Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

{*N*,*N*-Bis[(diphenylphosphino)methyl]aniline}-tetracarbonylmolybdenum(0)

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

Chemistry Department, Loughborough University, Leicestershire LE11 3TU, England

Correspondence e-mail:
m.r.j.elsegood@lboro.ac.uk

Key indicators

Single-crystal synchrotron study
T = 120 K
Mean σ(C–C) = 0.005 Å
R factor = 0.059
wR factor = 0.164
Data-to-parameter ratio = 25.3

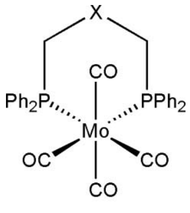
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 30 January 2007
Accepted 5 February 2007

The title compound, [Mo(CO)$_4${Ph$_2$PCH$_2$N(Ph)CH$_2$PPh$_2$}] or [Mo(C$_{32}$H$_{29}$NP$_2$)(CO)$_4$], is a tetracarbonylmolybdenum(0) complex of a chelating ditertiary phosphine with a P—C—N—C—P backbone. The geometry at the Mo centre is octahedral, while both diphenylphosphino centres coordinate in a *cis* fashion.

Comment

Organometallic compounds containing the group 6 metals Mo, Cr and W have been extensively studied over the past few decades for a variety of substitution reactions, one example being with phosphine ligands. Ligand substitution reactions have been accomplished in several ways, including under thermal or photolysis conditions, or displacement of labile precursors [*e.g.* piperidine, norbornadiene (nbd), THF, CH$_3$CN] from appropriate Mo starting materials. Thus, neutral, octahedral compounds of the general type Mo(CO)$_n$(P*R*$_3$)$_{6-n}$ (*n* = 3–5) can be obtained using monodentate tertiary phosphines. Of these, one particular class of compound of interest are the tetracarbonylmolybdenum(0) diphosphine complexes Mo(CO)$_4$(P—P) [P—P is a symmetric (Bookham *et al.*, 1993; Fernández *et al.*, 1996; Gaw *et al.*, 2000, 2002; Powell *et al.*, 1992) or non-symmetric ligand (Affandi *et al.*, 1989)]. Recent interest has also focused on tetracarbonylmolybdenum(0) complexes with bidentate ligands bearing group 15 (Heinze & Jacob, 2002) or group 16 donor centres (Heuer *et al.*, 2002). We describe here the synthesis of Mo(CO)$_4${Ph$_2$PCH$_2$N(Ph)CH$_2$PPh$_2$}, (1), and its single-crystal X-ray structure.



(1) X = NC$_6$H$_5$
(2) X = CH$_2$
(3) X = CCH$_2$

The molecular structure of compound (1) is shown in Fig. 1, with selected geometric data in Table 1, together with those for the related compounds Mo(CO$_4$){Ph$_2$P(CH$_2$)$_3$PPh$_2$}, (2) (Ueng & Hwang, 1991), and Mo(CO$_4$){Ph$_2$PCH$_2$C(CH$_2$)CH$_2$PPh$_2$}, (3) (Bookham *et al.*, 1993). The structure of (1) comprises a *cis*-chelating Ph$_2$PCH$_2$N(Ph)CH$_2$PPh$_2$ ligand and four terminal CO ligands. The Mo—P bond lengths in (1) are slightly shorter than those of (2) and (3). The variations in

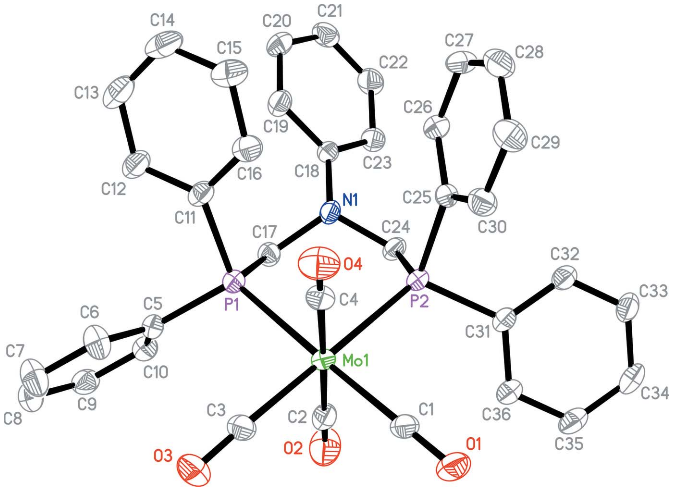


Figure 1
The molecular structure of (1), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. All H atoms have been omitted for clarity.

