

Crystal structures of *trans*-acetyldicarbonyl-(η^5 -cyclopentadienyl)(dimethylphenylphosphane)-molybdenum(II) and *trans*-acetyldicarbonyl-(η^5 -cyclopentadienyl)(ethyldiphenylphosphane)-molybdenum(II)

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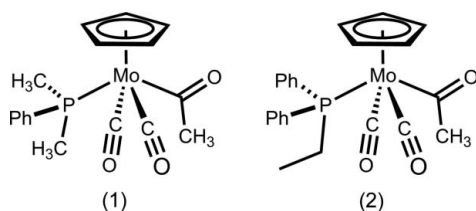
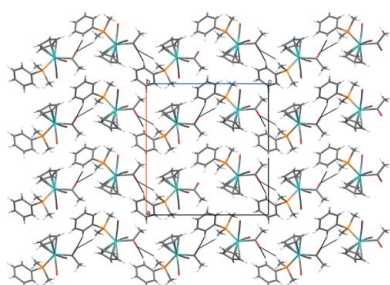
Keywords: crystal structure; phosphine; acetyl; piano-stool complex; divalent molybdenum

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The title compounds, $[\text{Mo}(\text{C}_5\text{H}_5)(\text{COCH}_3)\text{P}(\text{CH}_3)_2(\text{C}_6\text{H}_5)(\text{CO})_2]$, (1), and $[\text{Mo}(\text{C}_5\text{H}_5)(\text{COCH}_3)\text{P}(\text{C}_2\text{H}_5)(\text{C}_6\text{H}_5)_2(\text{CO})_2]$, (2), have been prepared by phosphine-induced migratory insertion from $[\text{Mo}(\text{C}_5\text{H}_5)(\text{CO})_3(\text{CH}_3)]$. Both complex molecules exhibit a four-legged piano-stool geometry with *trans*-disposed carbonyl ligands along with Mo–P bond lengths and C–Mo–P angles that reflect the relative steric pressure of the respective phosphine ligand. The structure of compound (1) exhibits a layered arrangement parallel to (100). Within the layers molecules are linked into chains along [001] by non-classical C–H···O interactions between the acetyl ligand of one molecule and the phenyl and methyl phosphine substituents of another. In the structure of complex (2), a chain motif of centrosymmetrical dimers is found along [010] through C–H···O interactions.

1. Chemical context

Cyclopentadienylmolybdenum polycarbonyl complexes $[\text{Mo}(\text{C}_5\text{H}_5)(\text{CO})_n]$ with ‘piano-stool’ geometries have been studied extensively for their fundamental organometallic reactivity. In particular, alkyl complexes of the form $[\text{Mo}(\text{C}_5\text{H}_5)(\text{CO})_3(\text{R})]$ have been studied for their migratory insertion reactivity (Barnett & Treichel, 1967; Butler *et al.*, 1967), affording $[\text{Mo}(\text{C}_5\text{H}_5)(\text{PR}_3)(\text{CO})_2(\text{COR})]$ acetyl complexes on exposure to phosphine ligands. Although the insertion reaction shows little dependence on the nature of the phosphine, the corresponding deinsertion shows a strong dependence on steric bulk of the phosphine, with bulkier groups giving enhanced deinsertion rates (Barnett, 1969; Barnett & Pollmann, 1974).



We have developed an interest in the solid-state structural properties of a series of piano-stool molybdenum acetyl complexes derived from migratory insertion with various phosphines, with the goal of understanding how modification

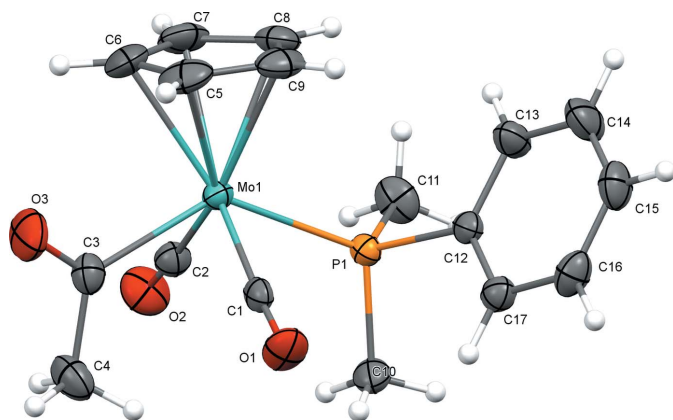


Figure 1
Molecular structure of (1) with displacement ellipsoids drawn at the 50% probability level.

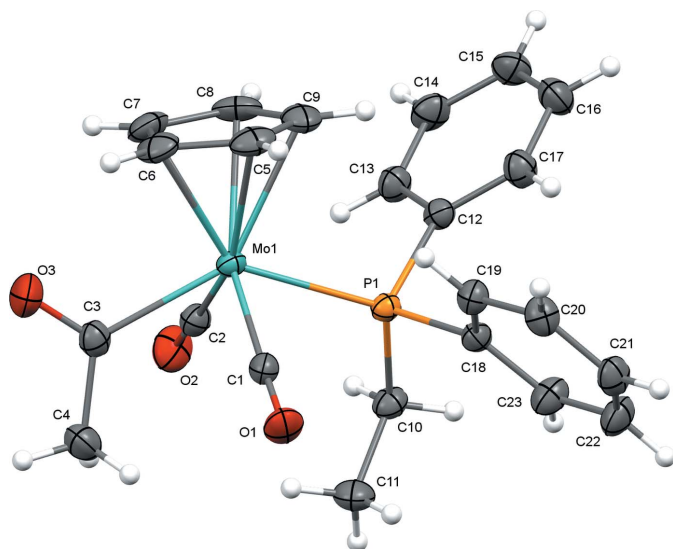


Figure 2
Molecular structure of (2) with displacement ellipsoids drawn at the 50% probability level.

of the phosphine substituents affects ground-state structure as well as solid-state packing. Recently, we reported an unusual example where orientation of the acetyl group in the solid state can be changed by introduction of furyl substituents on the phosphine ligand (Whited *et al.*, 2013). In this study, the structures obtained for dimethylphenylphosphine, $[\text{Mo}(\text{C}_5\text{H}_5)(\text{P}(\text{CH}_3)_2(\text{C}_6\text{H}_5))(\text{CO})_2(\text{COCH}_3)]$ (1), and ethyldiphenylphosphine, $[\text{Mo}(\text{C}_5\text{H}_5)(\text{P}(\text{C}_2\text{H}_5)(\text{C}_6\text{H}_5)_2)(\text{CO})_2(\text{COCH}_3)]$ (2), derivatives are compared.

2. Structural commentary

The molecular structures of (1) and (2) are illustrated in Figs. 1 and 2. In spite of the somewhat different steric environments provided by the phosphine ligands, the molecular structures are quite similar. Both complexes exhibit a *trans* disposition of carbonyl ligands common for compounds of this class. Complexes (1) and (2) both have structures where the oxygen

Table 1
Selected geometric parameters (\AA , $^\circ$) for (1).

