Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

A second polymorph of [1,3-bis(diphenylphosphino)propane]tetracarbonylmolybdenum(0)

Noelia M. Sanchez Ballester, Mark R. J. Elsegood* and Martin B. Smith

Chemistry Department, Loughborough University, Loughborough, Leicestershire LE11 3TU, England
Correspondence e-mail: m.r.j.elsegood@lboro.ac.uk

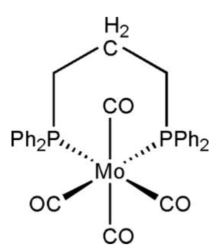
Received 2 May 2007; accepted 3 May 2007

Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.007 Å; R factor = 0.061; wR factor = 0.135; data-to-parameter ratio = 19.3.

A new polymorphic form of the title compound, $[Mo(C_{27}H_{26}P_2)(CO)_4]$, has been crystallized and structurally characterized. The new polymorph, (I), is monoclinic in the space group $P2_1/c$, with one molecule in the asymmetric unit, while the previously reported polymorph, (I′), is orthorhombic in the space group *Pnma*, with one half-molecule in the asymmetric unit and lies on a mirror plane. The geometry at the Mo centres is octahedral in both (I) and (I′), while both phosphines coordinate in a *cis* fashion.

Related literature

An orthorhombic polymorph (Ueng & Hwang, 1991) has been reported previously and shows a different packing arrangement in which molecules lie directly stacked in parallel columns with individual molecules lying on mirror planes. See also Sekabunga *et al.* (2002), Wu & Li (2003) and Balch *et al.* (1990) for broader information on phosphines in coordination chemistry and catalysis, and Sanchez Ballester *et al.* (2007) for a closely related structure.



Experimental

Crystal data

$[Mo(C_{27}H_{26}P_2)(CO)_4]$
M_r = 620.40
Monoclinic, $P2_1/n$
a = 15.5925 (6) Å
b = 8.8293 (3) Å
c = 21.0760 (6) Å
β = 91.979 (2)°
V = 2899.82 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.60 mm^{-1}
T = 120 (2) K
0.10 × 0.05 × 0.03 mm

Data collection

Bruker-Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.965, T_{max} = 0.982
33177 measured reflections
6604 independent reflections
5497 reflections with $I > 2\sigma(I)$
R_{int} = 0.076

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.135
S = 1.17
6604 reflections
343 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}

Table 1
Selected geometric parameters (Å, °) for (I) and a comparison with reported compounds (I′) and (II).

	(I)	(I′)	(II)
Mo—C(*trans* to C)	2.035 (4)/ 2.052 (5)	2.035 (7)/ 2.023 (7)	2.016 (3)/ 2.043 (3)
Mo—C(*trans* to P)	1.995 (4)/ 1.985 (5)	1.968 (5)/ 1.968 (5)	2.007 (3)/ 1.994 (3)
Mo—P	2.5239 (11)/ 2.5185 (11)	2.538 (1)/ 2.538 (1)	2.5005 (8)/ 2.4986 (8)
C—Mo—C(*trans* to C)	177.12 (17)	174.8 (3)	178.21 (12)
C—Mo—C(*cis*, av.)	89.34 (18)	88.7 (2)	89.72 (13)
P—Mo—P	89.30 (4)	89.74 (4)	86.75 (2)

Notes: (I) this work; (I′) orthorhombic polymorph (Ueng & Hwang, 1991); (II) $[Mo(CO_4)\{Ph_2PCH_2N(Ph)CH_2PPh_2\}]$ (Sanchez-Ballester *et al.*, 2007).

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

We acknowledge the EPSRC and Loughborough University for the provision of a studentship (NMSB). The authors acknowledge the EPSRC National Crystallography Service at the University of Southampton for the collection of data for (I).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2358).

References

Balch, A. L., Olmstead, M. M. & Rowley, S. P. (1990). *Inorg. Chim. Acta*, **168**, 255–264.
Bruker (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Hooft, R. W. W. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sanchez Ballester, N. M., Elsegood, M. R. J., Smith, M. B. & Brown, G. M. (2007). *Acta Cryst.* E**63**, m719–m721.
Sekabunga, E. J., Smith, M. L., Webb, T. R. & Hill, W. E. (2002). *Inorg. Chem.* **41**, 1205–1214.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. University of Göttingen, Germany.
Ueng, C.-H. & Hwang, G.-Y. (1991). *Acta Cryst.* C**47**, 522–525.
Wu, W. & Li, C.-J. (2003). *Chem. Commun.* pp. 1668–1669.

supplementary materials

Acta Cryst. (2007). E**63**, m1638 [**doi:10.1107/S160053680702185X**]

A second polymorph of [1,3-bis(diphenylphosphino)propane]tetracarbonylmolybdenum(0)

N. M. Sanchez Ballester, M. R. J. Elsegood and M. B. Smith

Comment

Ditertiary phosphines are valuable synthetic tools widely used in coordination chemistry and homogeneous catalysis. (Sekabunga *et al.*, 2002; Wu & Li, 2003; Balch *et al.*, 1990). A number of Mo—P—C—X—C—P (X = N or C) forming a six-membered chelated metallocycle have been described in the literature (Sanchez-Ballester *et al.*, 2007; Ueng & Hwang, 1991). A second polymorph of $Mo(CO)_4\{Ph_2P(CH_2)_3PPh_2\}$ **I** has been structurally determined (Figure 1), with selected geometric data in Table 1, together with those for the related compounds $Mo(CO_4)\{Ph_2P(CH_2)_3PPh_2\}$, **I'** (Ueng & Hwang, 1991), and $[Mo(CO_4)\{Ph_2PCH_2N(Ph)CH_2PPh_2\}]$, **II** (Sanchez-Ballester *et al.*, 2007). The Mo—P bond lengths and P—Mo—P bite angle in (I) are similar to (I') but slightly longer than **2**. The six-membered chelate ring in (I) adopts a chair conformation with C14 above the P_2C_2 mean plane by 0.747 (6)Å and Mo below the plane by 0.764 (3) Å. Figures 2 and 3 show packing plots of (I) and (I') viewed along the crystallographic *b* and *c* axes respectively. These show the substantial differences in packing between the two polymorphs. In (I) adjacent molecules are off-set and canted by 26° with respect to the *ab* plane with a whole molecule in the asymmetric unit, while in (I') molecules lie directly stacked in parallel columns with individual molecules lying on mirror planes.