Mo—C distances are as expected for the different π-acceptor properties of CO and –PPh_2 groups. The Mo—C—O bond angles are all close to linear. The P—Mo—P bite angle is similar to those of (2) and (3). As anticipated, this bite angle is enlarged with respect to those found in complexes of the type $Mo(CO)_4\{Ph_2PN(R)PPh_2\}$ [R = H, P—Mo—P 65.29 (6)°; R = 2-$MeOC_6H_4$, P—Mo—P = 65.78 (2)°] in which the chelating $Ph_2PN(R)PPh_2$ ligands adopt near planar four-membered ring conformations (Gaw *et al.*, 2000; Knorr & Strohmann, 1999). The six-membered chelate ring in (1) adopts a chair conformation with N1 above the P_2C_2 mean plane by 0.736 (3) Å and Mo below the plane by 0.986 (2) Å. The Mo—P—C—N—C—P metallacyclic ring is similar to those previously seen for other M—P—C—N—C—P compounds (Zhang *et al.*, 2002). The *N*-arene is twisted about the N1—C18 axis by 47.2 (2)° with respect to the central heterocycle.

Experimental

A solution of $Mo(CO)_4(nbd)$ (0.0613 g, 0.206 mmol) and $Ph_2PCH_2N(Ph)CH_2PPh_2$ (0.101 g, 0.206 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. 333–353 K, 10 ml) gave a pale-yellow solid which was collected by suction filtration. Yield 0.071 g, 50%. X-ray quality crystals of (1) were obtained by slow evaporation of the CH_2Cl_2/diethyl ether/petroleum ether filtrate. Calculated for $C_{36}H_{29}MoNO_4P_2\cdot0.5C_6H_{14}$: C 63.25, H 4.90, N 1.89; found: C 63.09, H 4.75, N 1.87%.

Crystal data

$[Mo(C_{32}H_{29}NP_2)(CO)_4]$
$M_r = 697.48$
Triclinic, $P\overline{1}$
$a = 10.2072$ (6) Å
$b = 11.2800$ (7) Å
$c = 14.5527$ (9) Å
$\alpha = 100.047$ (1)°
$\beta = 93.162$ (1)°
$\gamma = 101.316$ (1)°
$V = 1610.84$ (17) Å^3
$Z = 2$
$D_x = 1.438$ Mg m^{-3}
Synchrotron radiation
$\lambda = 0.6910$ Å
$\mu = 0.55$ mm^{-1}
$T = 120$ (2) K
Plate, colourless
0.15 × 0.06 × 0.03 mm

Data collection

Bruker APEX II CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.923$, $T_{max} = 0.984$
19074 measured reflections
10047 independent reflections
8204 reflections with $I > 2\sigma(I)$
$R_{int} = 0.065$
$\theta_{max} = 31.0$°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.164$
$S = 1.00$
10047 reflections
397 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0783P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.70$ e Å^{-3}
$\Delta\rho_{min} = -0.97$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °) for (1) and a comparison with reported compounds (2) and (3).

	(1)	(2)	(3)
Mo—C (*trans* to C)	2.016 (3)/2.043 (3)	2.035 (7)/2.023 (7)	2.016 (4)/2.030 (4)
Mo—C (*trans* to P)	2.007 (3)/1.994 (3)	1.968 (5)/1.968 (5)	1.999 (4)/1.986 (4)
Mo—P	2.5005 (8)/2.4986 (8)	2.538 (1)/2.538 (1)	2.5199 (11)/2.5094 (13)
C—Mo—C (*trans* to C)	178.21 (12)	174.8 (3)	171.0 (2)
C—Mo—C (*cis*, av.)	89.72 (13)	88.7 (2)	88.3 (2)
P—Mo—P	86.75 (2)	89.74 (4)	85.14 (4)

(1) This work. (2) Ueng & Hwang (1991). (3) Bookham *et al.* (1993).

H atoms were positioned geometrically (C—H = 0.95 Å for aryl H and 0.99 Å for methylene H), and refined using a riding model. U_{iso}(H) values were set to $1.2U_{eq}$(C).

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; 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 studentships (GMB, NMSB). The authors acknowledge Dr John Warren and Dr Timothy Prior for scientific support at Daresbury Laboratory Station 9.8 during the data collection of (1).

References

Affandi, S., Nelson, J. H. & Fischer, J. (1989). *Inorg. Chem.* **28**, 4536–4544.
Bookham, J. L., Clegg, W., McFarlane, W. & Raper, E. S. (1993). *J. Chem. Soc. Dalton Trans.* pp. 3567–3573.
Bruker (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). *APEX2* and *SAINT* (Version 7.23A). Bruker AXS Inc., Madison, Wisconsin, USA.
Fernández, E. J., Gimeno, M. C., Jones, P. G., Laguna, A., Laguna, M. & Olmos, E. (1996). *J. Chem. Soc. Dalton Trans.* pp. 3603–3608.
Gaw, K. G., Smith, M. B. & Slawin, A. M. Z. (2000). *New J. Chem.* **24**, 429–435.
Gaw, K. G., Smith, M. B. & Steed, J. W. (2002). *J. Organomet. Chem.* **664**, 294–297.
Heinze, K. & Jacob, V. (2002). *J. Chem. Soc. Dalton Trans.* pp. 2379–2385.
Heuer, B., Matthews, M. L., Reid, G. & Ripley, M. (2002). *J. Organomet. Chem.* **655**, 55–62.
Knorr, M. & Strohmann, C. (1999). *Organometallics*, **18**, 248–257.