Mo1—P1	2.4535 (9)	Mo1—C2	1.973 (4)
Mo1—C1	1.949 (3)	Mo1—C3	2.251 (4)
C1—Mo1—P1	76.95 (9)	C2—Mo1—P1	78.13 (11)
C1—Mo1—C2	106.40 (14)	C2—Mo1—C3	76.05 (15)
C1—Mo1—C3	72.37 (13)	C3—Mo1—P1	131.79 (9)

Table 2
Selected geometric parameters (\AA , $^\circ$) for (2).

Mo1—P1	2.4813 (6)	Mo1—C2	1.960 (2)
Mo1—C1	1.979 (2)	Mo1—C3	2.273 (2)
C1—Mo1—P1	79.07 (6)	C2—Mo1—C1	106.04 (9)
C1—Mo1—C3	75.46 (8)	C2—Mo1—C3	73.53 (9)
C2—Mo1—P1	79.67 (6)	C3—Mo1—P1	135.76 (6)

atom of the acetyl group points toward the cyclopentadienyl (Cp) ring. This orientation is also consistent with the majority of crystal structures of related complexes, with the exception of the recently reported tri(2-furyl)phosphine derivative, in which the acetyl group points away from the Cp ring, enabling intermolecular $\text{O} \cdots \text{H} - \text{C}$ interactions with the furyl group of a neighboring molecule (Whited *et al.*, 2013).

Selected geometric parameters for (1) and (2) are presented in Tables 1 and 2. The Mo1—P1 bond lengths [2.4535 (9) \AA for dimethylphenylphosphine derivative (1) and 2.4813 (6) \AA for ethyldiphenylphosphine derivative (2)] track with the steric bulk of the ligands and are consistent with the previously reported methyldiphenylphosphine complex (Whited *et al.*, 2012), which exhibits an Mo—P bond length [2.4620 (14) \AA] that is intermediate between those of (1) and (2). Along with a slightly longer Mo—P distance, the sterically bulkier derivative (2) exhibits a larger C3—Mo1—P1 angle [135.76 (6) $^\circ$] relative to (1) [131.79 (9) $^\circ$], again with the methyldiphenylphosphine derivative intermediate [132.27 (2) $^\circ$]. The steric effects of the phosphine ligands observed in the solid state are consistent with findings regarding decarbonylation rates for this class of complexes (Barnett & Pollmann, 1974), where the steric influence of bulkier phosphines enhances the rate of the decarbonylation reaction.

3. Supramolecular features

The extended structures of (1) and (2) are quite different, but the acetyl oxygen atom (O3) plays an important role in the packing of both structures. For dimethylphenylphosphine complex (1), there are $\text{C} - \text{H} \cdots \text{O}$ hydrogen-bonding interactions between O3 of the acetyl carbonyl on one Mo complex and H11C from a phosphine methyl substituent (2.45 \AA) and H13 from a phenyl group (2.36 \AA) on the same phosphine on a neighboring molecule (Table 3). These short contacts organize the molecules into chains parallel to [001] (Fig. 3). Additional short contacts (2.40 \AA) between O1 of a carbonyl ligand and H15 of a phosphine phenyl substituent within the chains are

Table 3
Hydrogen-bond geometry (Å, °) for (1).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C11—H11C...O3 ⁱ	0.98	2.45	3.344 (5)	152
C13—H13...O3 ⁱ	0.95	2.36	3.275 (5)	162

Symmetry code: (i) $-x + \frac{3}{2}, y - \frac{1}{2}, z - \frac{1}{2}$.

Table 4
Hydrogen-bond geometry (Å, °) for (2).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C8—H8...O2 ⁱ	0.93	2.63	3.414 (3)	142
C13—H13...O2 ⁱ	0.93	2.71	3.282 (3)	121
C22—H22...O3 ⁱⁱ	0.93	2.66	3.316 (3)	128

Symmetry codes: (i) $-x, -y, -z + 1$; (ii) $x, y + 1, z$.

present. The chains are arranged in layers parallel to (100). In contrast to the closely related methylphenylphosphine derivative (Whited *et al.*, 2012), (1) does not exhibit any π – π interactions between the Cp ring and a phosphine phenyl substituent. In contrast, the closest phenyl group is oriented perpendicular to the Cp ring with a distance of 3.00 Å between H17 of the phenyl group and the Cp centroid.

The supramolecular organization of ethyldiphenylphosphine derivative (2) is quite different, though it is still partly governed by hydrogen-bonding interactions involving O3 of the acetyl group. In this case, short contacts (2.66 Å) between O3 of the acetyl group and H22 of a phosphine phenyl substituent (Table 4) link the molecules into chains parallel to [010]. An additional set of short contacts between O2 of a carbonyl ligand and H8 from a Cp ring (2.63 Å) and H13 from a phenyl ring (2.71 Å) on an adjacent molecule organize the molecules into centrosymmetrical dimers, joining the unit cells along [010] (Fig. 4). Finally, another set of

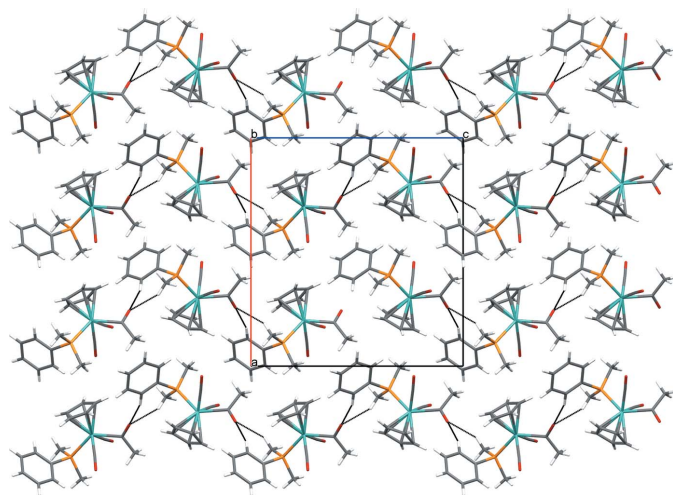


Figure 3
Crystal packing of (1) viewed along [010] showing the layered arrangement parallel to (100). Dashed lines indicate intermolecular C—H...O hydrogen-bonding interactions.

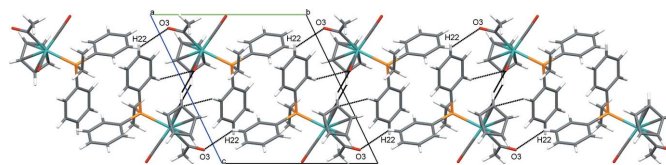


Figure 4
Crystal packing of (2) viewed along [100] showing chains of centrosymmetrical dimers.

centrosymmetrical dimers is formed through short contacts between C8/H8 units on Cp rings of adjacent molecules (Fig. 5).

