Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* ($\mathring{A}^2$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
I	0.0	0.10662 (2)	0.75	0.0254 (2)
Mn	0.0	−0.14258 (7)	0.75	0.0212 (2)
C(1)	0.0	−0.3132 (5)	0.75	0.0261 (17)
O(1)	0.0	−0.4179 (3)	0.75	0.0398 (13)
C(2)	−0.1805 (3)	−0.13314 (18)	0.62744 (22)	0.0251 (7)
O(2)	−0.2904 (3)	−0.12566 (13)	0.55679 (18)	0.0346 (6)

Table 2. *Geometric parameters* ($\mathring{A}$, °)

I—Mn	2.6940 (8)	Mn—C(1)	1.844 (5)
Mn—C(2)	1.887 (3)	C(1)—O(1)	1.133 (6)
C(2)—O(2)	1.121 (3)		
I—Mn—C(2)	86.9 (1)	C(1)—Mn—C(2)	93.1 (1)
C(2)—Mn—C(2A)	88.3 (2)	C(2)—Mn—C(2B)	173.8 (1)
C(2A)—Mn—C(2B)	91.3 (2)	Mn—C(2)—O(2)	178.1 (2)

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A New Modification of Hg[Mn(CO)5]2

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Abstract

The molecular structure of bis(pentacarbonylmanganese)mercury shows a linear Mn—Hg—Mn bond [Mn—Hg 2.614(1) Å]. The coordination of the Mn atoms is ide-
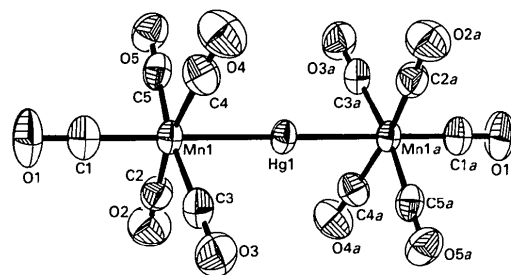
Fig. 1. Structure of Hg[Mn(CO)5]2.

alized octahedral, the CO ligands realizing an eclipsed conformation.

Comment

We have occasionally isolated crystals of Hg[Mn(CO)5]2 as a byproduct of the reaction of NaMn(CO)5 with μ-(dicyclohexylphosphanido)-dirhenium-octacarbonylmercury dichloride in THF solution at 298 K and recrystallization of the products from dichloromethane. As Guinier powder patterns of our sample proved not to be isotypic with the previously reported triclinic structure of Hg[Mn(CO)5]2 (Clegg & Wheatley, 1971; Katcher & Simon, 1972), we undertook a single-crystal structure determination. The new modification of Hg[Mn(CO)5]2 has a monoclinic structure with site symmetry $\bar{1}$ as is true for the triclinic structure. This means that the molecular geometries are the same for both structures with an eclipsed conformation of the carbonyl ligands. There is no significant difference between the measured bond lengths of the triclinic [2.610(2) Å] and the monoclinic [2.614(1) Å] structures. In the few other reported structures with Hg—Mn bonds, the bond lengths vary from 2.557(1) to 2.659(3) Å (Jaitner, Peringer, Huttner & Zolnai, 1981; Kuzmina, Ginzburg, Struchkov & Kursanov, 1983; Gäde & Weiss, 1981; Onaka, Kondo, Yamashita, Tatematsu, Kato, Goto & Ito, 1985). A comparison of the lattice energies of the two stuctures [calculated with the program *PCK*83 (Williams, 1983) and taking into account the nonbonding interactions C$\cdots$C, C$\cdots$O, O$\cdots$O with potential parameters from Mirsky (1978)] shows a slightly increased energy (−71.40 kJ mol^{-1}) for the more dense triclinic structure relative to the monoclinic one (−70.68 kJ mol^{-1}).

