7 (1)
Al(1)—N(1)—C(1)	114·5 (1)	Al(1)—N(1)—Al(1*a*)	89·7 (1)
C(1)—N(1)—Al(1*a*)	124·5 (2)	Al(1)—N(1)—C(5*a*)	116·9 (1)
C(1)—N(1)—C(5*a*)	107·1 (1)	Al(1*a*)—N(1)—C(5*a*)	103·9 (1)
Al(1)—N(2)—Al(2)	111·8 (1)	Al(1)—N(2)—C(3)	113·3 (1)
Al(2)—N(2)—C(3)	106·9 (1)	Al(1)—N(2)—C(4)	105·8 (1)
Al(2)—N(2)—C(4)	109·5 (1)	C(3)—N(2)—C(4)	109·6 (2)
N(1)—C(1)—C(2)	114·8 (1)	C(1)—C(2)—C(3)	115·2 (2)
N(2)—C(3)—C(2)	115·5 (2)	N(2)—C(4)—C(5)	111·3 (2)
C(4)—C(5)—N(1*a*)	110·8 (1)		

Related literature. The triclinic form of the title compound represents the second polymorph of a product resulting from reaction of trimethylaluminium with [14]aneN_4. Reaction of one equivalent of [14]aneN_4 with four equivalents of trimethylaluminium, carried out at 393 K in toluene, gives monoclinic crystals of (1) (Robinson, Rae, Campana & Byram, 1987). In addition, an isomorphous gallium derivative (Lee, Pennington, Robinson & Rogers, 1990) and a mixed aluminium/gallium derivative, $[Me_3Ga]_2$[14]ane$N_4$$[MeAl]_2$ (Robinson, Pennington, Lee, Self & Hrncir, 1990),

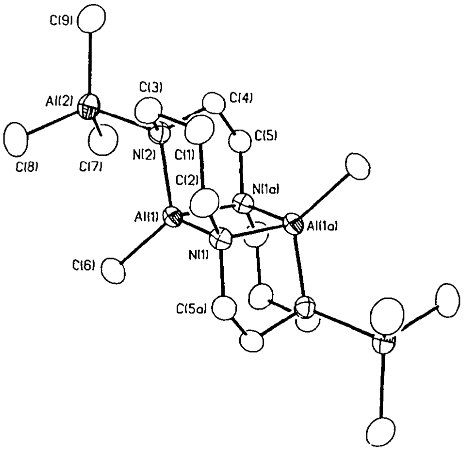

Fig. 1. Thermal ellipsoid plot (35% probability) of the molecule; H atoms are omitted.

which is isomorphous with the monoclinic form, have also been isolated.

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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-tricyclohexylphosphine-3-triisopropylphosphineplatinumtriosmium(2*Pt–Os*)(3*Os–Os*)

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Abstract. $[Os_3Pt(\mu\text{-}H)_2(CO)_{10}\{P(C_6H_{11})_3\}\{P(i\text{-}C_3H_7)_3\}]$, M_r = 1488·5, monoclinic, $P2_1/c$, a = 10·678 (2), b = 15·924 (4), c = 27·237 (5) Å, β = 100·92 (2)°, V = 4547 (2) Å^3, Z = 4, D_x = 2·17 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 115·85 cm^{-1}, F(000) = 2776, T = 298 K, R = 0·035 for 3227 unique observed reflections. The metal skeleton is of the butterfly form, with a non-bonding Pt···Os(3) distance of 3·837 (1) Å, and a butterfly angle, defined by the torsion angle Pt—Os(1)—Os(2)—Os(3), of 98·2 (1)°. The pseudo-octahedral ligand sphere on Os(3) is skewed out of the Os_3 plane by *ca* 11·5°.

Experimental. Pale yellow prisms from dichloromethane/hexane solution: crystal dimensions *ca* 0·4

