C10	0.1337 (4)	1.1185 (6)	1.0201 (12)	0.029 (3)
C11	0.1102 (4)	1.1046 (6)	0.8895 (11)	0.029 (2)
C12	0.0830 (4)	1.0317 (6)	0.8594 (11)	0.031 (3)
C13	0.0611 (4)	0.8551 (5)	0.7448 (12)	0.025 (2)
C14	0.0140 (4)	0.8375 (5)	0.6520 (11)	0.025 (2)
C15	0.0309 (4)	0.8116 (5)	0.5183 (11)	0.027 (3)
C16	0.0914 (5)	0.8060 (6)	0.4809 (12)	0.036 (3)
C17	0.1385 (4)	0.8239 (6)	0.5752 (11)	0.030 (3)
C18	0.1234 (4)	0.8497 (6)	0.7080 (10)	0.025 (2)
C19	0.0492 (4)	0.6651 (5)	1.1388 (11)	0.021 (2)
C20	0.0005 (4)	0.6971 (6)	1.2127 (10)	0.027 (2)
C21	−0.0386 (4)	0.6497 (6)	1.2901 (12)	0.035 (3)
C22	−0.0276 (5)	0.5687 (7)	1.2997 (12)	0.042 (3)
C23	0.0213 (4)	0.5357 (5)	1.2269 (13)	0.039 (3)
C24	0.0595 (4)	0.5821 (5)	1.1448 (12)	0.028 (2)
C25	0.1192 (4)	0.6778 (5)	0.8821 (11)	0.021 (2)
C26	0.0708 (4)	0.6572 (5)	0.7913 (11)	0.027 (3)
C27	0.0841 (5)	0.6198 (5)	0.6681 (12)	0.033 (3)
C28	0.1443 (5)	0.6012 (6)	0.6354 (13)	0.040 (3)
C29	0.1921 (4)	0.6192 (6)	0.7249 (12)	0.040 (3)
C30	0.1798 (5)	0.6592 (6)	0.8485 (12)	0.032 (3)
C31	0.1706 (4)	0.7421 (5)	1.1409 (11)	0.026 (2)
C32	0.1904 (4)	0.6840 (6)	1.2373 (12)	0.032 (3)
C33	0.2422 (4)	0.7015 (7)	1.3168 (11)	0.037 (3)
C34	0.2749 (4)	0.7719 (6)	1.3040 (12)	0.037 (3)
C35	0.2560 (4)	0.8259 (6)	1.2077 (12)	0.032 (3)
C36	0.2041 (4)	0.8124 (6)	1.1254 (11)	0.029 (2)

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

Mo—O	1.646 (6)	Mo—Cl3	2.324 (3)
Mo—Cl1	2.340 (3)	Mo—Cl4	2.353 (3)
Mo—Cl2	2.351 (3)		
O—Mo—Cl1	108.4 (3)	Cl1—Mo—Cl3	141.94 (10)
O—Mo—Cl2	101.8 (3)	Cl1—Mo—Cl4	86.08 (11)
O—Mo—Cl3	109.6 (3)	Cl2—Mo—Cl3	86.45 (11)
O—Mo—Cl4	101.2 (3)	Cl2—Mo—Cl4	157.06 (10)
Cl1—Mo—Cl2	86.67 (12)	Cl3—Mo—Cl4	85.93 (11)

H atoms were placed in calculated positions (C—H = 0.95 Å) and allowed to ride on the C atoms to which they are attached with $U_{iso}(H) = 1.2U_{eq}(C)$ in subsequent cycles of refinement.

Data collection: *DIF*4 (Stoe & Cie, 1990*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1990*b*). Program(s) used to solve structure: *SIR*92 (Altomare, Cascarano, Giacovazzo & Guagliardi, 1993). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *CAMERON* (Pearce & Watkin, 1993).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: MU1151). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Octacarbonyl-1κ^4C,2κ^4C-μ_3-[cyclohexylphosphanido(2–)]-μ-hydrido-1:2κ^2H-tricyclohexylphosphine-3κP-golddimanganese(*Mn—Mn*)

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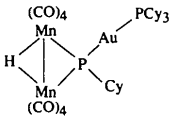
Abstract

The title compound, $[AuMn_2H(C_6H_{11}P)(C_{18}H_{33}P)(CO)_8]$, has an Mn—Mn bond length of 2.933 (1) Å which is symmetrically bridged by an H and a P atom. The 'coordination' of the P atom is completed by one cyclohexyl and one (tricyclohexylphosphine)gold 'ligand'.