In summary, we have reported the crystal structure of a new monoclinic, polymorph **I** that displays very similar Mo—P/Mo—CO bond lengths, bond angles and core molecular conformation to the known orthorhombic, polymorph **I'** (Ueng & Hwang, 1991), but substantial differences in crystal packing.

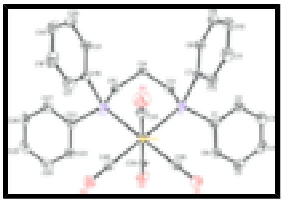
Experimental

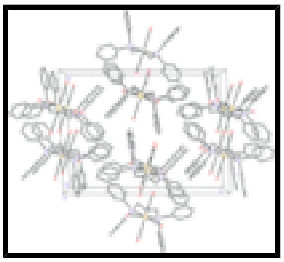
The preparation of **I** was carried out as follows. A solution of $Mo(CO)_4$(norbornadiene) (0.0408 g, 0.136 mmol) and $Ph_2P(CH_2)_3PPh_2$ (0.0559 g, 0.135 mmol) in CH_2Cl_2 (10 ml) was stirred for 12 h at room temperature under N_2. The volume was reduced to *ca.* 2-3 ml under reduced pressure. Addition of diethyl ether (20 ml) and petroleum ether (b.p. 60-80°C , 10 ml) gave a pale yellow solid which was collected by suction filtration. Yield: 0.0749 g, 89%. Suitable X-ray quality crystals of **1** were obtained by slow evaporation of the CH_2Cl_2/diethyl ether/petroleum ether filtrate. Selected data for **I**: $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ(P) 25.1 p.p.m. IR ν_{max} (KBr)/cm^{-1}: 2018, 1919, 1891, 1854 (CO).

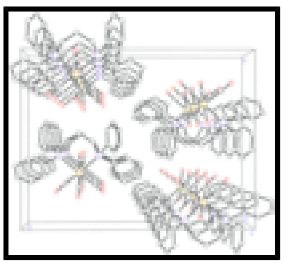
Refinement

H atoms were placed in geometric positions (C—H distance = 0.95 Å for aryl H; 0.99 Å for methylene H) using a riding model. U_{iso} values were set to $1.2U_{eq}$.

Figures







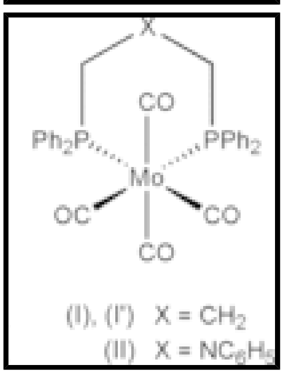


Fig. 1. View of **I**, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. All H atoms have been removed for clarity.
Fig. 2. Packing plot of **I** viewed parallel to the crystallographic *b* axis. Hydrogen atoms have been removed for clarity.
Fig. 3. Packing plot of **I'** (Ueng & Hwang, 1991) viewed parallel to the crystallographic *c* axis. H atoms have been removed for clarity.

[1,3-bis(diphenylphosphino)propane]tetracarbonylmolybdenum(0)

Crystal data

$[Mo(C_{27}H_{26}P_2)(C_1O_1)_4]$	F_{000} = 1264
M_r = 620.40	D_x = 1.421 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 6633 reflections
a = 15.5925 (6) Å	θ = 2.9–27.5°
b = 8.8293 (3) Å	μ = 0.60 mm^{-1}
c = 21.0760 (6) Å	*T* = 120 (2) K
β = 91.979 (2)°	Block, colourless

$V = 2899.82$ (17) Å^3	0.10 × 0.05 × 0.03 mm
$Z = 4$	

Data collection

Bruker-Nonius 95mm CCD camera on κ-goniostat diffractometer	6604 independent reflections
Radiation source: Bruker-Nonius FR591 rotating anode	5497 reflections with $I > 2\sigma(I)$
Monochromator: 10cm confocal mirrors	$R_{int} = 0.076$
$T = 120(2)$ K	$\theta_{max} = 27.6°$
φ and ω scans	$\theta_{min} = 3.0°$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$h = -20 \rightarrow 20$
$T_{min} = 0.965$, $T_{max} = 0.982$	$k = -11 \rightarrow 11$
33177 measured reflections	$l = -27 \rightarrow 27$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + 15.4402P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.061$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.135$	$\Delta\rho_{max} = 1.34$ e Å^{-3}
$S = 1.17$	$\Delta\rho_{min} = -0.72$ e Å^{-3}
6604 reflections	Extinction correction: none
343 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$