Powell, J., Lough, A. & Wang, F. (1992). *Organometallics*, **11**, 2289–2295.
Sheldrick, G. M. (2004). *SADABS*. Version 2004/1. University of Göttingen, Germany.
Ueng, C.-H. & Hwang, G.-Y. (1991). *Acta Cryst.* C**47**, 522–525.
Zhang, J., Vittal, J. J., Henderson, W., Wheaton, J. R., Hall, I. H., Hor, T. S. A. & Yan, Y. K. (2002). *J. Organomet. Chem.* **650**, 123–132.

supporting information

Acta Cryst. (2007). E**63**, m719–m721 [https://doi.org/10.1107/S1600536807006101]

{*N*,*N*-Bis[(diphenylphosphino)methyl]aniline}tetracarbonylmolybdenum(0)

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

{*N*,*N*-bis[(diphenylphosphino)methyl]aniline}tetracarbonylmolybdenum(0)

Crystal data

$[Mo(C_{32}H_{29}NP_2)(CO)_4]$
$M_r = 697.48$
Triclinic, $P\bar{1}$
Hall symbol: -P 1
$a = 10.2072$ (6) Å
$b = 11.2800$ (7) Å
$c = 14.5527$ (9) Å
$\alpha = 100.047$ (1)°
$\beta = 93.162$ (1)°
$\gamma = 101.316$ (1)°
$V = 1610.84$ (17) Å^3
$Z = 2$
$F(000) = 712$
$D_x = 1.438$ Mg m^{-3}
Synchrotron radiation, $\lambda = 0.6910$ Å
Cell parameters from 4141 reflections
$\theta = 2.4$–29.2°
$\mu = 0.55$ mm^{-1}
$T = 120$ K
Plate, colourless
0.15 × 0.06 × 0.03 mm

Data collection

Bruker APEX II CCD diffractometer
Radiation source: Daresbury SRS station 9.8
Silicon 111 monochromator
ω rotation with narrow frames scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)
$T_{min} = 0.923$, $T_{max} = 0.984$
19074 measured reflections
10047 independent reflections
8204 reflections with $I > 2\sigma(I)$
$R_{int} = 0.065$
$\theta_{max} = 31.0°$, $\theta_{min} = 1.4°$
$h = -15 \rightarrow 15$
$k = -16 \rightarrow 16$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.164$
$S = 1.00$
10047 reflections
397 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0783P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.70$ e Å^{-3}
$\Delta\rho_{min} = -0.97$ e Å^{-3}

Special details

Experimental. ^{1}H NMR (400 MHz, $CDCl_3$, p.p.m.): δ 7.54–6.02 (arom. H, 25H, *m*), 3.99 (CH_2, 4H, *s*). $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$, p.p.m.): δ 18.8. IR ν_{max} (KBr, cm^{-1}): 2024, 1927, 1901, 1885 (CO). Calculated for $C_{36}H_{29}MoNO_4P_2\cdot0.5C_6H_{14}$: C, 63.25; H, 4.90; N, 1.89. Found: C, 63.09; H, 4.75; N, 1.87%.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Mo1	−0.13066 (2)	0.85871 (2)	0.688421 (16)	0.01970 (8)
C1	−0.1835 (3)	1.0108 (3)	0.6580 (2)	0.0247 (6)
O1	−0.2141 (2)	1.0981 (2)	0.64239 (18)	0.0354 (5)
C2	−0.2241 (3)	0.8728 (3)	0.8072 (2)	0.0296 (6)
O2	−0.2767 (3)	0.8813 (3)	0.8753 (2)	0.0477 (7)
C3	−0.2992 (3)	0.7540 (3)	0.6186 (2)	0.0253 (6)
O3	−0.3979 (2)	0.6962 (2)	0.57755 (18)	0.0379 (6)
C4	−0.0417 (3)	0.8423 (3)	0.5657 (2)	0.0250 (6)
O4	0.0025 (3)	0.8353 (3)	0.49443 (17)	0.0362 (5)
P1	−0.05987 (7)	0.67674 (7)	0.73650 (5)	0.02001 (15)
C5	−0.1977 (3)	0.5496 (3)	0.7452 (2)	0.0236 (6)
C6	−0.2651 (4)	0.4766 (3)	0.6616 (2)	0.0344 (7)
H6	−0.2382	0.4934	0.6029	0.041*
C7	−0.3715 (4)	0.3796 (4)	0.6649 (3)	0.0418 (9)
H7	−0.4158	0.3292	0.6082	0.050*
C8	−0.4136 (4)	0.3557 (3)	0.7501 (3)	0.0372 (8)
H8	−0.4865	0.2894	0.7517	0.045*
C9	−0.3492 (3)	0.4286 (3)	0.8324 (3)	0.0322 (7)
H9	−0.3780	0.4123	0.8907	0.039*
C10	−0.2417 (3)	0.5265 (3)	0.8305 (2)	0.0281 (6)
H10	−0.1988	0.5771	0.8875	0.034*
C11	0.0602 (3)	0.5999 (3)	0.6749 (2)	0.0233 (5)
C12	0.0658 (3)	0.4767 (3)	0.6789 (2)	0.0310 (7)
H12	−0.0026	0.4271	0.7054	0.037*
C13	0.1717 (4)	0.4291 (3)	0.6437 (3)	0.0381 (8)
H13	0.1745	0.3459	0.6458	0.046*
C14	0.2736 (4)	0.4995 (4)	0.6056 (3)	0.0393 (8)
H14	0.3472	0.4662	0.5838	0.047*
C15	0.2665 (4)	0.6200 (4)	0.5997 (2)	0.0348 (7)
H15	0.3343	0.6686	0.5719	0.042*
C16	0.1609 (3)	0.6692 (3)	0.6344 (2)	0.0281 (6)
H16	0.1574	0.7517	0.6304	0.034*
C17	0.0254 (3)	0.7136 (3)	0.8578 (2)	0.0224 (5)
H17A	−0.0370	0.7426	0.9021	0.027*
H17B	0.0463	0.6375	0.8744	0.027*
N1	0.1492 (2)	0.8075 (2)	0.86898 (17)	0.0220 (5)