Comment

$[Mn_2(\mu\text{-H})\{\mu_3\text{-PCy}(AuPCy_3)\}(CO)_8]$, (I), where Cy is cyclohexyl, constitutes the first example of a μ-H and μ_3-P bridged transition metal–gold complex. It may be derived formally from the family of $[M_2(\mu\text{-H})(\mu\text{-PR}_2)(CO)_8]$ compounds (Haupt, Heinekamp, Flörke & Jüptner, 1992) by substitution of one *R* ligand by an $AuPR_3$ group.



(I)

Each Mn atom of the title compound attains slightly distorted octahedral coordination *via* the bridging H and P atoms and four carbonyl ligands. The CO groups display an almost eclipsed arrangement at both Mn centres. The position of the μ-H atom, which was confirmed from ^{1}H NMR measurements, could be located from ΔF maps and refined. It lies in the plane of the central Mn_2P ring with an average Mn—H distance of 1.78 (10) Å. The Mn—Mn single bond meets the requirement of 18 valence electrons for each metal and has a length of 2.933 (1) Å. This value is close to similar bond lengths found for the related μ-H and μ-P bridged compounds [$Mn_2(\mu$-H)(μ-PPh$_2$)(CO)$_8$] [2.937 (5) Å (Doedens, Robinson & Ibers, 1967)] and [$Mn_2(\mu$-H)-(μ-PHCy)(CO)$_8$] [2.940 (1) Å (Flörke & Haupt, 1994)]. The Mn_2P ring, with two equal Mn—P bond lengths of 2.334 (2) Å, shows a typical acute Mn—P—Mn angle [77.86 (5)°]. The Au—P bond lengths are very similar [Au—P1 2.312 (1) and Au—P2 2.320 (1) Å], whereas the enclosed angle at the Au atom of 171.12 (5)° deviates slightly from that of a linear P—Au—P group. This may well be due to packing distortions that arise from the bulky cyclohexyl ligands. One cyclohexyl group (C31–C36) is disordered over two sites. This disorder was successfully treated with a split model for atoms C31, C33, C34 and C36, with half occupation for each. Both orientations show the usual chair conformation and have common positions for C32 and C35.

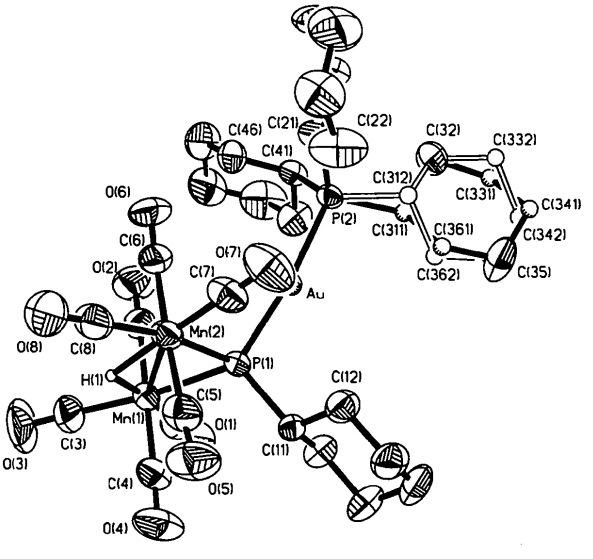


Fig. 1. Molecular structure of the title complex with displacement ellipsoids plotted at the 50% probability level.