Mo1	0.80343 (2)	0.83793 (4)	0.487754 (16)	0.01481 (11)
C1	0.9296 (3)	0.6703 (5)	0.3555 (2)	0.0186 (8)
C2	0.9387 (3)	0.5816 (5)	0.3015 (2)	0.0242 (10)
H2	0.8974	0.5055	0.2916	0.029*
C3	1.0067 (3)	0.6023 (6)	0.2622 (2)	0.0303 (11)
H3	1.0117	0.5404	0.2256	0.036*
C4	1.0675 (3)	0.7125 (6)	0.2758 (2)	0.0287 (11)
H4	1.1143	0.7271	0.2488	0.034*
C5	1.0595 (3)	0.8013 (5)	0.3293 (2)	0.0246 (10)
H5	1.1016	0.8763	0.3392	0.029*
C6	0.9911 (3)	0.7827 (5)	0.3685 (2)	0.0207 (9)
H6	0.9858	0.8465	0.4045	0.025*
C7	0.7567 (3)	0.5721 (5)	0.3543 (2)	0.0184 (9)
C8	0.7362 (3)	0.4209 (5)	0.3431 (2)	0.0225 (9)
H8	0.7663	0.3438	0.3661	0.027*
C9	0.6719 (3)	0.3813 (6)	0.2986 (2)	0.0300 (11)
H9	0.6579	0.2777	0.2917	0.036*
C10	0.6289 (3)	0.4922 (6)	0.2646 (2)	0.0315 (12)
H10	0.5855	0.4651	0.2340	0.038*
C11	0.6485 (3)	0.6432 (6)	0.2749 (2)	0.0295 (11)
H11	0.6186	0.7195	0.2513	0.035*
C12	0.7115 (3)	0.6835 (5)	0.3196 (2)	0.0237 (9)
H12	0.7243	0.7875	0.3268	0.028*
P1	0.84233 (7)	0.63373 (12)	0.41003 (5)	0.0155 (2)
C13	0.8838 (3)	0.4563 (5)	0.4452 (2)	0.0177 (8)
H13A	0.8924	0.3829	0.4105	0.021*
H13B	0.9407	0.4771	0.4656	0.021*
C14	0.8270 (3)	0.3816 (5)	0.4945 (2)	0.0191 (9)
H14A	0.7668	0.3834	0.4780	0.023*
H14B	0.8442	0.2742	0.4992	0.023*
C15	0.8309 (3)	0.4562 (5)	0.5601 (2)	0.0189 (9)
H15A	0.8920	0.4738	0.5723	0.023*
H15B	0.8081	0.3834	0.5910	0.023*
P2	0.77312 (7)	0.63617 (12)	0.56842 (5)	0.0163 (2)
C16	0.6601 (3)	0.5822 (5)	0.5736 (2)	0.0202 (9)
C17	0.6009 (3)	0.6958 (6)	0.5873 (2)	0.0298 (11)
H17	0.6197	0.7978	0.5917	0.036*
C18	0.5153 (3)	0.6608 (7)	0.5947 (2)	0.0349 (12)
H18	0.4757	0.7387	0.6041	0.042*
C19	0.4870 (3)	0.5118 (7)	0.5882 (2)	0.0325 (12)
H19	0.4283	0.4877	0.5938	0.039*
C20	0.5442 (3)	0.4000 (6)	0.5738 (2)	0.0301 (11)
H20	0.5247	0.2987	0.5684	0.036*
C21	0.6308 (3)	0.4339 (5)	0.5667 (2)	0.0228 (9)
H21	0.6699	0.3553	0.5573	0.027*
C22	0.8008 (3)	0.6718 (5)	0.65245 (19)	0.0187 (8)
C23	0.8620 (3)	0.7794 (5)	0.6713 (2)	0.0235 (10)
H23	0.8875	0.8415	0.6403	0.028*
C24	0.8860 (3)	0.7965 (6)	0.7353 (2)	0.0273 (10)

H24	0.9280	0.8699	0.7474	0.033*
C25	0.8495 (3)	0.7081 (6)	0.7811 (2)	0.0274 (11)
H25	0.8659	0.7204	0.8246	0.033*
C26	0.7884 (3)	0.6010 (6)	0.7628 (2)	0.0283 (11)
H26	0.7626	0.5397	0.7939	0.034*
C27	0.7647 (3)	0.5830 (5)	0.6990 (2)	0.0238 (10)
H27	0.7232	0.5088	0.6871	0.029*
C28	0.8256 (3)	0.9952 (5)	0.4224 (2)	0.0184 (9)
O1	0.8362 (2)	1.0873 (4)	0.38470 (15)	0.0285 (8)
C29	0.9276 (3)	0.8437 (5)	0.5207 (2)	0.0185 (8)
O2	0.9971 (2)	0.8496 (4)	0.53965 (16)	0.0288 (7)
C30	0.7700 (3)	0.9967 (5)	0.5489 (2)	0.0202 (9)
O3	0.7490 (2)	1.0904 (4)	0.58319 (15)	0.0297 (8)
C31	0.6779 (3)	0.8437 (5)	0.4551 (2)	0.0229 (9)
O4	0.6091 (2)	0.8598 (5)	0.43742 (18)	0.0382 (9)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.01750 (18)	0.00963 (18)	0.01729 (18)	−0.00135 (15)	0.00041 (12)	−0.00034 (14)
C1	0.019 (2)	0.015 (2)	0.022 (2)	0.0007 (17)	0.0004 (16)	0.0038 (17)
C2	0.023 (2)	0.020 (2)	0.030 (2)	−0.0005 (18)	0.0050 (18)	−0.0030 (18)
C3	0.029 (3)	0.030 (3)	0.032 (3)	0.004 (2)	0.010 (2)	−0.006 (2)
C4	0.025 (3)	0.029 (3)	0.032 (3)	0.002 (2)	0.010 (2)	0.008 (2)
C5	0.019 (2)	0.020 (2)	0.035 (3)	−0.0004 (18)	−0.0008 (18)	0.0070 (19)
C6	0.021 (2)	0.016 (2)	0.025 (2)	−0.0005 (17)	0.0015 (17)	0.0013 (17)
C7	0.020 (2)	0.016 (2)	0.019 (2)	−0.0021 (17)	0.0033 (16)	−0.0028 (16)
C8	0.024 (2)	0.018 (2)	0.026 (2)	−0.0045 (18)	0.0024 (17)	−0.0054 (18)
C9	0.029 (3)	0.030 (3)	0.031 (3)	−0.010 (2)	0.001 (2)	−0.013 (2)
C10	0.025 (3)	0.043 (3)	0.026 (2)	−0.009 (2)	−0.0016 (19)	−0.007 (2)
C11	0.031 (3)	0.032 (3)	0.025 (2)	0.000 (2)	−0.0042 (19)	0.005 (2)
C12	0.026 (2)	0.020 (2)	0.025 (2)	−0.0025 (19)	−0.0012 (18)	0.0013 (18)
P1	0.0170 (5)	0.0107 (5)	0.0190 (5)	−0.0010 (4)	0.0008 (4)	−0.0002 (4)
C13	0.018 (2)	0.013 (2)	0.022 (2)	−0.0013 (16)	0.0010 (16)	0.0005 (16)
C14	0.025 (2)	0.008 (2)	0.025 (2)	−0.0022 (16)	0.0045 (17)	−0.0011 (16)
C15	0.023 (2)	0.013 (2)	0.021 (2)	−0.0002 (17)	0.0013 (16)	0.0029 (16)
P2	0.0189 (5)	0.0117 (5)	0.0182 (5)	−0.0013 (4)	0.0007 (4)	−0.0002 (4)
C16	0.017 (2)	0.024 (2)	0.020 (2)	−0.0036 (18)	0.0009 (16)	0.0048 (17)
C17	0.031 (3)	0.025 (3)	0.034 (3)	0.001 (2)	0.005 (2)	−0.002 (2)
C18	0.022 (2)	0.045 (3)	0.038 (3)	0.005 (2)	0.008 (2)	0.003 (2)
C19	0.019 (2)	0.047 (3)	0.032 (3)	−0.006 (2)	0.0029 (19)	0.005 (2)
C20	0.027 (3)	0.032 (3)	0.031 (3)	−0.015 (2)	0.000 (2)	0.005 (2)
C21	0.028 (2)	0.018 (2)	0.023 (2)	−0.0052 (19)	−0.0010 (17)	0.0011 (17)
C22	0.022 (2)	0.013 (2)	0.020 (2)	0.0055 (18)	0.0012 (16)	−0.0030 (16)
C23	0.028 (2)	0.016 (2)	0.026 (2)	−0.0014 (19)	−0.0006 (18)	−0.0018 (18)
C24	0.032 (3)	0.021 (2)	0.029 (2)	0.000 (2)	−0.0040 (19)	−0.0054 (19)
C25	0.035 (3)	0.027 (3)	0.019 (2)	0.010 (2)	−0.0027 (19)	−0.0040 (18)
C26	0.035 (3)	0.030 (3)	0.020 (2)	0.004 (2)	0.0072 (19)	0.0017 (19)

