H atoms were refined isotropically in observed positions. After anisotropic refinement of the non-H atoms, one of the CF_3 groups (C5) showed evidence of disorder, manifested in larger fluorine displacement parameters and significant residual electron density between the fluorine atomic positions. A disorder model including 28 additional parameters lowered wR_2(all data) from 22.16 to 11.20%. Several different disorder models were tried, including combinations of restrictions on C—F bond distances and restraints on anisotropic displacement parameters. None of the models tried were completely satisfactory in that a wide range of C—F bond lengths were observed for the disordered atoms. In the final model, restraints on displacement parameters were not included since these tended to raise the *R* factors without influencing the observed CF_3 group geometry. Restraints were applied, however, to C—F bond distances on the C5 atom using a C—F bond distance of 1.34 Å, with a standard deviation of 0.01. The final C—F distances for C1 ranged from 1.309 (4) to 1.343 (5) Å, with a mean of 1.32 [2] Å; on the C5 atom, the range was 1.271 (9) to 1.403 (8) Å, with a mean C—F distance of 1.32 [6] Å. The angles around C1 ranged from 104.9 (3) to 114.7 (3)°, with a mean of 109 [3]°, while the angles around C5 ranged from 91.3 (12) to 123.3 (11)°, with a mean of 109 [10]°.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXTL-Plus*.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: BS1010). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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μ-Hydrido-μ-dicyclohexylphosphido-bis[dicyclohexylphosphine(tricarbonyl)rhenium]

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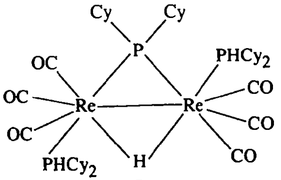
Abstract

The title compound, hexacarbonyl-1κ^3C,2κ^3C-μ-dicyclohexylphosphido-1:2κ^2P-bis(dicyclohexylphosphine)-1κP,2κP-μ-hydrido-dirhenium(*Re*—*Re*), $[Re_2H(C_{12}H_{22}P)(C_{12}H_{23}P)_2(CO)_6]$, has an Re—Re bond length of 3.2185 (8) Å which is symmetrically bridged by a H and a P atom. Two terminal $PH(C_6H_{11})_2$ ligands adopt *cis* positions with respect to the phosphido bridge and have a mutually *trans* configuration.

Comment

The study of phosphido-bridged homo- and heteronuclear transition metal cluster compounds is of great interest due to their metal–metal bond stabilities under various reaction conditions (Haupt, Balsaa & Flörke, 1988). The title compound, $[Re_2(\mu\text{-}H)(\mu\text{-}PCy_2)(CO)_6(PHCy_2)_2]$, (I) (Cy is cyclohexyl), is a new dinuclear example which was obtained from the reaction of $[Re_2(\mu\text{-}H)(\mu\text{-}PCy_2)(CO)_6(NCCH_3)_2]$ with $HPCy_2$ in $CHCl_3$ solution at 343 K.



(I)

The Re atom in (I) is surrounded by six non-metal ligands forming a distorted-octahedral coordination geometry (Fig. 1). These ligands are three carbonyl groups in *trans* positions with respect to the bridging μ-H and μ-P atoms, and a $PHCy_2$ ligand attached in an axial position. The second half of the molecule is symmetry related by a crystallographic twofold axis which runs through both bridging ligands. The two coordination octahedra around the metal atoms thus share one common edge along this axis.

The Re—Re bond length of 3.2185 (8) Å fulfills the requirement of 18 valence electrons for each metal atom.