Experimental

The title compound was produced from the reaction of [$Mn_2(\mu$-H)(μ-PCyH)(CO)$_8$] with 1,8-diazabicyclo[5.4.0]-undec-7-ene in tetrahydrofuran solution at 298 K for 30 min, followed by addition of [AuCl(PCy$_3$)]. From this solution, the yellow title compound and its orange isomer [Mn_2-(μ-AuPCy$_3$)(μ-PCyH)(CO)$_8$] were isolated. After recrystallization from CH_2Cl_2 solution the two isomers were separated by sorting the crystals by hand.

Crystal data

[AuMn$_2$H(C$_6$H$_{11}$P)-(C$_{18}$H$_{33}$P)(CO)$_8$]
M_r = 926.47
Triclinic
$P\bar{1}$
a = 9.684 (2) Å
b = 12.764 (2) Å
c = 16.773 (3) Å
α = 76.840 (10)°
β = 75.110 (10)°
γ = 71.030 (10)°
V = 1871.1 (6) Å^3
Z = 2
D_x = 1.644 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 30 reflections
θ = 8–15°
μ = 4.705 mm^{-1}
T = 293 (2) K
Plate
0.45 × 0.26 × 0.08 mm
Yellow

Data collection

Siemens *R3m/V* diffractometer
ω–2θ scans
Absorption correction: empirical *via* ψ scans
T_{min} = 0.417, T_{max} = 0.796
8325 measured reflections
8048 independent reflections
6528 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0261
θ_{max} = 27.56°
h = −12 → 12
k = −16 → 16
l = 0 → 21
3 standard reflections monitored every 400 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F)$ = 0.0397
$wR(F^2)$ = 0.0935
S = 1.022
8048 reflections
405 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.5442P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.764 e Å^{-3}
$\Delta\rho_{min}$ = −1.210 e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	U_{eq}
Au	0.04946 (2)	0.45143 (2)	0.267418 (13)	0.03656 (7)
Mn1	−0.19199 (9)	0.78257 (7)	0.24680 (6)	0.0441 (2)
Mn2	0.08946 (10)	0.73029 (7)	0.12541 (5)	0.0469 (2)
P1	0.02737 (14)	0.63959 (11)	0.26138 (8)	0.0354 (3)
P2	0.08104 (15)	0.26892 (11)	0.25104 (9)	0.0375 (3)
C1	−0.2966 (6)	0.7480 (6)	0.3491 (4)	0.054 (2)