C18	0.2662 (3)	0.7786 (3)	0.9091 (2)	0.0209 (5)
C19	0.3125 (3)	0.6768 (3)	0.8644 (2)	0.0279 (6)
H19	0.2619	0.6251	0.8103	0.034*
C20	0.4310 (3)	0.6502 (3)	0.8977 (2)	0.0303 (7)
H20	0.4608	0.5806	0.8663	0.036*
C21	0.5060 (3)	0.7235 (3)	0.9761 (3)	0.0331 (7)
H21	0.5878	0.7055	0.9984	0.040*
C22	0.4607 (3)	0.8236 (3)	1.0219 (2)	0.0321 (7)
H22	0.5114	0.8739	1.0765	0.038*
C23	0.3413 (3)	0.8522 (3)	0.9892 (2)	0.0261 (6)
H23	0.3115	0.9214	1.0214	0.031*
C24	0.1326 (3)	0.9344 (3)	0.8885 (2)	0.0227 (5)
H24A	0.2187	0.9886	0.9173	0.027*
H24B	0.0650	0.9421	0.9341	0.027*
P2	0.07867 (7)	0.98633 (7)	0.78070 (5)	0.01930 (15)
C25	0.2311 (3)	1.0066 (3)	0.71928 (19)	0.0204 (5)
C26	0.3469 (3)	0.9680 (3)	0.7441 (2)	0.0253 (6)
H26	0.3504	0.9278	0.7961	0.030*
C27	0.4581 (3)	0.9880 (3)	0.6929 (2)	0.0299 (6)
H27	0.5370	0.9614	0.7103	0.036*
C28	0.4542 (3)	1.0466 (3)	0.6169 (2)	0.0312 (7)
H28	0.5307	1.0612	0.5829	0.037*
C29	0.3375 (3)	1.0839 (3)	0.5905 (2)	0.0323 (7)
H29	0.3338	1.1231	0.5380	0.039*
C30	0.2263 (3)	1.0632 (3)	0.6417 (2)	0.0274 (6)
H30	0.1465	1.0880	0.6235	0.033*
C31	0.0780 (3)	1.1464 (3)	0.8338 (2)	0.0220 (5)
C32	0.1966 (3)	1.2350 (3)	0.8529 (2)	0.0260 (6)
H32	0.2789	1.2128	0.8374	0.031*
C33	0.1961 (3)	1.3553 (3)	0.8943 (2)	0.0296 (6)
H33	0.2778	1.4150	0.9066	0.036*
C34	0.0770 (3)	1.3888 (3)	0.9178 (2)	0.0286 (6)
H34	0.0767	1.4714	0.9457	0.034*
C35	−0.0418 (3)	1.3012 (3)	0.9006 (2)	0.0303 (7)
H35	−0.1235	1.3234	0.9176	0.036*
C36	−0.0412 (3)	1.1803 (3)	0.8581 (2)	0.0265 (6)
H36	−0.1230	1.1207	0.8457	0.032*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.01699 (13)	0.01926 (13)	0.02395 (14)	0.00605 (9)	0.00209 (9)	0.00448 (9)
C1	0.0199 (13)	0.0262 (14)	0.0310 (15)	0.0079 (11)	0.0041 (11)	0.0093 (12)
O1	0.0315 (12)	0.0337 (13)	0.0472 (14)	0.0153 (10)	0.0040 (10)	0.0153 (11)
C2	0.0296 (16)	0.0209 (14)	0.0385 (17)	0.0065 (12)	0.0082 (13)	0.0030 (12)
O2	0.0563 (18)	0.0376 (15)	0.0491 (16)	0.0070 (13)	0.0307 (14)	0.0031 (12)
C3	0.0203 (13)	0.0273 (14)	0.0282 (14)	0.0049 (11)	0.0016 (11)	0.0058 (11)
O3	0.0255 (12)	0.0391 (14)	0.0435 (14)	0.0008 (10)	−0.0041 (10)	0.0016 (11)