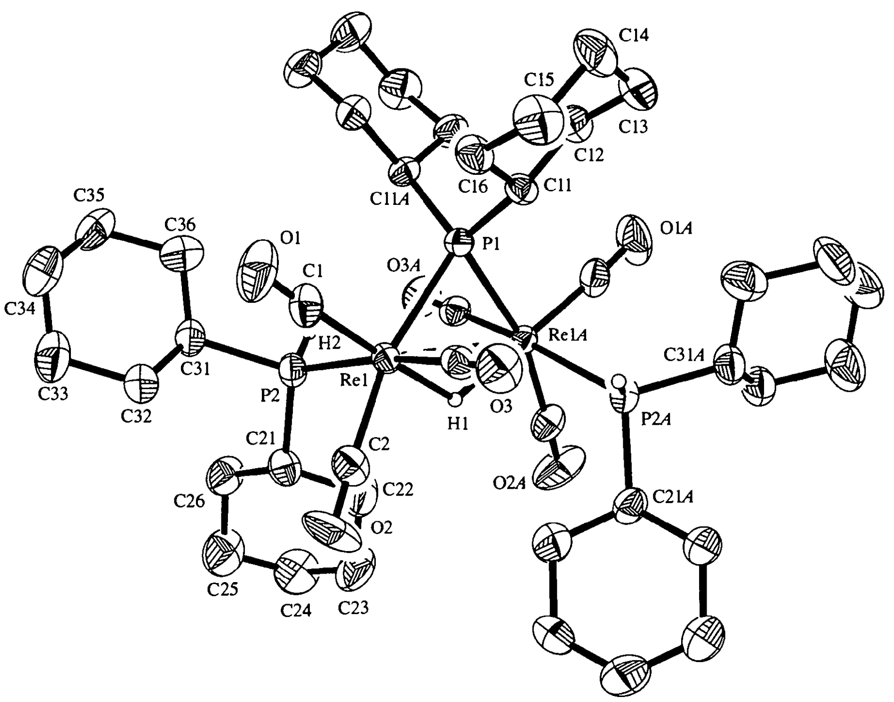


Fig. 1. The molecular structure of (I) with the cyclohexyl H atoms omitted. Displacement ellipsoids are plotted at the 50% probability level.

It is elongated by about 0.077 Å compared with that of the unsubstituted carbonyl complex [$Re_2(\mu\text{-}H)(\mu\text{-}PCy_2)(CO)_8$], (II) (Flörke & Haupt, 1992), and is 0.025 Å longer than the Re—Re bond in [$Re_2(\mu\text{-}H)(\mu\text{-}PPh_2)(CO)_6(PPh_3)_2$], (III) (Ph is phenyl), hitherto the longest bonding Re—Re distance reported for such dinuclear carbonyl compounds (Haupt, Balsaa & Flörke, 1987). Imposed by the crystallographic symmetry, the Re—μ-P bond lengths are equal with values of 2.467 (2) Å and the enclosed Re—P—Re angle is 81.45 (7)°. The μ-H position was determined from a difference Fourier map and refined; the Re—H bond lengths and Re—H—Re angle are 1.84 (4) Å and 122 (5)°, respectively. The terminal $PHCy_2$ ligands, with an Re—P bond length of 2.494 (2) Å, have a mutually *trans* configuration and are in *cis* positions with respect to the phosphido bridge.

Only three other related disubstituted carbonyl cluster compounds have been characterized so far by X-ray structure determination. The same *cis* arrangement of the non-carbonyl ligands as for (I) is found in [$Mn_2(\mu\text{-}H)(\mu\text{-}PPh_2)(CO)_6({}^tBuNC)_2$] (Iggo, Mays, Raithby & Hendrick, 1983), whereas an arrangement with the phosphine ligands positioned *trans* with respect to the phosphido bridge is realised with both the already mentioned compound (III) and [$Mn_2(\mu\text{-}H)(\mu\text{-}PCy_2)(CO)_6(PMe_3)_2$] (Arif, Jones & Schwab, 1986). Both compounds (I) and (III), with their different *cis* and *trans* configurations, reveal strong intramolecular steric interactions due to the bulky phosphine ligands. This can be seen from a comparison of the torsion angles. When viewed down the Re—Re bond in (I), the axial CO and $PHCy_2$ ligands are in approximately eclipsed positions, with a torsion angle of 21.0 (2)°. For the equatorial CO groups, the average value is 16.2 (5)°. Similar conditions hold for compound (III) with a torsion angle of 12.6° (average) for the axial CO groups and of 16.0 and 25.3° for the equatorial CO and PPh_3 ligands, respectively. The related unsubstituted compounds (II) and [$Re_2(\mu\text{-}H)(\mu\text{-}PPh_2)(CO)_8$] (Flörke & Haupt, 1994) show substantially smaller torsion angles, ranging from 2.3 to 6.2° (average values). Monosubstituted Mn or Re carbonyl cluster compounds [$M_2(\mu\text{-}H)(\mu\text{-}PR_2)(CO)_7(PL_3)$], where L_3 is HCy_2 or Ph_3, also exhibit these terminal phosphine ligands in *trans* positions with respect to the Re—μ-P bond (Arif, Jones & Schwab, 1986; Haupt, Balsaa, Flörke, 1988).

Experimental

Crystals of (I) were obtained from the reaction of [$Re_2(\mu\text{-}H)(\mu\text{-}PCy_2)(CO)_6(NCCH_3)_2$] with $HPCy_2$ in $CHCl_3$ solution at 343 K. The product was recrystallized from a $CHCl_3$–pentane solution.