C27	0.023 (2)	0.024 (2)	0.025 (2)	−0.0018 (19)	0.0029 (18)	−0.0003 (18)
C28	0.021 (2)	0.015 (2)	0.019 (2)	−0.0014 (17)	0.0004 (16)	−0.0009 (16)
O1	0.045 (2)	0.0165 (17)	0.0241 (17)	0.0013 (15)	0.0010 (14)	0.0027 (13)
C29	0.022 (2)	0.0091 (19)	0.025 (2)	−0.0034 (17)	0.0009 (17)	0.0002 (16)
O2	0.0236 (18)	0.0283 (19)	0.0342 (18)	−0.0024 (15)	−0.0047 (14)	0.0040 (15)
C30	0.022 (2)	0.016 (2)	0.023 (2)	−0.0033 (18)	0.0022 (17)	0.0017 (17)
O3	0.043 (2)	0.0217 (18)	0.0245 (17)	0.0036 (16)	0.0052 (15)	−0.0011 (14)
C31	0.025 (2)	0.019 (2)	0.025 (2)	−0.0050 (19)	−0.0034 (17)	−0.0011 (18)
O4	0.0215 (18)	0.046 (2)	0.046 (2)	−0.0021 (17)	−0.0058 (16)	0.0017 (18)

Geometric parameters (Å, °)

Mo1—C30	1.985 (5)	C14—C15	1.532 (6)
Mo1—C28	1.995 (4)	C14—H14A	0.9900
Mo1—C29	2.035 (4)	C14—H14B	0.9900
Mo1—C31	2.052 (5)	C15—P2	1.838 (4)
Mo1—P2	2.5185 (11)	C15—H15A	0.9900
Mo1—P1	2.5239 (11)	C15—H15B	0.9900
C1—C2	1.392 (6)	P2—C16	1.832 (4)
C1—C6	1.401 (6)	P2—C22	1.836 (4)
C1—P1	1.840 (4)	C16—C21	1.393 (6)
C2—C3	1.380 (6)	C16—C17	1.401 (7)
C2—H2	0.9500	C17—C18	1.384 (7)
C3—C4	1.382 (7)	C17—H17	0.9500
C3—H3	0.9500	C18—C19	1.393 (8)
C4—C5	1.381 (7)	C18—H18	0.9500
C4—H4	0.9500	C19—C20	1.371 (8)
C5—C6	1.381 (6)	C19—H19	0.9500
C5—H5	0.9500	C20—C21	1.396 (7)
C6—H6	0.9500	C20—H20	0.9500
C7—C8	1.391 (6)	C21—H21	0.9500
C7—C12	1.400 (6)	C22—C27	1.390 (6)
C7—P1	1.831 (4)	C22—C23	1.394 (6)
C8—C9	1.394 (6)	C23—C24	1.396 (6)
C8—H8	0.9500	C23—H23	0.9500
C9—C10	1.375 (8)	C24—C25	1.381 (7)
C9—H9	0.9500	C24—H24	0.9500
C10—C11	1.384 (7)	C25—C26	1.388 (7)
C10—H10	0.9500	C25—H25	0.9500
C11—C12	1.385 (7)	C26—C27	1.389 (7)
C11—H11	0.9500	C26—H26	0.9500
C12—H12	0.9500	C27—H27	0.9500
P1—C13	1.841 (4)	C28—O1	1.152 (5)
C13—C14	1.537 (6)	C29—O2	1.143 (5)
C13—H13A	0.9900	C30—O3	1.154 (5)
C13—H13B	0.9900	C31—O4	1.133 (6)
C30—Mo1—C28	90.68 (17)	P1—C13—H13B	108.3
C30—Mo1—C29	91.63 (18)	H13A—C13—H13B	107.4
C28—Mo1—C29	91.88 (17)	C15—C14—C13	114.8 (4)