O1	−0.3633 (6)	0.7271 (5)	0.4145 (3)	0.083 (2)
C2	−0.2636 (7)	0.6859 (6)	0.2110 (4)	0.058 (2)
O2	−0.3155 (6)	0.6300 (5)	0.1923 (4)	0.089 (2)
C3	−0.3358 (8)	0.9024 (7)	0.2093 (5)	0.071 (2)
O3	−0.4252 (7)	0.9783 (6)	0.1871 (5)	0.123 (3)
C4	−0.1321 (7)	0.8777 (5)	0.2885 (4)	0.057 (2)
O4	−0.1015 (6)	0.9380 (5)	0.3167 (4)	0.095 (2)
C5	0.1706 (8)	0.8153 (6)	0.1648 (4)	0.060 (2)
O5	0.2283 (7)	0.8654 (5)	0.1866 (4)	0.093 (2)
C6	0.0181 (8)	0.6383 (6)	0.0845 (4)	0.058 (2)
O6	−0.0160 (7)	0.5803 (5)	0.0567 (3)	0.086 (2)
C7	0.2695 (8)	0.6299 (6)	0.1062 (4)	0.061 (2)
O7	0.3821 (6)	0.5626 (5)	0.0958 (4)	0.093 (2)
C8	0.0954 (9)	0.8259 (6)	0.0254 (4)	0.067 (2)
O8	0.1018 (8)	0.8853 (5)	−0.0368 (3)	0.099 (2)
C11	0.1293 (5)	0.6532 (5)	0.3374 (3)	0.037 (1)
C12	0.2937 (6)	0.5858 (6)	0.3202 (4)	0.056 (2)
C13	0.3755 (7)	0.5981 (7)	0.3827 (5)	0.073 (2)
C14	0.3026 (8)	0.5636 (8)	0.4722 (5)	0.081 (2)
C15	0.1405 (7)	0.6294 (7)	0.4894 (4)	0.067 (2)
C16	0.0579 (7)	0.6181 (6)	0.4263 (4)	0.054 (2)
C21	0.1973 (7)	0.2465 (6)	0.1476 (5)	0.066 (2)
C22	0.3333 (10)	0.2796 (9)	0.1266 (6)	0.100 (3)
C23	0.4120 (10)	0.2754 (8)	0.0358 (5)	0.094 (3)
C24	0.4285 (12)	0.1709 (10)	0.0056 (6)	0.117 (4)
C25	0.2967 (10)	0.1373 (9)	0.0266 (6)	0.107 (3)
C26	0.2108 (7)	0.1386 (5)	0.1173 (4)	0.056 (2)
C311	0.1376 (13)	0.1679 (9)	0.3477 (8)	0.042 (3)
C32	0.1666 (9)	0.0455 (5)	0.3384 (5)	0.072 (2)
C331	0.2074 (19)	−0.0294 (15)	0.4229 (11)	0.081 (5)
C341	0.3417 (18)	−0.0081 (13)	0.4434 (11)	0.076 (4)
C35	0.3055 (10)	0.1087 (8)	0.4493 (5)	0.093 (3)
C361	0.2689 (13)	0.1911 (10)	0.3690 (7)	0.044 (3)
C312	0.1940 (14)	0.1566 (10)	0.3165 (8)	0.043 (3)
C332	0.2655 (17)	−0.0451 (12)	0.3904 (9)	0.066 (4)
C342	0.2713 (22)	−0.0060 (14)	0.4672 (10)	0.079 (5)
C362	0.2062 (16)	0.1938 (11)	0.3941 (8)	0.057 (3)
C41	−0.0968 (6)	0.2460 (5)	0.2518 (4)	0.0431 (13)
C42	−0.2083 (7)	0.2709 (7)	0.3327 (4)	0.069 (2)
C43	−0.3615 (8)	0.2585 (9)	0.3308 (6)	0.100 (3)
C44	−0.4228 (8)	0.3331 (8)	0.2584 (6)	0.090 (3)
C45	−0.3155 (7)	0.3080 (8)	0.1777 (6)	0.086 (3)
C46	−0.1633 (6)	0.3202 (6)	0.1768 (4)	0.059 (2)
H1	−0.099 (7)	0.829 (6)	0.152 (4)	0.080

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

Au—P2	2.3197 (14)	Mn1—H1	1.69 (8)
Au—P1	2.3217 (14)	Mn2—P1	2.334 (2)
Mn1—P1	2.334 (2)	Mn2—H1	1.86 (10)
Mn1—Mn2	2.9332 (13)		
P2—Au—P1	171.12 (5)	C11—P1—Mn1	115.3 (2)
P1—Mn1—Mn2	51.08 (4)	Au—P1—Mn1	123.23 (6)
P1—Mn2—Mn1	51.06 (4)	C11—P1—Mn2	115.0 (2)
Mn1—H1—Mn2	111 (4)	Au—P1—Mn2	112.89 (6)
C11—P1—Au	109.3 (2)	Mn1—P1—Mn2	77.86 (5)

Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1018). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(triethylammonium) Bis(2-mercaptobenzoato-*O*,*S*)dioxomolybdate(VI)

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Abstract

Dioxobis(2,4-pentanedionato)molybdenum reacts with mercaptobenzoic acid neutralized with triethylamine in methanol to yield the ionic compound bis(triethylammonium) (*OC*-6-12)-bis[2-mercaptobenzoato(2−)]-dioxomolybdate(VI), $(C_6H_{16}N)_2[MoO_2(C_7H_4O_2S)_2]$. This compound has been characterized by conventional spectroscopic methods and the results of the X-ray crystal structure analysis show the presence of a dianionic $[MoO_2(SC_6H_4CO_2)_2]^{2-}$ complex and two Et_3NH^+ cations.

Comment

The synthesis of the title compound, (I), was undertaken as part of our current research (Carrillo, Gouzerh & Jeannin, 1985; Bustos *et al.* 1991; Carrillo, Robert