4. Database survey

The current version of the Cambridge Structural Database (Version 5.35, updated November 2013; Allen, 2002) has nine entries corresponding to molybdenum acyl complexes of the general form $[\text{Mo}(\text{C}_5\text{H}_5)(\text{CO})_2(\text{PR}_3)(\text{COR})]$, as well as five tungsten complexes with the same ligand types. No chromium complexes with the same ligand set are in the database. The *trans*-dicarbonyl structure, as observed for (1) and (2), is preferred except in cases where the phosphine and acyl

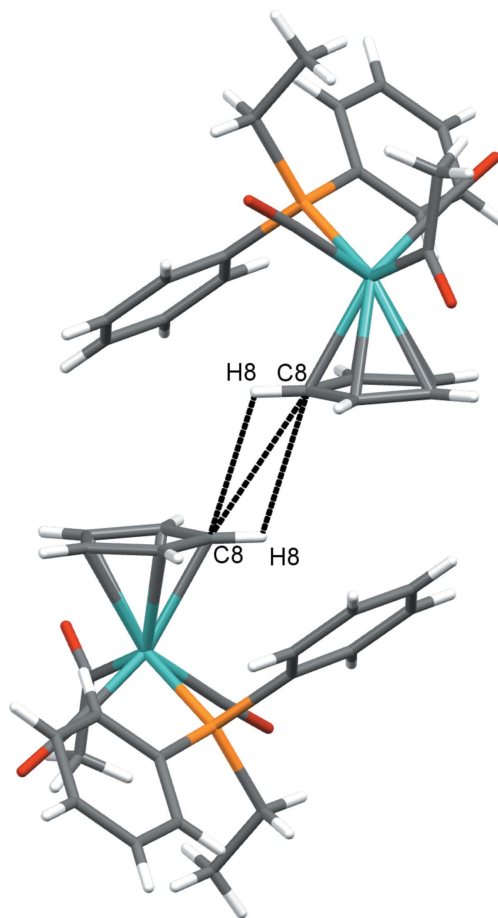


Figure 5
Centrosymmetrical dimers of (2) connected through C8/H8 interactions of Cp rings on adjacent molecules.

Table 5
 Experimental details.

	(1)	(2)
Crystal data		
Chemical formula	[Mo(C ₅ H ₅)(C ₂ H ₃ O)(C ₈ H ₁₁ P)(CO) ₂]	[Mo(C ₅ H ₅)(C ₂ H ₃ O)(C ₁₄ H ₁₅ P)(CO) ₂]
<i>M_r</i>	398.23	474.32
Crystal system, space group	Orthorhombic, <i>Pna</i> 2 ₁	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	173	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	16.374 (2), 6.8898 (10), 15.208 (2)	8.2451 (8), 11.6132 (11), 12.5265 (12)
α , β , γ (°)	90, 90, 90	63.617 (4), 77.167 (5), 84.671 (6)
<i>V</i> (Å ³)	1715.6 (4)	1047.65 (18)
<i>Z</i>	4	2
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.87	0.72
Crystal size (mm)	0.4 × 0.4 × 0.19	0.32 × 0.26 × 0.21
Data collection		
Diffractometer	Rigaku XtaLAB mini	Rigaku XtaLAB mini
Absorption correction	Multi-scan (<i>REQAB</i> ; Rigaku, 1998)	Multi-scan (<i>REQAB</i> ; Rigaku, 1998)
<i>T</i> _{min} , <i>T</i> _{max}	0.707, 0.848	0.712, 0.859
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	17021, 3923, 3639	11081, 4797, 4365
<i>R</i> _{int}	0.035	0.029
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.649	0.649
Refinement		
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> [<i>F</i> ²], <i>S</i>	0.021, 0.045, 1.05	0.028, 0.068, 1.09
No. of reflections	3923	4797
No. of parameters	202	255
No. of restraints	1	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.21, -0.27	0.30, -0.82
Absolute structure	Flack <i>x</i> determined using 1649 quotients [(<i>I</i> ⁺ - <i>I</i> ⁻)/(<i>I</i> ⁺ + <i>I</i> ⁻)] (Parsons <i>et al.</i> , 2013).	-
Absolute structure parameter	0.007 (18)	-

Computer programs: *CrystalClear* (Rigaku, 2011), *SHELXS* and *SHELXL* (Sheldrick, 2008), *SIR2008* (Burla *et al.*, 2007) and *OLEX2* (Dolomanov *et al.*, 2009).

ligands are covalently linked, forcing them to be *cis* (Adams *et al.*, 1991; Mercier *et al.*, 1993; Yan *et al.*, 2009). The preference for a *trans* geometry is likely at least partly steric in nature, since the only example with a *cis*-dicarbonyl geometry without linked phosphine and acyl ligands is for a molybdenum formyl with a small trimethylphosphine ligand and a bulky pentamethylcyclopentadienyl ligand (Asdar *et al.*, 1989).

5. Synthesis and crystallization

CpMo(CO)₃(CH₃). This compound was prepared by a modification of the method used of Gladysz *et al.* (1979), as previously reported by Whited & Hofmeister (2014).

CpMo(CO)₂(PMe₂Ph)(COCH₃) (1). In an inert-atmosphere glove box, CpMo(CO)₃(CH₃) (113 mg, 0.435 mmol) was dissolved in 2 ml acetonitrile. In a separate vial, dimethylphenylphosphine (97.0 mg, 0.702 mmol) was dissolved in 2 ml acetonitrile. The vials were combined and the resulting solution was stirred for 1 week. Solvent was removed *in vacuo*, leaving a yellow–orange solid that was triturated with pentane (5 ml) and isolated by filtration to afford the desired product in pure form as a yellow powder (112 mg, 65%). Crystalline material was obtained as yellow–orange prisms by chilling a concentrated diethyl ether solution at 233 K. ¹H NMR (400 MHz, CDCl₃): δ 7.67–7.58 (*m*, 2H, Ar–H), 7.50–7.41 (*m*,

3H, Ar–H), 4.97 (*d*, *J* = 1.1 Hz, 5H, Cp H), 2.58 (*s*, 3H, C(O)CH₃), 1.91 (*d*, ²*J*_{PH} = 8.9 Hz, 6H, P(CH₃)₂). ¹³C{¹H} NMR (101 MHz, CDCl₃): δ 267.3 (*d*, ²*J*_{PC} = 13 Hz, -COCH₃), 237.8 (*d*, ²*J*_{PC} = 24 Hz, -CO), 139.2 (*d*, ¹*J*_{PC} = 40 Hz, C_{ipso} from *Ph*-P), 130.3 (*d*, ⁴*J*_{PC} = 2 Hz, C_{para} from *Ph*-P), 129.6 (*d*, ²*J*_{PC} = 10 Hz, C_{ortho} from *Ph*-P), 128.9 (*d*, ²*J*_{PC} = 10 Hz, C_{meta} from *Ph*-P), 96.0 (*s*, Cp ring), 51.7 (*s*, -COCH₃), 20.0 (*d*, ¹*J*_{PC} = 33 Hz, P(CH₃)₂). ³¹P{¹H} NMR (162 MHz, CDCl₃): δ 33.1 (*s*). IR (CH₂Cl₂, NaCl, cm⁻¹) ν (CO): 1931, 1846, 1601 (acetyl).