C30—Mo1—C31	86.08 (19)	C15—C14—H14A	108.6
C28—Mo1—C31	86.41 (18)	C13—C14—H14A	108.6
C29—Mo1—C31	177.12 (17)	C15—C14—H14B	108.6
C30—Mo1—P2	90.11 (13)	C13—C14—H14B	108.6
C28—Mo1—P2	178.65 (13)	H14A—C14—H14B	107.5
C29—Mo1—P2	89.19 (12)	C14—C15—P2	116.9 (3)
C31—Mo1—P2	92.56 (13)	C14—C15—H15A	108.1
C30—Mo1—P1	178.66 (13)	P2—C15—H15A	108.1
C28—Mo1—P1	89.88 (13)	C14—C15—H15B	108.1
C29—Mo1—P1	89.56 (12)	P2—C15—H15B	108.1
C31—Mo1—P1	92.75 (13)	H15A—C15—H15B	107.3
P2—Mo1—P1	89.30 (4)	C16—P2—C22	100.5 (2)
C2—C1—C6	118.1 (4)	C16—P2—C15	104.9 (2)
C2—C1—P1	120.7 (3)	C22—P2—C15	98.03 (19)
C6—C1—P1	121.2 (3)	C16—P2—Mo1	115.30 (14)
C3—C2—C1	121.3 (4)	C22—P2—Mo1	119.13 (15)
C3—C2—H2	119.4	C15—P2—Mo1	116.30 (14)
C1—C2—H2	119.4	C21—C16—C17	118.6 (4)
C2—C3—C4	120.2 (5)	C21—C16—P2	123.5 (4)
C2—C3—H3	119.9	C17—C16—P2	117.9 (4)
C4—C3—H3	119.9	C18—C17—C16	120.5 (5)
C5—C4—C3	119.3 (4)	C18—C17—H17	119.7
C5—C4—H4	120.4	C16—C17—H17	119.7
C3—C4—H4	120.4	C17—C18—C19	120.3 (5)
C6—C5—C4	120.9 (4)	C17—C18—H18	119.9
C6—C5—H5	119.5	C19—C18—H18	119.9
C4—C5—H5	119.5	C20—C19—C18	119.6 (5)
C5—C6—C1	120.3 (4)	C20—C19—H19	120.2
C5—C6—H6	119.9	C18—C19—H19	120.2
C1—C6—H6	119.9	C19—C20—C21	120.6 (5)
C8—C7—C12	118.6 (4)	C19—C20—H20	119.7
C8—C7—P1	123.5 (3)	C21—C20—H20	119.7
C12—C7—P1	117.9 (3)	C16—C21—C20	120.4 (5)
C7—C8—C9	120.7 (5)	C16—C21—H21	119.8
C7—C8—H8	119.7	C20—C21—H21	119.8
C9—C8—H8	119.7	C27—C22—C23	118.2 (4)
C10—C9—C8	119.9 (5)	C27—C22—P2	119.8 (3)
C10—C9—H9	120.0	C23—C22—P2	121.8 (3)
C8—C9—H9	120.0	C22—C23—C24	120.4 (4)
C9—C10—C11	120.2 (5)	C22—C23—H23	119.8
C9—C10—H10	119.9	C24—C23—H23	119.8
C11—C10—H10	119.9	C25—C24—C23	120.8 (5)
C10—C11—C12	120.2 (5)	C25—C24—H24	119.6
C10—C11—H11	119.9	C23—C24—H24	119.6
C12—C11—H11	119.9	C24—C25—C26	119.1 (4)
C11—C12—C7	120.4 (4)	C24—C25—H25	120.5
C11—C12—H12	119.8	C26—C25—H25	120.5
C7—C12—H12	119.8	C25—C26—C27	120.3 (4)
C7—P1—C1	100.96 (19)	C25—C26—H26	119.9

C7—P1—C13	104.1 (2)	C27—C26—H26	119.9
C1—P1—C13	98.26 (19)	C26—C27—C22	121.2 (5)
C7—P1—Mo1	116.27 (14)	C26—C27—H27	119.4
C1—P1—Mo1	118.66 (15)	C22—C27—H27	119.4
C13—P1—Mo1	115.81 (14)	O1—C28—Mo1	178.3 (4)
C14—C13—P1	115.8 (3)	O2—C29—Mo1	178.7 (4)
C14—C13—H13A	108.3	O3—C30—Mo1	178.2 (4)
P1—C13—H13A	108.3	O4—C31—Mo1	174.2 (4)
C14—C13—H13B	108.3		