C4	0.0193 (13)	0.0258 (14)	0.0295 (15)	0.0056 (11)	−0.0013 (11)	0.0039 (11)
O4	0.0342 (13)	0.0491 (15)	0.0275 (12)	0.0129 (11)	0.0063 (9)	0.0069 (11)
P1	0.0200 (3)	0.0180 (3)	0.0222 (3)	0.0049 (3)	0.0013 (3)	0.0032 (3)
C5	0.0230 (13)	0.0165 (12)	0.0311 (15)	0.0042 (10)	0.0028 (11)	0.0039 (11)
C6	0.0343 (17)	0.0308 (17)	0.0325 (17)	−0.0022 (14)	−0.0014 (13)	0.0021 (13)
C7	0.039 (2)	0.0361 (19)	0.041 (2)	−0.0067 (16)	−0.0017 (15)	−0.0008 (15)
C8	0.0309 (17)	0.0238 (15)	0.054 (2)	0.0000 (13)	0.0072 (15)	0.0061 (15)
C9	0.0290 (16)	0.0278 (15)	0.0442 (19)	0.0089 (13)	0.0138 (14)	0.0121 (14)
C10	0.0268 (15)	0.0243 (14)	0.0346 (16)	0.0077 (12)	0.0033 (12)	0.0064 (12)
C11	0.0263 (14)	0.0227 (13)	0.0211 (13)	0.0101 (11)	0.0003 (10)	−0.0005 (10)
C12	0.0340 (17)	0.0263 (15)	0.0324 (16)	0.0135 (13)	0.0005 (13)	−0.0022 (12)
C13	0.048 (2)	0.0319 (17)	0.0355 (18)	0.0215 (16)	−0.0026 (15)	−0.0046 (14)
C14	0.0382 (19)	0.046 (2)	0.0367 (18)	0.0245 (17)	0.0052 (14)	−0.0017 (15)
C15	0.0322 (17)	0.044 (2)	0.0315 (17)	0.0158 (15)	0.0071 (13)	0.0049 (14)
C16	0.0265 (15)	0.0299 (15)	0.0289 (15)	0.0096 (12)	0.0030 (11)	0.0040 (12)
C17	0.0224 (13)	0.0199 (13)	0.0249 (13)	0.0042 (10)	−0.0006 (10)	0.0052 (10)
N1	0.0235 (12)	0.0159 (10)	0.0259 (12)	0.0054 (9)	−0.0018 (9)	0.0014 (9)
C18	0.0199 (13)	0.0186 (12)	0.0260 (13)	0.0060 (10)	0.0016 (10)	0.0071 (10)
C19	0.0308 (16)	0.0224 (14)	0.0304 (15)	0.0078 (12)	−0.0014 (12)	0.0031 (11)
C20	0.0307 (16)	0.0256 (15)	0.0387 (17)	0.0142 (13)	0.0018 (13)	0.0080 (13)
C21	0.0217 (14)	0.0341 (17)	0.0459 (19)	0.0052 (13)	−0.0012 (13)	0.0160 (15)
C22	0.0264 (15)	0.0322 (16)	0.0361 (17)	0.0045 (13)	−0.0073 (13)	0.0073 (13)
C23	0.0241 (14)	0.0256 (14)	0.0278 (14)	0.0054 (11)	−0.0023 (11)	0.0046 (11)
C24	0.0243 (13)	0.0195 (12)	0.0255 (13)	0.0089 (11)	0.0000 (10)	0.0032 (10)
P2	0.0188 (3)	0.0167 (3)	0.0234 (3)	0.0065 (3)	0.0023 (3)	0.0031 (3)
C25	0.0181 (12)	0.0182 (12)	0.0232 (13)	0.0029 (10)	0.0002 (9)	0.0009 (10)
C26	0.0229 (14)	0.0230 (14)	0.0318 (15)	0.0081 (11)	0.0022 (11)	0.0061 (11)
C27	0.0216 (14)	0.0322 (16)	0.0373 (17)	0.0112 (12)	0.0058 (12)	0.0034 (13)
C28	0.0236 (15)	0.0338 (17)	0.0332 (16)	0.0011 (12)	0.0057 (12)	0.0025 (13)
C29	0.0287 (16)	0.0376 (18)	0.0287 (16)	0.0030 (14)	0.0002 (12)	0.0061 (13)
C30	0.0226 (14)	0.0290 (15)	0.0304 (15)	0.0042 (12)	0.0013 (11)	0.0068 (12)
C31	0.0249 (14)	0.0186 (12)	0.0237 (13)	0.0083 (10)	0.0015 (10)	0.0036 (10)
C32	0.0243 (14)	0.0211 (13)	0.0318 (15)	0.0079 (11)	0.0002 (11)	0.0001 (11)
C33	0.0342 (16)	0.0177 (13)	0.0362 (17)	0.0062 (12)	0.0002 (13)	0.0030 (12)
C34	0.0431 (18)	0.0201 (13)	0.0255 (14)	0.0150 (13)	0.0024 (12)	0.0028 (11)
C35	0.0348 (17)	0.0286 (15)	0.0329 (16)	0.0175 (13)	0.0088 (13)	0.0063 (12)
C36	0.0272 (15)	0.0229 (14)	0.0309 (15)	0.0101 (12)	0.0036 (11)	0.0034 (11)