CpMo(CO)₂(PEtPh₂)(COCH₃) (2). In an inert-atmosphere glove box, CpMo(CO)₃(CH₃) (105 mg, 0.404 mmol) was dissolved in 2 ml acetonitrile. Ethyldiphenylphosphine (129 mg, 0.602 mmol) was added and the resulting solution was stirred for one week. Solvent was removed *in vacuo*, leaving a yellow solid that was triturated with pentane (5 ml) and isolated by filtration to afford the desired product in pure form as a yellow powder (106 mg, 55%). Crystalline material was obtained as yellow blocks by slow evaporation of diethyl ether from a concentrated solution at ambient temperature. ¹H NMR (400 MHz, CDCl₃): δ 7.50–7.42 (*m*, 10H, Ar–H), 4.92 (*d*, *J* = 1.2 Hz, 5H, Cp H), 2.68 (apparent quint, ²*J*_{PH} = ³*J*_{HH} = 7.8 Hz, 2H, PCH₂CH₃), 2.63 (*s*, 3H, C(O)CH₃), 1.17 (*dt*, ³*J*_{PH} = 18.0 Hz, ²*J*_{PH} = 7.5 Hz, 3H, PCH₂CH₃). ¹³C{¹H} NMR (101 MHz, CDCl₃): δ 266.4 (*d*, ²*J*_{PC} = 11 Hz, -COCH₃), 238.5 (*d*, ²*J*_{PC} = 23 Hz, -CO), 135.9 (*d*, ¹*J*_{PC} = 40 Hz, C_{ipso} from *Ph*-

P), 132.1 (*d*, $^2J_{\text{PC}} = 10$ Hz, C_{ortho} from *Ph-P*), 130.4 (*d*, $^4J_{\text{PC}} = 2$ Hz, C_{para} from *Ph-P*), 128.7 (*d*, $^2J_{\text{PC}} = 9$ Hz, C_{meta} from *Ph-P*), 96.5 (*s*, Cp ring), 51.0 (*s*, $-\text{COCH}_3$), 26.3 (*d*, $^1J_{\text{PC}} = 32$ Hz, PCH_2CH_3), 9.0 (*d*, $^2J_{\text{PC}} = 2$ Hz, PCH_2CH_3). $^{31}\text{P}\{^1\text{H}\}$ NMR (162 MHz, CDCl_3): δ 59.3 (*s*). IR (CH_2Cl_2 , NaCl, cm^{-1}) $\nu(\text{CO})$: 1937, 1859, 1610 (acetyl).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. H-atoms were treated in calculated positions and refined in the riding-model approximation with distances of C–H = 0.95, 1.00 and 0.98 Å for the phenyl, cyclopentadienyl and alkyl groups, respectively, and with $U_{\text{iso}}(\text{H}) = k \times U_{\text{eq}}(\text{C})$, $k = 1.2$ for phenyl and cyclopentadienyl groups and 1.5 for alkyl groups. Methyl group H atoms were allowed to rotate in order to find the best rotameric conformation.

A small number of low-angle reflections [three for (1); six for (2)] were rejected from these high-quality data sets due to the arrangement of the instrument with a conservatively sized beam stop and a fixed-position detector. The large number of reflections in the data sets (and the Fourier-transform relationship of intensities to atoms) ensures that no particular bias was thereby introduced.

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supporting information

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Crystal structures of *trans*-acetyldicarbonyl(η^5 -cyclopentadienyl)(dimethylphenylphosphane)molybdenum(II) and *trans*-acetyldicarbonyl(η^5 -cyclopentadienyl)(ethyldiphenylphosphane)molybdenum(II)

Matthew T. Whited, Gretchen E. Hofmeister, Connor J. Hodges, Laramie T. Jensen, Samuel H. Keyes, Aurapat Ngamnithiporn and Daron E. Janzen

Computing details

For both compounds, data collection: *CrystalClear* (Rigaku, 2011); cell refinement: *CrystalClear* (Rigaku, 2011); data reduction: *CrystalClear* (Rigaku, 2011). Program(s) used to solve structure: *SHELXS* (Sheldrick, 2008) for (1); *SIR2008* (Burla *et al.*, 2007) for (2). For both compounds, program(s) used to refine structure: *SHELXL* (Sheldrick, 2008); molecular graphics: Olex2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: Olex2 (Dolomanov *et al.*, 2009).

(1) *trans*-Acetyldicarbonyl-(η^5 -cyclopentadienyl)-(dimethylphenylphosphine)-molybdenum(II)

Crystal data

[Mo(C₅H₅)(C₂H₃O)(C₈H₁₁P)(CO)₂]
 $M_r = 398.23$
 Orthorhombic, *Pna*2₁
 Hall symbol: P 2c -2n
 $a = 16.374$ (2) Å
 $b = 6.8898$ (10) Å
 $c = 15.208$ (2) Å
 $V = 1715.6$ (4) Å³
 $Z = 4$

$F(000) = 808$
 $D_x = 1.542$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
 Cell parameters from 16109 reflections
 $\theta = 3.2$ – 27.6°
 $\mu = 0.87$ mm⁻¹
 $T = 173$ K
 Prism, yellow
 $0.4 \times 0.4 \times 0.19$ mm

Data collection

Rigaku XtaLAB mini
 diffractometer
 Detector resolution: 6.849 pixels mm⁻¹
 ω scans
 Absorption correction: multi-scan
 (*REQAB*; Rigaku, 1998)
 $T_{\min} = 0.707$, $T_{\max} = 0.848$
 17021 measured reflections

3923 independent reflections
 3639 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$
 $\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.2^\circ$
 $h = -21 \rightarrow 21$
 $k = -8 \rightarrow 8$
 $l = -19 \rightarrow 19$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.021$
 $wR(F^2) = 0.045$
 $S = 1.05$

3923 reflections
 202 parameters
 1 restraint
 Primary atom site location: heavy-atom method

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0142P)^2 + 0.493P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.002$$

$$\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$$

Absolute structure: Flack x determined using 1649 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013).