C6—C1—C2—C3	0.7 (7)	C13—C14—C15—P2	75.6 (4)
P1—C1—C2—C3	−176.3 (4)	C14—C15—P2—C16	77.7 (4)
C1—C2—C3—C4	0.0 (8)	C14—C15—P2—C22	−179.1 (3)
C2—C3—C4—C5	0.1 (8)	C14—C15—P2—Mo1	−50.9 (4)
C3—C4—C5—C6	−0.9 (7)	C30—Mo1—P2—C16	81.8 (2)
C4—C5—C6—C1	1.6 (7)	C29—Mo1—P2—C16	173.4 (2)
C2—C1—C6—C5	−1.4 (6)	C31—Mo1—P2—C16	−4.3 (2)
P1—C1—C6—C5	175.5 (3)	P1—Mo1—P2—C16	−97.04 (17)
C12—C7—C8—C9	−0.2 (7)	C30—Mo1—P2—C22	−37.9 (2)
P1—C7—C8—C9	−178.0 (3)	C29—Mo1—P2—C22	53.8 (2)
C7—C8—C9—C10	0.8 (7)	C31—Mo1—P2—C22	−124.0 (2)
C8—C9—C10—C11	−0.6 (8)	P1—Mo1—P2—C22	143.33 (16)
C9—C10—C11—C12	−0.1 (8)	C30—Mo1—P2—C15	−154.9 (2)
C10—C11—C12—C7	0.7 (7)	C29—Mo1—P2—C15	−63.2 (2)
C8—C7—C12—C11	−0.6 (7)	C31—Mo1—P2—C15	119.1 (2)
P1—C7—C12—C11	177.4 (4)	P1—Mo1—P2—C15	26.34 (16)
C8—C7—P1—C1	99.0 (4)	C22—P2—C16—C21	−106.0 (4)
C12—C7—P1—C1	−78.8 (4)	C15—P2—C16—C21	−4.7 (4)
C8—C7—P1—C13	−2.5 (4)	Mo1—P2—C16—C21	124.6 (3)
C12—C7—P1—C13	179.7 (3)	C22—P2—C16—C17	71.7 (4)
C8—C7—P1—Mo1	−131.1 (3)	C15—P2—C16—C17	173.0 (4)
C12—C7—P1—Mo1	51.0 (4)	Mo1—P2—C16—C17	−57.7 (4)
C2—C1—P1—C7	−34.5 (4)	C21—C16—C17—C18	0.7 (7)
C6—C1—P1—C7	148.6 (4)	P2—C16—C17—C18	−177.1 (4)
C2—C1—P1—C13	71.7 (4)	C16—C17—C18—C19	−0.2 (8)
C6—C1—P1—C13	−105.2 (4)	C17—C18—C19—C20	−0.9 (8)
C2—C1—P1—Mo1	−162.8 (3)	C18—C19—C20—C21	1.4 (8)
C6—C1—P1—Mo1	20.3 (4)	C17—C16—C21—C20	−0.2 (7)
C28—Mo1—P1—C7	−83.9 (2)	P2—C16—C21—C20	177.4 (3)
C29—Mo1—P1—C7	−175.8 (2)	C19—C20—C21—C16	−0.8 (7)
C31—Mo1—P1—C7	2.5 (2)	C16—P2—C22—C27	35.7 (4)
P2—Mo1—P1—C7	95.04 (16)	C15—P2—C22—C27	−71.1 (4)
C28—Mo1—P1—C1	36.9 (2)	Mo1—P2—C22—C27	162.6 (3)
C29—Mo1—P1—C1	−55.0 (2)	C16—P2—C22—C23	−148.8 (4)
C31—Mo1—P1—C1	123.3 (2)	C15—P2—C22—C23	104.4 (4)
P2—Mo1—P1—C1	−144.21 (16)	Mo1—P2—C22—C23	−21.8 (4)
C28—Mo1—P1—C13	153.4 (2)	C27—C22—C23—C24	0.1 (7)
C29—Mo1—P1—C13	61.5 (2)	P2—C22—C23—C24	−175.5 (4)
C31—Mo1—P1—C13	−120.2 (2)	C22—C23—C24—C25	−0.3 (7)
P2—Mo1—P1—C13	−27.70 (16)	C23—C24—C25—C26	0.2 (7)

C7—P1—C13—C14	−75.1 (3)	C24—C25—C26—C27	0.2 (7)
C1—P1—C13—C14	−178.7 (3)	C25—C26—C27—C22	−0.5 (7)
Mo1—P1—C13—C14	53.8 (3)	C23—C22—C27—C26	0.3 (7)
P1—C13—C14—C15	−77.1 (4)	P2—C22—C27—C26	176.0 (4)

Selected geometric parameters (Å, °) for (I) and a comparison with reported compounds (I') and (II)

	1	1'	2
Mo—C(trans to C)	2.035 (4)/2.052 (5)	2.035 (7)/2.023 (7)	2.016 (3)/2.043 (3)
Mo—C(trans to P)	1.995 (4)/1.985 (5)	1.968 (5)/1.968 (5)	2.007 (3)/1.994 (3)
Mo—P	2.5239 (11)/2.5185 (11)	2.538 (1)/2.538 (1)	2.5005 (8)/2.4986 (8)
C—Mo—C(trans to C)	177.12 (17)	174.8 (3)	178.21 (12)
C—Mo—C(cis, av.)	89.34 (18)	88.7 (2)	89.72 (13)
P—Mo—P	89.30 (4)	89.74 (4)	86.75 (2)

Notes: (I) this work; (I') orthorhombic polymorph (Ueng & Hwang, 1991); (II) [Mo(CO_4){$Ph_2PCH_2N(Ph)CH_2PPh_2$}] (Sanchez-Ballester *et al.*, 2007).