Crystal data

[$Re_2H(C_{12}H_{22}P)(C_{12}H_{23}P)_2(CO)_6$]
M_r = 1135.3
Monoclinic
$C2/c$

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 7–15°

$a = 17.491$ (4) Å
$b = 14.236$ (3) Å
$c = 18.656$ (4) Å
$\beta = 100.24$ (2)°
$V = 4571.4$ (16) Å^3
$Z = 4$
$D_x = 1.650$ Mg m^{-3}
D_m not measured

$\mu = 5.438$ mm^{-1}
$T = 293$ (2) K
Prism
0.21 × 0.16 × 0.15 mm
Colourless

Data collection

Siemens *R*3*m* diffractometer
ω–2θ scans
Absorption correction:
ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.389$, $T_{\max} = 0.442$
5451 measured reflections
5290 independent reflections
3502 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.0352$
$\theta_{\max} = 27.56°$
$h = -22 \rightarrow 22$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 24$
3 standard reflections every 400 reflections intensity decay: 4%

Refinement

Refinement on F^2
$R(F) = 0.0424$
$wR(F^2) = 0.0635$
$S = 0.944$
5290 reflections
245 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0192P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.630$ e Å^{-3}
$\Delta\rho_{\min} = -0.709$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.00004 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Re1	0.040342 (13)	0.28762 (2)	0.178908 (12)	0.02716 (8)
P1	0	0.1563 (2)	1/4	0.0259 (5)
P2	−0.08685 (9)	0.32913 (12)	0.10207 (8)	0.0330 (4)
C1	0.0737 (3)	0.2156 (6)	0.1031 (3)	0.043 (2)
O1	0.0950 (3)	0.1709 (4)	0.0592 (3)	0.071 (2)
C2	0.0771 (3)	0.4044 (5)	0.1452 (3)	0.039 (2)
O2	0.1051 (3)	0.4719 (4)	0.1269 (3)	0.072 (2)
C3	0.1442 (3)	0.2704 (4)	0.2328 (3)	0.0327 (14)
O3	0.2086 (2)	0.2634 (3)	0.2595 (2)	0.0526 (13)
C11	0.0822 (3)	0.0808 (4)	0.2944 (3)	0.0291 (13)
C12	0.0591 (3)	0.0061 (4)	0.3465 (3)	0.041 (2)
C13	0.1322 (4)	−0.0412 (5)	0.3895 (4)	0.050 (2)
C14	0.1814 (4)	−0.0840 (5)	0.3399 (4)	0.059 (2)
C15	0.2029 (4)	−0.0116 (5)	0.2873 (4)	0.055 (2)
C16	0.1306 (3)	0.0350 (4)	0.2437 (3)	0.040 (2)
C21	−0.1087 (3)	0.4576 (4)	0.0971 (3)	0.038 (2)
C22	−0.1134 (5)	0.4962 (5)	0.1715 (4)	0.067 (2)
C23	−0.1315 (5)	0.6021 (5)	0.1703 (4)	0.073 (2)
C24	−0.2026 (4)	0.6267 (6)	0.1162 (4)	0.067 (2)
C25	−0.1949 (4)	0.5923 (5)	0.0421 (4)	0.070 (2)
C26	−0.1800 (4)	0.4862 (5)	0.0427 (4)	0.052 (2)
C31	−0.1080 (4)	0.2897 (5)	0.0063 (3)	0.0427 (15)
C32	−0.0513 (4)	0.3315 (5)	−0.0391 (3)	0.049 (2)
C33	−0.0767 (5)	0.3016 (6)	−0.1192 (4)	0.075 (3)
C34	−0.0820 (6)	0.1989 (6)	−0.1275 (4)	0.088 (3)
C35	−0.1390 (5)	0.1585 (6)	−0.0825 (4)	0.073 (2)
C36	−0.1164 (5)	0.1861 (5)	−0.0021 (4)	0.063 (2)

Table 2. *Selected geometric parameters (Å, °)*

Re1—P1	2.467 (2)	Re1—Re1$^{\rm i}$	3.2185 (8)
Re1—P2	2.494 (2)	Re1—H1	1.84 (4)
P1—Re1—P2	100.22 (4)	C11$^{\rm i}$—P1—C11	109.6 (4)
P1—Re1—Re1$^{\rm i}$	49.28 (3)	Re1$^{\rm i}$—P1—Re1	81.45 (7)
P2—Re1—Re1$^{\rm i}$	89.81 (4)	Re1$^{\rm i}$—H1—Re1	122 (5)

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

H atoms were refined riding on their carrier atoms, except for H1 and H2, which were located from the difference map and their positions refined.

Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1995). Program(s) used to refine structure: *SHELXTL*.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: SK1060). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dichlorodimethylbis(3-methyladenine-N^7)-tin(IV)

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Abstract

The title compound, [$SnCl_2(CH_3)_2(C_6H_7N_5)_2$], has been prepared from dimethyltin dichloride and 3-methyladenine in methanol. The coordination of the Sn atom