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Table 1. *Fractional coordinates and isotropic thermal parameters* (Å^2) (*equivalent isotropic parameters* U_{eq} *for anisotropic atoms*) *with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
Pt	0·37918 (7)	0·20832 (4)	0·07005 (3)	0·037
Os(1)	0·36448 (7)	0·16819 (4)	0·16585 (3)	0·037
Os(2)	0·28384 (7)	0·32698 (4)	0·13393 (3)	0·040
Os(3)	0·54048 (7)	0·31906 (4)	0·19064 (3)	0·035
P(1)	0·3765 (4)	0·2376 (3)	−0·0145 (2)	0·039
P(2)	0·7546 (4)	0·2843 (3)	0·2271 (2)	0·043
O(1)	0·2766 (14)	0·1673 (9)	0·2646 (6)	0·087 (4)
O(2)	0·5020 (14)	0·0027 (9)	0·1850 (5)	0·081 (4)
O(3)	0·1176 (15)	0·0958 (9)	0·1094 (6)	0·096 (5)
O(4)	0·2938 (16)	0·5069 (11)	0·1004 (6)	0·110 (5)
O(5)	0·2034 (14)	0·3718 (9)	0·2299 (6)	0·092 (5)
O(6)	0·0142 (16)	0·2919 (9)	0·0833 (6)	0·095 (5)
O(7)	0·5396 (14)	0·5077 (9)	0·1932 (5)	0·087 (4)
O(8)	0·4638 (13)	0·3138 (7)	0·2939 (5)	0·070 (4)
O(9)	0·6278 (14)	0·3337 (8)	0·0902 (6)	0·078 (4)
O(10)	0·4769 (14)	0·0340 (9)	0·0692 (5)	0·086 (4)
C(1)	0·3122 (19)	0·1689 (11)	0·2271 (8)	0·064 (6)
C(2)	0·4468 (17)	0·0662 (11)	0·1774 (7)	0·053 (5)
C(3)	0·213 (2)	0·124 (1)	0·132 (1)	0·075 (6)
C(4)	0·288 (2)	0·437 (1)	0·114 (1)	0·075 (6)
C(5)	0·2366 (18)	0·3525 (11)	0·1932 (7)	0·054 (5)
C(6)	0·123 (2)	0·305 (1)	0·103 (1)	0·070 (6)
C(7)	0·5452 (17)	0·4336 (12)	0·1931 (7)	0·058 (5)
C(8)	0·4883 (18)	0·3169 (11)	0·2544 (7)	0·058 (5)
C(9)	0·5894 (18)	0·3262 (11)	0·1273 (7)	0·055 (5)
C(10)	0·4401 (17)	0·1008 (11)	0·0701 (7)	0·053 (5)
C(11)	0·7708 (17)	0·2286 (11)	0·2878 (7)	0·053 (5)
C(12)	0·710 (2)	0·141 (1)	0·280 (1)	0·095 (8)
C(13)	0·904 (2)	0·220 (1)	0·319 (1)	0·081 (7)
C(14)	0·8374 (18)	0·2302 (11)	0·1822 (7)	0·060 (5)
C(15)	0·975 (2)	0·199 (1)	0·206 (1)	0·095 (8)
C(16)	0·766 (2)	0·156 (1)	0·154 (1)	0·078 (7)
C(17)	0·8605 (19)	0·3766 (12)	0·2438 (8)	0·069 (6)
C(18)	0·874 (2)	0·430 (1)	0·197 (1)	0·085 (7)
C(19)	0·815 (2)	0·429 (1)	0·284 (1)	0·090 (7)
C(111)	0·2997 (14)	0·1497 (9)	−0·0537 (6)	0·034 (4)
C(112)	0·1881 (18)	0·1108 (11)	−0·0347 (7)	0·059 (5)
C(113)	0·1390 (18)	0·0331 (11)	−0·0638 (7)	0·063 (6)
C(114)	0·1031 (19)	0·0518 (12)	−0·1173 (7)	0·064 (6)
C(115)	0·2029 (19)	0·0901 (12)	−0·1382 (7)	0·067 (6)
C(116)	0·2550 (18)	0·1717 (11)	−0·1082 (7)	0·056 (5)
C(121)	0·2942 (17)	0·3349 (11)	−0·0412 (7)	0·053 (5)
C(122)	0·1602 (19)	0·3364 (12)	−0·0354 (7)	0·063 (6)
C(123)	0·085 (2)	0·416 (2)	−0·059 (1)	0·102 (8)
C(124)	0·155 (3)	0·492 (2)	−0·039 (1)	0·108 (9)
C(125)	0·287 (3)	0·493 (2)	−0·042 (1)	0·104 (8)
C(126)	0·3610 (18)	0·4135 (12)	−0·0187 (7)	0·064 (6)
C(131)	0·5384 (16)	0·2471 (10)	−0·0252 (7)	0·047 (5)
C(132)	0·625 (2)	0·175 (1)	−0·005 (1)	0·086 (7)
C(133)	0·767 (3)	0·191 (2)	−0·007 (1)	0·106 (8)
C(134)	0·780 (3)	0·208 (2)	−0·059 (1)	0·110 (9)
C(135)	0·699 (3)	0·283 (2)	−0·077 (1)	0·100 (8)
C(136)	0·557 (2)	0·265 (1)	−0·075 (1)	0·099 (8)
H(1)	0·34160	0·32050	0·07390	0·050
H(2)	0·53200	0·20380	0·18380	0·050