Fig. 1

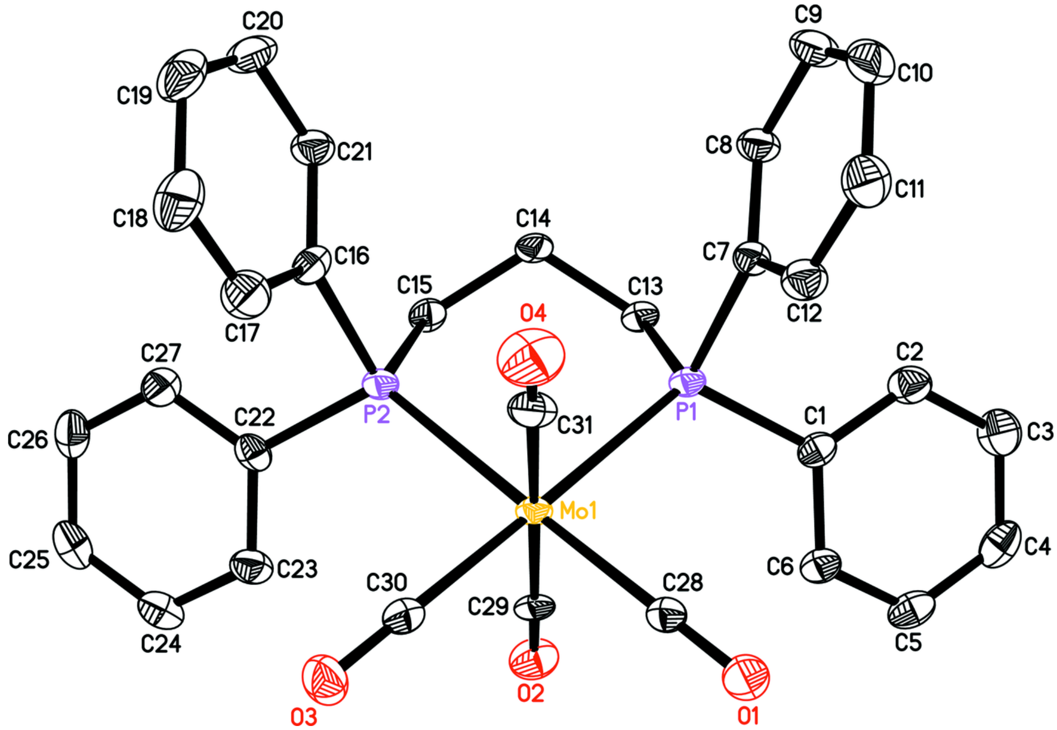

C20
C9
C19
C10
C21
C8
C18
C11
C14
C16
C7
C15
C13
C12
C17
O4
C27
C2
C22
P2
P1
C1
C26
C31
C3
Mo1
C25
C23
C6
C4
C30
C28
C29
C24
C5
O3
O2
O1