Geometric parameters (Å, º)

Mo1—C3	1.994 (3)	N1—C24	1.455 (4)
Mo1—C1	2.007 (3)	C18—C23	1.395 (4)
Mo1—C2	2.016 (3)	C18—C19	1.398 (4)
Mo1—C4	2.043 (3)	C19—C20	1.385 (4)
Mo1—P2	2.4986 (8)	C19—H19	0.9500
Mo1—P1	2.5005 (8)	C20—C21	1.377 (5)
C1—O1	1.145 (4)	C20—H20	0.9500
C2—O2	1.152 (4)	C21—C22	1.382 (5)

C3—O3	1.156 (4)	C21—H21	0.9500
C4—O4	1.151 (4)	C22—C23	1.400 (4)
P1—C11	1.823 (3)	C22—H22	0.9500
P1—C5	1.828 (3)	C23—H23	0.9500
P1—C17	1.865 (3)	C24—P2	1.860 (3)
C5—C10	1.393 (4)	C24—H24A	0.9900
C5—C6	1.403 (4)	C24—H24B	0.9900
C6—C7	1.392 (5)	P2—C25	1.832 (3)
C6—H6	0.9500	P2—C31	1.838 (3)
C7—C8	1.387 (5)	C25—C26	1.388 (4)
C7—H7	0.9500	C25—C30	1.394 (4)
C8—C9	1.378 (5)	C26—C27	1.395 (4)
C8—H8	0.9500	C26—H26	0.9500
C9—C10	1.401 (4)	C27—C28	1.387 (5)
C9—H9	0.9500	C27—H27	0.9500
C10—H10	0.9500	C28—C29	1.394 (5)
C11—C16	1.389 (4)	C28—H28	0.9500
C11—C12	1.413 (4)	C29—C30	1.396 (4)
C12—C13	1.383 (5)	C29—H29	0.9500
C12—H12	0.9500	C30—H30	0.9500
C13—C14	1.384 (6)	C31—C32	1.389 (4)
C13—H13	0.9500	C31—C36	1.391 (4)
C14—C15	1.392 (5)	C32—C33	1.386 (4)
C14—H14	0.9500	C32—H32	0.9500
C15—C16	1.387 (4)	C33—C34	1.386 (5)
C15—H15	0.9500	C33—H33	0.9500
C16—H16	0.9500	C34—C35	1.385 (5)
C17—N1	1.462 (4)	C34—H34	0.9500
C17—H17A	0.9900	C35—C36	1.397 (4)
C17—H17B	0.9900	C35—H35	0.9500
N1—C18	1.419 (4)	C36—H36	0.9500

C3—Mo1—C1	90.78 (13)	C18—N1—C17	117.0 (2)
C3—Mo1—C2	89.67 (13)	C24—N1—C17	115.5 (2)
C1—Mo1—C2	91.66 (13)	C23—C18—C19	118.4 (3)
C3—Mo1—C4	88.59 (12)	C23—C18—N1	122.4 (3)
C1—Mo1—C4	87.92 (12)	C19—C18—N1	119.1 (3)
C2—Mo1—C4	178.21 (12)	C20—C19—C18	120.9 (3)
C3—Mo1—P2	177.99 (9)	C20—C19—H19	119.5
C1—Mo1—P2	90.49 (9)	C18—C19—H19	119.5
C2—Mo1—P2	88.74 (10)	C21—C20—C19	120.7 (3)
C4—Mo1—P2	93.01 (8)	C21—C20—H20	119.6
C3—Mo1—P1	91.92 (9)	C19—C20—H20	119.6
C1—Mo1—P1	176.40 (9)	C20—C21—C22	119.1 (3)
C2—Mo1—P1	85.98 (9)	C20—C21—H21	120.4
C4—Mo1—P1	94.52 (9)	C22—C21—H21	120.4
P2—Mo1—P1	86.75 (2)	C21—C22—C23	121.0 (3)
O1—C1—Mo1	178.7 (3)	C21—C22—H22	119.5