× 0·2 × 0·1 mm; systematic absences: $k = 2n + 1$ in $0k0$; $l = 2n + 1$ in $h0l$; Enraf–Nonius CAD-4F diffractometer; graphite monochromator; $\theta/2\theta$ scan mode; cell parameters refined by least-squares methods from setting angles of 21 independent reflections with $11 < \theta < 13°$; intensities measured to $\theta = 20·0°$ over range hkl 0 to 10, −15 to 15, −26 to 26; $\bar{6}26$ and $\bar{3}, \bar{3}, \overline{11}$ measured every 2 h with a 15% decay over 139 h data collection; 9086 data measured, 4240 independent data with 3227 having $I > 3·0\sigma(I)$ considered observed and used in structure determination and refinement; R_{int} 0·193 before and 0·043 after absorption correction; corrected for Lorentz–polarization and absorption (*DIFABS*; Walker & Stuart, 1983), max., min. values of applied absorption correction 1·46, 0·80; solved by direct methods (*MITHRIL*; Gilmore, 1984) and subsequent full-matrix least squares; anisotropic thermal parameters for Os, Pt and P atoms, isotropic thermal parameters for the O and C atoms; H atoms included at calculated positions [C—H = 1·0 Å: Os—H, Pt—H *ca* 1·85 Å (*HYDEX*; Orpen, 1980)]; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o)]^{-1}$; max. Δ/σ 0·017, av. 0·001; $\Delta\rho_{\text{max}}$ + 0·85, $\Delta\rho_{\text{min}}$ −0·83 e Å^{-3}; final $R = 0·035$, $wR = 0·043$, using 243 parameters; $S = 2·62$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71–151); calculations carried out on a MicroVAX 3600 computer 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 atomic labelling scheme and molecular structure are shown in Fig. 1.

Related literature. The gross structural features of the title complex are very similar to those of the previously reported bis(triphenylphosphine) (Farrugia, Howard, Mitrprachachon, Stone & Woodward, 1981), and bis(tricyclohexylphosphine) derivatives (Farrugia, 1988). The major difference lies in the

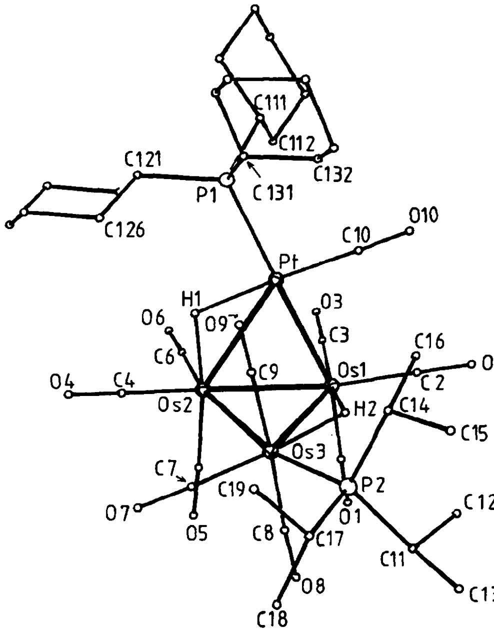

Fig. 1. Molecular structure and atomic labelling scheme for the title compound. Anisotropic atoms are shown at the 10% probability level, isotropic atoms as spheres of arbitrary radius.

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* Lists of structure factors, anisotropic thermal parameters, calculated hydrogen positional parameters, and a complete listing of bond lengths and angles and torsion angles, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53837 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

Os(1)—Pt	2·720 (1)	Os(1)—Os(2)	2·758 (1)
Os(2)—Pt	2·882 (1)	Os(1)—Os(3)	3·046 (1)
Os(3)—Pt	3·837 (1)	Os(2)—Os(3)	2·882 (1)
Pt—P(1)	2·344 (5)	Os(3)—Pt(2)	2·379 (5)
Pt—C(10)	1·83 (2)	Os(1)—C(1)	1·86 (3)
Os(1)—C(2)	1·85 (2)	Os(1)—C(3)	1·85 (3)
Os(2)—C(4)	1·83 (3)	Os(2)—C(5)	1·83 (2)
Os(2)—C(6)	1·80 (3)	Os(3)—C(7)	1·83 (2)
Os(3)—C(8)	1·92 (2)	Os(3)—C(9)	1·90 (2)