Experimental

Crystal data

Hg[Mn(CO)5]2	Mo $K\alpha$ radiation
M_r = 590.6	λ = 0.71073 Å
Monoclinic	Cell parameters from 25
$P2_1/c$	reflections
a = 6.462 (1) Å	θ = 8–18°
b = 6.278 (1) Å	μ = 11.76 mm^{-1}
c = 18.720 (3) Å	T = 293 K
β = 95.89 (1)°	

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

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

	x	y	z	U_{eq}
Hg1	0.0000	0.0000	0.0000	0.044 (1)
Mn1	0.8145 (1)	0.1510 (1)	0.8800 (1)	0.040 (1)
C1	0.6810 (8)	0.2585 (10)	0.7969 (3)	0.058 (3)
O1	0.5967 (8)	0.3256 (9)	0.7458 (3)	0.086 (3)
C2	0.7599 (9)	0.3781 (10)	0.9364 (3)	0.056 (3)
O2	0.7271 (9)	0.5218 (7)	0.9720 (3)	0.085 (4)
C3	1.0673 (8)	0.2717 (9)	0.8676 (3)	0.054 (3)
O3	1.2240 (6)	0.3474 (8)	0.8584 (3)	0.076 (3)
C4	0.8944 (9)	−0.1007 (11)	0.8390 (3)	0.058 (4)
O4	0.9455 (8)	−0.2528 (8)	0.8135 (3)	0.083 (3)
C5	0.5827 (8)	0.0083 (9)	0.9062 (3)	0.049 (3)
O5	0.4400 (7)	−0.0809 (8)	0.9212 (3)	0.066 (3)

Table 2. *Geometric parameters* (Å, °)

Hg1—Mn1	2.614 (1)	Mn1—C3	1.837 (5)
Mn1—C1	1.829 (6)	Mn1—C4	1.853 (6)
Mn1—C2	1.830 (6)	Mn1—C5	1.855 (5)

Mn1—Hg1—Mn1a	180.0	Hg1—Mn1—C3	85.6 (2)
Hg1—Mn1—C1	179.0 (2)	Hg1—Mn1—C4	85.6 (2)
Hg1—Mn1—C2	83.4 (2)	Hg1—Mn1—C5	84.8 (2)

$V = 755.4(2)$ Å^3

$Z = 2$

$D_x = 2.596$ Mg m^{-3}

Prism

0.21 × 0.18 × 0.15 mm

Yellow

Data collection

Siemens $R3m$/V diffractometer

ω–2θ scans

Absorption correction: empirical *via* ψ scans

$T_{min} = 0.14$, $T_{max} = 0.41$

3934 measured reflections

1749 independent reflections

1491 observed reflections [$F > 4\sigma(F)$]

$R_{int} = 0.019$

$\theta_{max} = 27.5°$

$h = -8 \rightarrow 8$

$k = 0 \rightarrow 8$

$l = 0 \rightarrow 25$

3 standard reflections monitored every 400 reflections intensity variation: 0%

Refinement

Refinement on F

Final $R = 0.027$

$wR = 0.029$

$S = 1.543$

1491 reflections

107 parameters

Calculated weights

$w = 1/[\Sigma^2(F)+0.0001F^2]$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.85$ e Å^{-3}

$\Delta\rho_{min} = -0.88$ e Å^{-3}

Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1990)

Data collection, all calculations and graphics: *SHELXTL-Plus*.

The structure was refined by full-matrix least squares with *SHELXTL-Plus*. All atoms were assigned anisotropic displacement parameters in the refinement.

Lists of structure factors, anisotropic thermal parameters and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55174 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1007]

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trans-1,3-Bis(diphenylphosphinoyl)cyclohexane

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Abstract

The relative configuration of the two diphenylphosphinoyl groups was determined as *trans* by X-ray structure analysis. The cyclohexane ring has a chair conformation with one diphenylphosphinoyl group in an equatorial position and the other in an axial position.

Comment

As part of synthetic studies of the Lewis acid-promoted double addition reactions of secondary phosphines with α,β-unsaturated aldehydes and ketones, the title compound (1) was obtained from diphenylphosphine and 2-cyclohexenone in the presence of catalytic amounts of NbCl$_5$ and stoichiometric BF$_3$.OEt$_2$ (Hashimoto, Maeta, Matsumoto, Morooka, Ohba & Suzuki, 1992). Its melting point is 555-556 K; NMR and IR data are given in Hashimoto *et al.* (1992). The O1—P1—C1—H(C1) and O2—P2—C3—H(C3) torsion angles are nearly 180°. There is an imbalance in the P—C—C bond angles for the Ph groups of 4.7(2)-7.3(2)°, which may be the result of non-bonded H···H repulsions. The H atoms of C1, C8 and C18 and those of C3, C24 and C26 are arranged in