Fig. 2

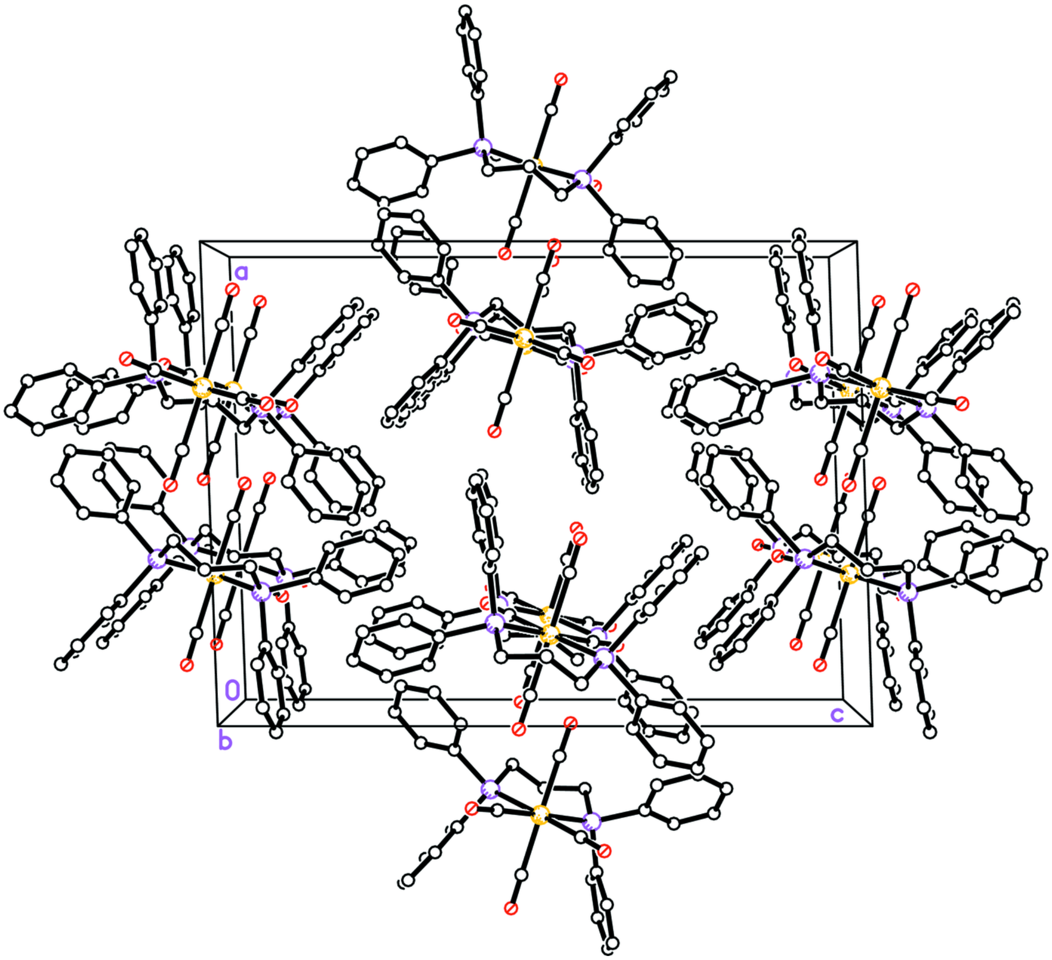


Fig. 3

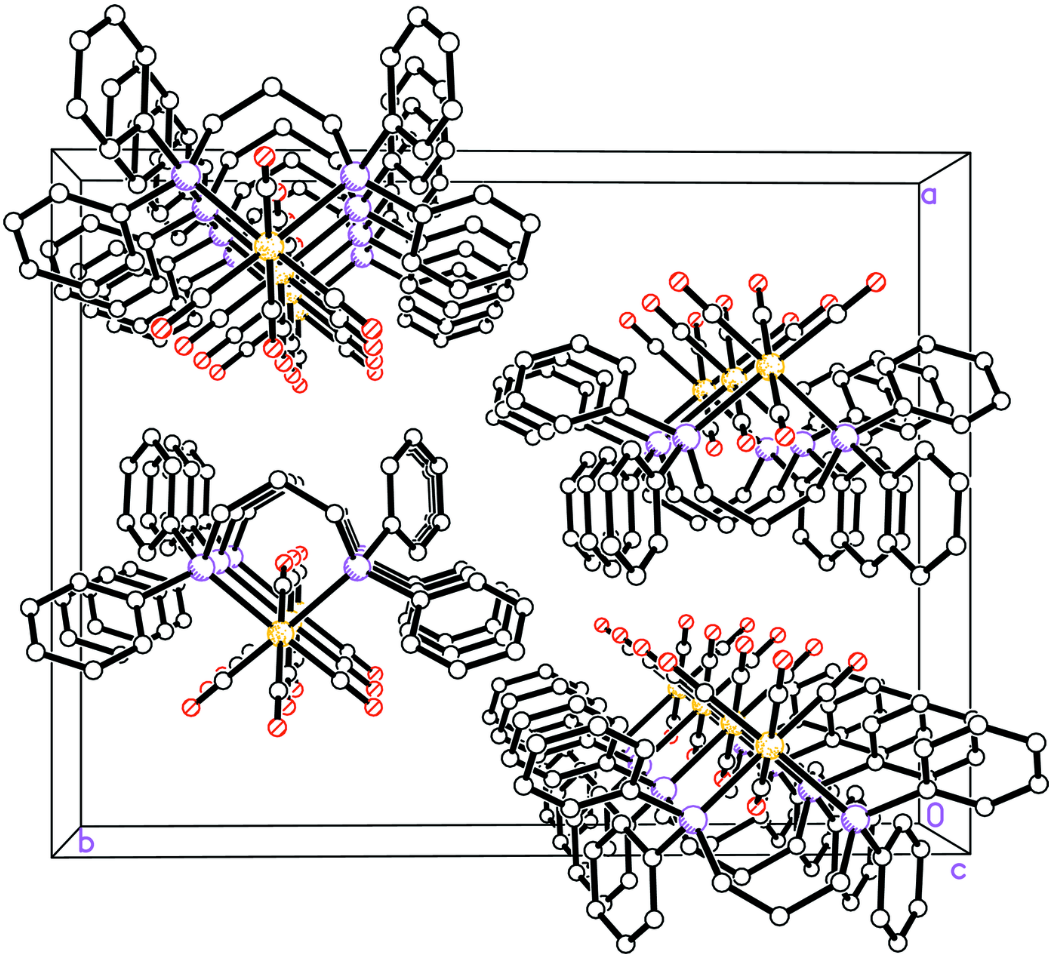
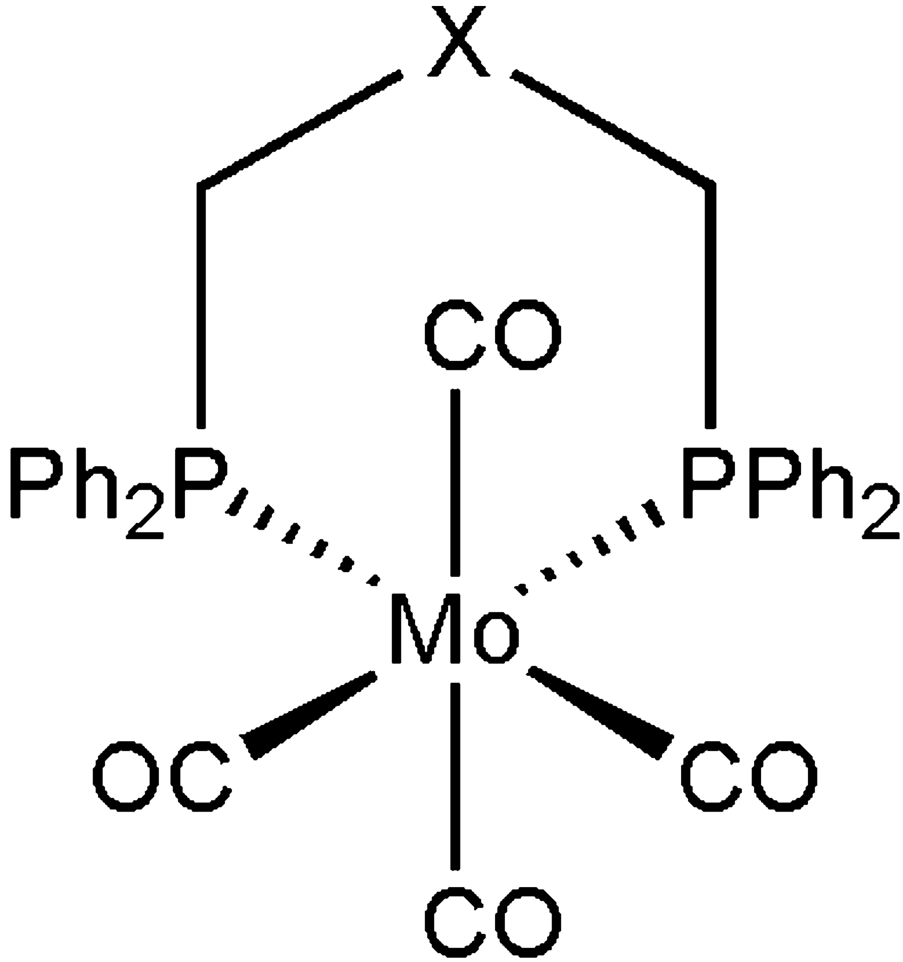


Fig. 4



(I), (I') X = CH_2

(II) X = NC_6H_5