Absolute structure parameter: 0.007 (18)

Special details

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Mo1	0.79993 (2)	0.60128 (3)	0.24997 (2)	0.02038 (7)
P1	0.89764 (5)	0.44416 (12)	0.15052 (5)	0.02144 (17)
O1	0.95421 (15)	0.8609 (4)	0.26796 (19)	0.0403 (8)
O2	0.82922 (19)	0.2063 (4)	0.34585 (19)	0.0449 (7)
O3	0.7578 (2)	0.7896 (5)	0.4257 (2)	0.0533 (8)
C1	0.89797 (19)	0.7587 (4)	0.2637 (2)	0.0238 (7)
C2	0.8215 (2)	0.3542 (5)	0.3113 (2)	0.0278 (8)
C3	0.8089 (2)	0.6869 (5)	0.3925 (2)	0.0300 (8)
C4	0.8795 (3)	0.6224 (6)	0.4511 (3)	0.0439 (11)
H4A	0.8657	0.4984	0.4791	0.066*
H4B	0.8891	0.7206	0.4966	0.066*
H4C	0.9289	0.6067	0.4155	0.066*
C5	0.7040 (2)	0.8435 (6)	0.2268 (3)	0.0432 (13)
H5	0.7125	0.9674	0.2525	0.052*
C6	0.6620 (2)	0.6856 (7)	0.2659 (3)	0.0465 (11)
H6	0.6375	0.6850	0.3225	0.056*
C7	0.6631 (2)	0.5310 (7)	0.2065 (3)	0.0425 (10)
H7	0.6393	0.4069	0.2156	0.051*
C8	0.7056 (2)	0.5909 (6)	0.1309 (3)	0.0368 (9)
H8	0.7156	0.5137	0.0802	0.044*
C9	0.7308 (2)	0.7839 (6)	0.1427 (3)	0.0376 (9)
H9	0.7604	0.8604	0.1017	0.045*
C10	0.9940 (2)	0.3716 (6)	0.2006 (2)	0.0331 (8)
H10A	1.0193	0.4841	0.2292	0.050*
H10B	1.0308	0.3215	0.1551	0.050*
H10C	0.9839	0.2702	0.2444	0.050*
C11	0.8649 (3)	0.2188 (5)	0.0985 (3)	0.0373 (9)
H11A	0.8542	0.1210	0.1439	0.056*
H11B	0.9080	0.1720	0.0591	0.056*
H11C	0.8150	0.2421	0.0646	0.056*
C12	0.9282 (2)	0.5990 (5)	0.0594 (2)	0.0237 (7)
C13	0.8797 (2)	0.6100 (6)	-0.0161 (2)	0.0337 (9)
H13	0.8334	0.5280	-0.0220	0.040*

C14	0.8991 (3)	0.7407 (6)	-0.0823 (3)	0.0452 (10)
H14	0.8653	0.7487	-0.1329	0.054*
C15	0.9663 (3)	0.8582 (6)	-0.0759 (2)	0.0386 (10)
H15	0.9791	0.9467	-0.1218	0.046*
C16	1.0155 (2)	0.8471 (5)	-0.0018 (3)	0.0347 (9)
H16	1.0628	0.9266	0.0027	0.042*
C17	0.9959 (2)	0.7194 (5)	0.0662 (2)	0.0274 (7)
H17	1.0290	0.7148	0.1175	0.033*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.01676 (11)	0.02175 (12)	0.02264 (12)	-0.00044 (9)	-0.00022 (16)	0.00141 (16)
P1	0.0228 (4)	0.0208 (4)	0.0207 (4)	0.0008 (3)	-0.0010 (4)	-0.0001 (3)
O1	0.0323 (13)	0.0419 (15)	0.047 (2)	-0.0136 (11)	0.0007 (12)	-0.0156 (13)
O2	0.0580 (18)	0.0296 (15)	0.0471 (18)	0.0000 (13)	-0.0037 (14)	0.0108 (14)
O3	0.056 (2)	0.065 (2)	0.0386 (16)	0.0241 (17)	0.0116 (14)	-0.0095 (16)
C1	0.0267 (15)	0.0265 (15)	0.0183 (19)	0.0055 (12)	0.0014 (14)	-0.0051 (13)
C2	0.0254 (18)	0.029 (2)	0.0285 (18)	-0.0025 (14)	-0.0001 (14)	-0.0009 (15)
C3	0.035 (2)	0.0295 (19)	0.0259 (18)	0.0013 (16)	0.0048 (15)	0.0024 (15)
C4	0.058 (3)	0.046 (3)	0.027 (2)	0.008 (2)	-0.006 (2)	-0.0063 (18)
C5	0.0278 (19)	0.039 (2)	0.063 (4)	0.0139 (16)	-0.0087 (18)	-0.0031 (18)
C6	0.0172 (16)	0.077 (3)	0.046 (3)	0.0088 (18)	-0.0001 (18)	-0.002 (2)
C7	0.020 (2)	0.053 (3)	0.054 (3)	-0.0112 (18)	-0.0108 (17)	0.013 (2)
C8	0.026 (2)	0.048 (2)	0.036 (2)	0.0003 (16)	-0.0141 (16)	0.0018 (17)
C9	0.0264 (19)	0.039 (2)	0.048 (2)	0.0015 (16)	-0.0110 (18)	0.0168 (19)
C10	0.028 (2)	0.040 (2)	0.031 (2)	0.0105 (16)	0.0008 (16)	0.0041 (15)
C11	0.052 (3)	0.0237 (19)	0.036 (2)	-0.0019 (17)	-0.0030 (18)	-0.0057 (17)
C12	0.0227 (17)	0.0273 (18)	0.0210 (16)	0.0045 (14)	0.0005 (14)	-0.0029 (14)
C13	0.032 (2)	0.043 (2)	0.026 (2)	-0.0112 (16)	-0.0042 (15)	0.0037 (16)
C14	0.050 (3)	0.062 (3)	0.025 (2)	-0.009 (2)	-0.0047 (18)	0.0115 (18)
C15	0.046 (2)	0.039 (2)	0.030 (2)	-0.0019 (18)	0.0108 (17)	0.0091 (17)
C16	0.032 (2)	0.031 (2)	0.042 (2)	-0.0065 (16)	0.0104 (17)	-0.0019 (17)
C17	0.0254 (18)	0.0292 (19)	0.0277 (18)	-0.0006 (15)	0.0010 (14)	-0.0034 (15)

Geometric parameters (Å, °)

Mo1—P1	2.4535 (9)	C6—C7	1.397 (6)
Mo1—C1	1.949 (3)	C7—H7	0.9500
Mo1—C2	1.973 (4)	C7—C8	1.406 (6)
Mo1—C3	2.251 (4)	C8—H8	0.9500
Mo1—C5	2.319 (4)	C8—C9	1.403 (5)
Mo1—C6	2.344 (3)	C9—H9	0.9500
Mo1—C7	2.385 (4)	C10—H10A	0.9800
Mo1—C8	2.382 (4)	C10—H10B	0.9800
Mo1—C9	2.351 (3)	C10—H10C	0.9800
P1—C10	1.822 (4)	C11—H11A	0.9800
P1—C11	1.823 (4)	C11—H11B	0.9800