O2—C2—Mo1	179.6 (3)
O3—C3—Mo1	178.1 (3)
O4—C4—Mo1	176.2 (3)
C11—P1—C5	103.40 (14)
C11—P1—C17	99.70 (13)
C5—P1—C17	101.38 (14)
C11—P1—Mo1	122.17 (10)
C5—P1—Mo1	114.87 (10)
C17—P1—Mo1	112.43 (9)
C10—C5—C6	119.2 (3)
C10—C5—P1	122.8 (2)
C6—C5—P1	117.8 (2)
C7—C6—C5	119.8 (3)
C7—C6—H6	120.1
C5—C6—H6	120.1
C8—C7—C6	120.7 (3)
C8—C7—H7	119.7
C6—C7—H7	119.7
C9—C8—C7	119.7 (3)
C9—C8—H8	120.2
C7—C8—H8	120.2
C8—C9—C10	120.5 (3)
C8—C9—H9	119.8
C10—C9—H9	119.8
C5—C10—C9	120.0 (3)
C5—C10—H10	120.0
C9—C10—H10	120.0
C16—C11—C12	118.8 (3)
C16—C11—P1	118.8 (2)
C12—C11—P1	121.8 (2)
C13—C12—C11	119.4 (3)
C13—C12—H12	120.3
C11—C12—H12	120.3
C12—C13—C14	121.6 (3)
C12—C13—H13	119.2
C14—C13—H13	119.2
C13—C14—C15	118.9 (3)
C13—C14—H14	120.5
C15—C14—H14	120.5
C16—C15—C14	120.2 (3)
C16—C15—H15	119.9
C14—C15—H15	119.9
C15—C16—C11	121.0 (3)
C15—C16—H16	119.5
C11—C16—H16	119.5
N1—C17—P1	113.2 (2)
N1—C17—H17A	108.9
P1—C17—H17A	108.9
C23—C22—H22	119.5
C18—C23—C22	119.8 (3)
C18—C23—H23	120.1
C22—C23—H23	120.1
N1—C24—P2	112.1 (2)
N1—C24—H24A	109.2
P2—C24—H24A	109.2
N1—C24—H24B	109.2
P2—C24—H24B	109.2
H24A—C24—H24B	107.9
C25—P2—C31	101.21 (13)
C25—P2—C24	103.34 (13)
C31—P2—C24	98.14 (13)
C25—P2—Mo1	117.00 (9)
C31—P2—Mo1	118.07 (10)
C24—P2—Mo1	116.15 (10)
C26—C25—C30	119.3 (3)
C26—C25—P2	124.5 (2)
C30—C25—P2	116.2 (2)
C25—C26—C27	120.2 (3)
C25—C26—H26	119.9
C27—C26—H26	119.9
C28—C27—C26	120.4 (3)
C28—C27—H27	119.8
C26—C27—H27	119.8
C27—C28—C29	119.8 (3)
C27—C28—H28	120.1
C29—C28—H28	120.1
C28—C29—C30	119.6 (3)
C28—C29—H29	120.2
C30—C29—H29	120.2
C25—C30—C29	120.6 (3)
C25—C30—H30	119.7
C29—C30—H30	119.7
C32—C31—C36	118.8 (3)
C32—C31—P2	120.9 (2)
C36—C31—P2	120.3 (2)
C33—C32—C31	120.7 (3)
C33—C32—H32	119.6
C31—C32—H32	119.6
C34—C33—C32	120.3 (3)
C34—C33—H33	119.9
C32—C33—H33	119.9
C35—C34—C33	119.7 (3)
C35—C34—H34	120.2
C33—C34—H34	120.2
C34—C35—C36	119.9 (3)
C34—C35—H35	120.0

N1—C17—H17B	108.9	C36—C35—H35	120.0
P1—C17—H17B	108.9	C31—C36—C35	120.6 (3)
H17A—C17—H17B	107.8	C31—C36—H36	119.7
C18—N1—C24	118.7 (2)	C35—C36—H36	119.7