Mean carbonyl C—O 1·18 (3)

Os(1)—Pt—P(1)	175·5 (2)	Os(1)—Pt—C(10)	82·2 (6)
H(1)—Pt—P(1)	84·2 (2)	H(2)—Os(1)—C(3)	164·1 (7)
Pt—Os(1)—C(1)	160·5 (6)	Os(2)—Os(1)—C(2)	166·1 (6)
Os(1)—Os(2)—C(4)	160·6 (7)	H(1)—Os(2)—C(5)	169·9 (6)
Os(3)—Os(2)—C(6)	165·9 (7)	P(2)—Os(3)—Os(2)	167·1 (2)
H(2)—Os(3)—C(7)	176·4 (6)	C(8)—Os(3)—C(9)	177·4 (8)

wider butterfly angle for the title complex, *cf.* 98·2 (1) *versus* 88·4 ($bis\text{-}PPh_3$) and 87·5° ($bis\text{-}PCy_3$). This is also seen in an increased non-bonding Os—Pt wing-tip–wing-tip distance, *cf.* Os(3)—Pt = 3·837 (1) *versus* 3·506 (1) ($bis\text{-}PPh_3$) and 3·530 (1) Å ($bis\text{-}PCy_3$). This is a reflection of the flexibility of the Os_3Pt butterfly metal skeleton. In addition the ligand sphere around Os(3) is twisted by *ca* 11·5° out of the Os_3 plane, as defined by the angles between the Os_3 plane and the mean plane Os(3), P(2), C(7), O(7), H(2). In the bis(tricyclohexylphosphine) derivative this twist is similar (11·8°), but it is much smaller in the bis(triphenylphosphine) derivative (0·4°).

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Structure of 1,4,7-Tris[(2*S*)-2-hydroxypropyl]-1,4,7-triazacyclononanenickel(II) Dibromide Monohydrate

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Abstract. $[Ni(C_{15}H_{33}N_3O_3)]Br_2.H_2O$, $M_r = 539{\cdot}96$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}7046\ (9)$, $b = 13{\cdot}742\ (3)$, $c = 16{\cdot}285\ (1)$ Å, $V = 2171{\cdot}8\ (5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}65$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 45{\cdot}65$ cm^{-1}, $F(000) = 1104$, $T = 298$ K, $R = 0{\cdot}040$ for 2823 unique observed reflections. The Ni atom is coordinated octahedrally to the *fac*-N_3O_3 donor set of the macrocyclic ligand, and a molecule of water is hydrogen bonded to one of the ligand hydroxyl groups. The twist angle φ, away from idealized octahedral coordination, is 19·0° between the N_3 donor set and the O_3 set.

Experimental. The title complex was synthesized as previously described (Robb, 1987), and recrystallized from water. A pale-purple prism *ca* 0·8 × 0·6 × 0·4 mm was mounted on a glass fibre; systematic absences: $h = 2n + 1$ in $h00$, $k = 2n + 1$ in $0k0$; $l = 2n + 1$ in $00l$; Enraf–Nonius CAD-4F diffractometer; graphite monochromator; $\theta/2\theta$ scan mode; cell parameters refined by least-squares methods from setting angles of 25 independent reflections with $11 < \theta < 13^\circ$; intensities measured to $\theta = 25{\cdot}0^\circ$ in two octants, hkl and $\overline{hkl}$ with $|h|$ 0–11, $|k|$ 0–16, $|l|$ 0–19; $2\bar{9}2$ and $\bar{1}\bar{7}1$ measured every 2 h with no significant decay noted over 69 h data collection; 4403 measured data yielded 3811 independent data, containing 1632 Friedel pairs; of these 2823 having $I > 3{\cdot}0\sigma(I)$ were considered observed and used in structure determination and refinement; R_{int} before absorption correction 0·020, after correction 0·019; corrected for Lorentz–polarization and absorption (*DIFABS*; Walker & Stuart, 1983), with max., min. applied absorption corrections of 1·10, 0·87; solved by direct methods (*MITHRIL*; Gilmore, 1984) and subsequent full-matrix least squares; anisotropic thermal parameters for all non-H atoms; C—H H atoms included at calculated positions (C—H = 1·0 Å), O—H H-atom positions obtained from difference synthesis, fixed contributions to structure factors for all H atoms, with $U = 0{\cdot}05$ Å^2; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o)]^{-1}$; max. $\Delta/\sigma =$

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