P1—C12	1.819 (4)	C11—H11C	0.9800
O1—C1	1.161 (4)	C12—C13	1.397 (5)
O2—C2	1.153 (4)	C12—C17	1.388 (5)
O3—C3	1.208 (4)	C13—H13	0.9500
C3—C4	1.525 (5)	C13—C14	1.388 (5)
C4—H4A	0.9800	C14—H14	0.9500
C4—H4B	0.9800	C14—C15	1.370 (6)
C4—H4C	0.9800	C15—H15	0.9500
C5—H5	0.9500	C15—C16	1.387 (6)
C5—C6	1.417 (6)	C16—H16	0.9500
C5—C9	1.413 (6)	C16—C17	1.395 (5)
C6—H6	0.9500	C17—H17	0.9500
C1—Mo1—P1	76.95 (9)	C9—C5—H5	126.0
C1—Mo1—C2	106.40 (14)	C9—C5—C6	107.9 (4)
C1—Mo1—C3	72.37 (13)	Mo1—C6—H6	120.0
C1—Mo1—C5	100.01 (14)	C5—C6—Mo1	71.4 (2)
C1—Mo1—C6	130.12 (14)	C5—C6—H6	126.0
C1—Mo1—C7	156.39 (14)	C7—C6—Mo1	74.5 (2)
C1—Mo1—C8	129.21 (14)	C7—C6—C5	107.9 (4)
C1—Mo1—C9	99.96 (13)	C7—C6—H6	126.0
C2—Mo1—P1	78.13 (11)	Mo1—C7—H7	121.9
C2—Mo1—C3	76.05 (15)	C6—C7—Mo1	71.2 (2)
C2—Mo1—C5	144.49 (15)	C6—C7—H7	125.9
C2—Mo1—C6	109.72 (15)	C6—C7—C8	108.1 (4)
C2—Mo1—C7	97.13 (15)	C8—C7—Mo1	72.7 (2)
C2—Mo1—C8	116.74 (14)	C8—C7—H7	125.9
C2—Mo1—C9	151.18 (14)	Mo1—C8—H8	121.4
C3—Mo1—P1	131.79 (9)	C7—C8—Mo1	73.0 (2)
C3—Mo1—C5	90.13 (14)	C7—C8—H8	125.7
C3—Mo1—C6	84.19 (15)	C9—C8—Mo1	71.5 (2)
C3—Mo1—C7	112.45 (14)	C9—C8—C7	108.6 (4)
C3—Mo1—C8	141.51 (14)	C9—C8—H8	125.7
C3—Mo1—C9	124.02 (14)	Mo1—C9—H9	120.4
C5—Mo1—P1	131.72 (11)	C5—C9—Mo1	71.2 (2)
C5—Mo1—C6	35.38 (15)	C5—C9—H9	126.3
C5—Mo1—C7	57.84 (15)	C8—C9—Mo1	74.0 (2)
C5—Mo1—C8	57.75 (14)	C8—C9—C5	107.5 (4)
C5—Mo1—C9	35.21 (14)	C8—C9—H9	126.3
C6—Mo1—P1	143.25 (12)	P1—C10—H10A	109.5
C6—Mo1—C7	34.36 (15)	P1—C10—H10B	109.5
C6—Mo1—C8	57.40 (15)	P1—C10—H10C	109.5
C6—Mo1—C9	58.34 (15)	H10A—C10—H10B	109.5
C7—Mo1—P1	110.60 (12)	H10A—C10—H10C	109.5
C8—Mo1—P1	86.61 (11)	H10B—C10—H10C	109.5
C8—Mo1—C7	34.30 (14)	P1—C11—H11A	109.5
C9—Mo1—P1	97.03 (11)	P1—C11—H11B	109.5
C9—Mo1—C7	57.58 (14)	P1—C11—H11C	109.5

C9—Mo1—C8	34.49 (13)	H11A—C11—H11B	109.5
C10—P1—Mo1	115.36 (13)	H11A—C11—H11C	109.5
C10—P1—C11	101.66 (18)	H11B—C11—H11C	109.5
C11—P1—Mo1	116.85 (14)	C13—C12—P1	120.1 (3)
C12—P1—Mo1	112.96 (11)	C17—C12—P1	120.8 (3)
C12—P1—C10	103.97 (17)	C17—C12—C13	118.9 (3)
C12—P1—C11	104.47 (17)	C12—C13—H13	120.0
O1—C1—Mo1	175.6 (3)	C14—C13—C12	120.1 (3)
O2—C2—Mo1	175.7 (3)	C14—C13—H13	120.0
O3—C3—Mo1	120.7 (3)	C13—C14—H14	119.5
O3—C3—C4	116.8 (3)	C15—C14—C13	121.0 (4)
C4—C3—Mo1	122.4 (3)	C15—C14—H14	119.5
C3—C4—H4A	109.5	C14—C15—H15	120.3
C3—C4—H4B	109.5	C14—C15—C16	119.5 (4)
C3—C4—H4C	109.5	C16—C15—H15	120.3
H4A—C4—H4B	109.5	C15—C16—H16	119.9
H4A—C4—H4C	109.5	C15—C16—C17	120.2 (3)
H4B—C4—H4C	109.5	C17—C16—H16	119.9
Mo1—C5—H5	119.0	C12—C17—C16	120.3 (3)
C6—C5—Mo1	73.3 (2)	C12—C17—H17	119.8
C6—C5—H5	126.0	C16—C17—H17	119.8
C9—C5—Mo1	73.6 (2)		
Mo1—P1—C12—C13	−84.0 (3)	C7—C8—C9—Mo1	64.1 (3)
Mo1—P1—C12—C17	90.8 (3)	C7—C8—C9—C5	0.3 (4)
Mo1—C5—C6—C7	−66.0 (3)	C9—C5—C6—Mo1	66.0 (2)
Mo1—C5—C9—C8	65.6 (2)	C9—C5—C6—C7	0.0 (4)
Mo1—C6—C7—C8	−63.8 (3)	C10—P1—C12—C13	150.3 (3)
Mo1—C7—C8—C9	−63.1 (2)	C10—P1—C12—C17	−35.0 (3)
Mo1—C8—C9—C5	−63.7 (2)	C11—P1—C12—C13	44.0 (3)
P1—C12—C13—C14	174.4 (3)	C11—P1—C12—C17	−141.2 (3)
P1—C12—C17—C16	−175.7 (3)	C12—C13—C14—C15	1.1 (6)
C5—C6—C7—Mo1	64.0 (2)	C13—C12—C17—C16	−0.8 (5)
C5—C6—C7—C8	0.2 (4)	C13—C14—C15—C16	−0.3 (6)
C6—C5—C9—Mo1	−65.8 (3)	C14—C15—C16—C17	−1.1 (6)
C6—C5—C9—C8	−0.2 (4)	C15—C16—C17—C12	1.6 (5)
C6—C7—C8—Mo1	62.8 (3)	C17—C12—C13—C14	−0.5 (6)
C6—C7—C8—C9	−0.3 (4)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C11—H11C...O3 ⁱ	0.98	2.45	3.344 (5)	152
C13—H13...O3 ⁱ	0.95	2.36	3.275 (5)	162

Symmetry code: (i) $-x+3/2, y-1/2, z-1/2$.