C2—Mo1—P1—C11	−171.65 (15)	C19—C20—C21—C22	0.8 (5)
C4—Mo1—P1—C11	10.07 (14)	C20—C21—C22—C23	−0.9 (5)
P2—Mo1—P1—C11	−82.69 (12)	C19—C18—C23—C22	0.7 (5)
C3—Mo1—P1—C5	−27.57 (14)	N1—C18—C23—C22	−175.7 (3)
C2—Mo1—P1—C5	61.96 (15)	C21—C22—C23—C18	0.1 (5)
C4—Mo1—P1—C5	−116.31 (14)	C18—N1—C24—P2	133.9 (2)
P2—Mo1—P1—C5	150.92 (11)	C17—N1—C24—P2	−79.5 (3)
C3—Mo1—P1—C17	−142.82 (13)	N1—C24—P2—C25	−73.8 (2)
C2—Mo1—P1—C17	−53.29 (14)	N1—C24—P2—C31	−177.4 (2)
C4—Mo1—P1—C17	128.44 (13)	N1—C24—P2—Mo1	55.7 (2)
P2—Mo1—P1—C17	35.67 (10)	C1—Mo1—P2—C25	−94.18 (13)
C11—P1—C5—C10	121.7 (3)	C2—Mo1—P2—C25	174.17 (14)
C17—P1—C5—C10	18.7 (3)	C4—Mo1—P2—C25	−6.24 (14)
Mo1—P1—C5—C10	−102.7 (3)	P1—Mo1—P2—C25	88.13 (11)
C11—P1—C5—C6	−61.4 (3)	C1—Mo1—P2—C31	27.08 (14)
C17—P1—C5—C6	−164.4 (3)	C2—Mo1—P2—C31	−64.56 (14)
Mo1—P1—C5—C6	74.2 (3)	C4—Mo1—P2—C31	115.03 (14)
C10—C5—C6—C7	−2.4 (5)	P1—Mo1—P2—C31	−150.61 (11)
P1—C5—C6—C7	−179.4 (3)	C1—Mo1—P2—C24	143.22 (13)
C5—C6—C7—C8	1.3 (6)	C2—Mo1—P2—C24	51.58 (14)
C6—C7—C8—C9	−0.2 (6)	C4—Mo1—P2—C24	−128.83 (13)
C7—C8—C9—C10	0.0 (5)	P1—Mo1—P2—C24	−34.46 (10)
C6—C5—C10—C9	2.2 (5)	C31—P2—C25—C26	111.6 (3)
P1—C5—C10—C9	179.1 (2)	C24—P2—C25—C26	10.3 (3)
C8—C9—C10—C5	−1.0 (5)	Mo1—P2—C25—C26	−118.7 (2)
C5—P1—C11—C16	163.2 (2)	C31—P2—C25—C30	−69.5 (2)
C17—P1—C11—C16	−92.5 (3)	C24—P2—C25—C30	−170.7 (2)
Mo1—P1—C11—C16	31.9 (3)	Mo1—P2—C25—C30	60.3 (2)
C5—P1—C11—C12	−26.2 (3)	C30—C25—C26—C27	1.3 (4)
C17—P1—C11—C12	78.1 (3)	P2—C25—C26—C27	−179.8 (2)
Mo1—P1—C11—C12	−157.5 (2)	C25—C26—C27—C28	0.0 (5)
C16—C11—C12—C13	0.9 (5)	C26—C27—C28—C29	−1.0 (5)
P1—C11—C12—C13	−169.7 (3)	C27—C28—C29—C30	0.8 (5)
C11—C12—C13—C14	0.8 (5)	C26—C25—C30—C29	−1.5 (5)
C12—C13—C14—C15	−2.2 (6)	P2—C25—C30—C29	179.5 (2)
C13—C14—C15—C16	2.0 (5)	C28—C29—C30—C25	0.5 (5)
C14—C15—C16—C11	−0.4 (5)	C25—P2—C31—C32	−25.4 (3)
C12—C11—C16—C15	−1.1 (5)	C24—P2—C31—C32	80.1 (3)
P1—C11—C16—C15	169.8 (3)	Mo1—P2—C31—C32	−154.4 (2)
C11—P1—C17—N1	69.0 (2)	C25—P2—C31—C36	156.5 (2)
C5—P1—C17—N1	174.9 (2)	C24—P2—C31—C36	−98.0 (3)
Mo1—P1—C17—N1	−61.9 (2)	Mo1—P2—C31—C36	27.5 (3)
P1—C17—N1—C18	−128.0 (2)	C36—C31—C32—C33	−0.8 (5)

P1—C17—N1—C24	84.9 (3)	P2—C31—C32—C33	−179.0 (2)
C24—N1—C18—C23	22.5 (4)	C31—C32—C33—C34	0.4 (5)
C17—N1—C18—C23	−123.5 (3)	C32—C33—C34—C35	0.6 (5)
C24—N1—C18—C19	−153.9 (3)	C33—C34—C35—C36	−1.1 (5)
C17—N1—C18—C19	60.1 (4)	C32—C31—C36—C35	0.3 (5)
C23—C18—C19—C20	−0.8 (5)	P2—C31—C36—C35	178.4 (2)
N1—C18—C19—C20	175.7 (3)	C34—C35—C36—C31	0.7 (5)
C18—C19—C20—C21	0.1 (5)		