(2) *trans*-Acetyldicarbonyl(η^5 -cyclopentadienyl)(ethyldiphenylphosphane)molybdenum(II)

Crystal data

[Mo(C₅H₅)(C₂H₃O)(C₁₄H₁₅P)(CO)₂]
 $M_r = 474.32$
 Triclinic, $P\bar{1}$
 $a = 8.2451$ (8) Å
 $b = 11.6132$ (11) Å
 $c = 12.5265$ (12) Å
 $\alpha = 63.617$ (4)°
 $\beta = 77.167$ (5)°
 $\gamma = 84.671$ (6)°
 $V = 1047.65$ (18) Å³

$Z = 2$
 $F(000) = 484$
 $D_x = 1.504$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 10268 reflections
 $\theta = 3.2$ – 27.6 °
 $\mu = 0.72$ mm⁻¹
 $T = 173$ K
 Prism, yellow
 $0.32 \times 0.26 \times 0.21$ mm

Data collection

Rigaku XtaLAB mini
 diffractometer
 Detector resolution: 6.849 pixels mm⁻¹
 ω scans
 Absorption correction: multi-scan
 (REQAB; Rigaku, 1998)
 $T_{\min} = 0.712$, $T_{\max} = 0.859$
 11081 measured reflections

4797 independent reflections
 4365 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$
 $\theta_{\max} = 27.5$ °, $\theta_{\min} = 3.2$ °
 $h = -10 \rightarrow 10$
 $k = -15 \rightarrow 15$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.068$
 $S = 1.09$
 4797 reflections
 255 parameters
 0 restraints
 Primary atom site location: heavy-atom method

Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.029P)^2 + 0.4738P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.30$ e Å⁻³
 $\Delta\rho_{\min} = -0.82$ e Å⁻³

Special details

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Mo1	0.25391 (2)	0.22164 (2)	0.24017 (2)	0.01783 (6)
P1	0.15696 (6)	0.36677 (5)	0.33857 (5)	0.01874 (11)
O1	0.2231 (2)	0.48000 (14)	0.01116 (14)	0.0328 (4)
O2	-0.1013 (2)	0.10837 (16)	0.37595 (17)	0.0379 (4)
O3	0.2283 (2)	0.07548 (18)	0.09140 (19)	0.0459 (5)
C1	0.2279 (3)	0.3845 (2)	0.0971 (2)	0.0225 (4)
C2	0.0285 (3)	0.1541 (2)	0.3233 (2)	0.0248 (4)
C3	0.1579 (3)	0.1604 (2)	0.1164 (2)	0.0256 (5)
C4	0.0061 (3)	0.2187 (2)	0.0626 (2)	0.0361 (6)

H4A	-0.0914	0.1955	0.1265	0.054*
H4B	-0.0043	0.1869	0.0055	0.054*
H4C	0.0177	0.3106	0.0217	0.054*
C5	0.5510 (3)	0.2184 (2)	0.2032 (2)	0.0313 (5)
H5	0.6164	0.2873	0.1426	0.038*
C6	0.4935 (3)	0.1161 (2)	0.1898 (2)	0.0319 (5)
H6	0.5130	0.1063	0.1183	0.038*
C7	0.4005 (3)	0.0304 (2)	0.3044 (2)	0.0331 (5)
H7	0.3493	-0.0456	0.3213	0.040*
C8	0.4000 (3)	0.0813 (2)	0.3878 (2)	0.0321 (5)
H8	0.3485	0.0450	0.4696	0.039*
C9	0.4922 (3)	0.1978 (2)	0.3243 (2)	0.0309 (5)
H9	0.5107	0.2517	0.3575	0.037*
C10	-0.0688 (3)	0.3899 (2)	0.3745 (2)	0.0247 (4)
H10A	-0.1210	0.3075	0.4308	0.030*
H10B	-0.0899	0.4456	0.4151	0.030*
C11	-0.1500 (3)	0.4481 (2)	0.2632 (2)	0.0347 (5)
H11A	-0.1027	0.5316	0.2084	0.052*
H11B	-0.2675	0.4560	0.2887	0.052*
H11C	-0.1307	0.3934	0.2226	0.052*
C12	0.2069 (3)	0.30616 (19)	0.49000 (19)	0.0219 (4)
C13	0.1367 (3)	0.1892 (2)	0.5807 (2)	0.0273 (5)
H13	0.0644	0.1458	0.5636	0.033*
C14	0.1734 (3)	0.1371 (2)	0.6958 (2)	0.0317 (5)
H14	0.1254	0.0593	0.7554	0.038*
C15	0.2814 (3)	0.2006 (2)	0.7222 (2)	0.0338 (5)
H15	0.3055	0.1660	0.7996	0.041*
C16	0.3534 (3)	0.3154 (2)	0.6333 (2)	0.0347 (5)
H16	0.4268	0.3577	0.6508	0.042*
C17	0.3166 (3)	0.3686 (2)	0.5174 (2)	0.0284 (5)
H17	0.3656	0.4461	0.4580	0.034*
C18	0.2429 (3)	0.53126 (19)	0.26053 (19)	0.0216 (4)
C19	0.4009 (3)	0.5528 (2)	0.18706 (19)	0.0245 (4)
H19	0.4597	0.4851	0.1765	0.029*
C20	0.4719 (3)	0.6756 (2)	0.1291 (2)	0.0272 (5)
H20	0.5786	0.6889	0.0813	0.033*
C21	0.3841 (3)	0.7775 (2)	0.1424 (2)	0.0294 (5)
H21	0.4309	0.8595	0.1028	0.035*
C22	0.2268 (3)	0.7569 (2)	0.2148 (2)	0.0334 (5)
H22	0.1674	0.8253	0.2235	0.040*
C23	0.1566 (3)	0.6340 (2)	0.2749 (2)	0.0306 (5)
H23	0.0514	0.6205	0.3248	0.037*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.01765 (10)	0.01435 (9)	0.01900 (10)	0.00154 (6)	-0.00451 (7)	-0.00496 (7)
P1	0.0185 (2)	0.0163 (2)	0.0196 (3)	0.00010 (19)	-0.0039 (2)	-0.0062 (2)

O1	0.0459 (10)	0.0201 (8)	0.0262 (8)	0.0016 (7)	-0.0129 (8)	-0.0023 (7)
O2	0.0284 (9)	0.0325 (9)	0.0484 (11)	-0.0103 (7)	0.0027 (8)	-0.0168 (8)
O3	0.0520 (12)	0.0396 (10)	0.0655 (13)	0.0108 (9)	-0.0235 (10)	-0.0368 (10)
C1	0.0230 (10)	0.0208 (10)	0.0270 (11)	0.0015 (8)	-0.0070 (9)	-0.0126 (9)
C2	0.0264 (11)	0.0180 (10)	0.0297 (11)	0.0006 (8)	-0.0084 (9)	-0.0089 (9)
C3	0.0294 (11)	0.0214 (11)	0.0252 (11)	-0.0043 (9)	-0.0052 (9)	-0.0087 (9)
C4	0.0410 (14)	0.0355 (13)	0.0378 (14)	0.0009 (11)	-0.0194 (12)	-0.0161 (11)
C5	0.0166 (10)	0.0304 (12)	0.0373 (13)	0.0045 (9)	-0.0036 (9)	-0.0077 (10)
C6	0.0258 (12)	0.0348 (13)	0.0348 (13)	0.0148 (10)	-0.0068 (10)	-0.0173 (11)
C7	0.0329 (13)	0.0183 (11)	0.0421 (14)	0.0091 (9)	-0.0128 (11)	-0.0074 (10)
C8	0.0244 (11)	0.0354 (13)	0.0247 (11)	0.0100 (10)	-0.0098 (9)	-0.0022 (10)
C9	0.0190 (11)	0.0379 (13)	0.0396 (13)	0.0083 (9)	-0.0118 (10)	-0.0191 (11)
C10	0.0203 (10)	0.0230 (11)	0.0284 (11)	0.0009 (8)	-0.0025 (9)	-0.0105 (9)
C11	0.0265 (12)	0.0369 (13)	0.0442 (15)	0.0082 (10)	-0.0157 (11)	-0.0184 (12)
C12	0.0228 (10)	0.0208 (10)	0.0213 (10)	0.0024 (8)	-0.0051 (8)	-0.0086 (8)
C13	0.0321 (12)	0.0214 (11)	0.0272 (11)	-0.0026 (9)	-0.0069 (10)	-0.0086 (9)
C14	0.0400 (13)	0.0220 (11)	0.0260 (12)	0.0008 (10)	-0.0070 (10)	-0.0041 (9)
C15	0.0439 (14)	0.0332 (13)	0.0254 (12)	0.0074 (11)	-0.0163 (11)	-0.0110 (10)
C16	0.0403 (14)	0.0349 (13)	0.0348 (13)	-0.0014 (11)	-0.0171 (11)	-0.0157 (11)
C17	0.0320 (12)	0.0259 (11)	0.0267 (11)	-0.0045 (9)	-0.0063 (10)	-0.0100 (9)
C18	0.0246 (10)	0.0175 (10)	0.0215 (10)	-0.0004 (8)	-0.0073 (8)	-0.0062 (8)
C19	0.0267 (11)	0.0219 (11)	0.0255 (11)	-0.0002 (8)	-0.0046 (9)	-0.0111 (9)
C20	0.0281 (11)	0.0283 (12)	0.0234 (11)	-0.0072 (9)	-0.0003 (9)	-0.0105 (9)
C21	0.0398 (13)	0.0193 (11)	0.0269 (12)	-0.0065 (9)	-0.0080 (10)	-0.0063 (9)
C22	0.0392 (14)	0.0199 (11)	0.0418 (14)	0.0019 (10)	-0.0078 (11)	-0.0146 (10)
C23	0.0297 (12)	0.0240 (11)	0.0347 (13)	0.0004 (9)	-0.0020 (10)	-0.0118 (10)

Geometric parameters (Å, °)

Mo1—P1	2.4813 (6)	C10—H10A	0.9700
Mo1—C1	1.979 (2)	C10—H10B	0.9700
Mo1—C2	1.960 (2)	C10—C11	1.530 (3)
Mo1—C3	2.273 (2)	C11—H11A	0.9600
Mo1—C5	2.390 (2)	C11—H11B	0.9600
Mo1—C6	2.342 (2)	C11—H11C	0.9600
Mo1—C7	2.321 (2)	C12—C13	1.398 (3)
Mo1—C8	2.340 (2)	C12—C17	1.393 (3)
Mo1—C9	2.368 (2)	C13—H13	0.9300
P1—C10	1.839 (2)	C13—C14	1.386 (3)
P1—C12	1.836 (2)	C14—H14	0.9300
P1—C18	1.841 (2)	C14—C15	1.385 (3)
O1—C1	1.158 (3)	C15—H15	0.9300
O2—C2	1.158 (3)	C15—C16	1.380 (3)
O3—C3	1.223 (3)	C16—H16	0.9300
C3—C4	1.514 (3)	C16—C17	1.395 (3)
C4—H4A	0.9600	C17—H17	0.9300
C4—H4B	0.9600	C18—C19	1.389 (3)
C4—H4C	0.9600	C18—C23	1.392 (3)

C5—H5	0.9300	C19—H19	0.9300
C5—C6	1.408 (3)	C19—C20	1.396 (3)
C5—C9	1.403 (3)	C20—H20	0.9300
C6—H6	0.9300	C20—C21	1.384 (3)
C6—C7	1.422 (3)	C21—H21	0.9300
C7—H7	0.9300	C21—C22	1.379 (3)
C7—C8	1.410 (4)	C22—H22	0.9300
C8—H8	0.9300	C22—C23	1.395 (3)
C8—C9	1.417 (3)	C23—H23	0.9300
C9—H9	0.9300		
C1—Mo1—P1	79.07 (6)	C6—C7—Mo1	73.06 (12)
C1—Mo1—C3	75.46 (8)	C6—C7—H7	126.2
C1—Mo1—C5	97.88 (8)	C8—C7—Mo1	73.12 (13)
C1—Mo1—C6	109.55 (9)	C8—C7—C6	107.7 (2)
C1—Mo1—C7	144.10 (9)	C8—C7—H7	126.2
C1—Mo1—C8	153.01 (9)	Mo1—C8—H8	120.3
C1—Mo1—C9	118.12 (9)	C7—C8—Mo1	71.68 (12)
C2—Mo1—P1	79.67 (6)	C7—C8—H8	126.2
C2—Mo1—C1	106.04 (9)	C7—C8—C9	107.6 (2)
C2—Mo1—C3	73.53 (9)	C9—C8—Mo1	73.60 (12)
C2—Mo1—C5	155.94 (9)	C9—C8—H8	126.2
C2—Mo1—C6	129.67 (9)	Mo1—C9—H9	121.0
C2—Mo1—C7	99.26 (9)	C5—C9—Mo1	73.70 (13)
C2—Mo1—C8	99.09 (9)	C5—C9—C8	108.7 (2)
C2—Mo1—C9	128.81 (9)	C5—C9—H9	125.7
C3—Mo1—P1	135.76 (6)	C8—C9—Mo1	71.38 (12)
C3—Mo1—C5	110.95 (8)	C8—C9—H9	125.7
C3—Mo1—C6	82.22 (8)	P1—C10—H10A	108.7
C3—Mo1—C7	88.08 (8)	P1—C10—H10B	108.7
C3—Mo1—C8	122.06 (9)	H10A—C10—H10B	107.6
C3—Mo1—C9	139.73 (8)	C11—C10—P1	114.02 (16)
C5—Mo1—P1	107.89 (6)	C11—C10—H10A	108.7
C6—Mo1—P1	140.94 (6)	C11—C10—H10B	108.7
C6—Mo1—C5	34.59 (8)	C10—C11—H11A	109.5
C6—Mo1—C9	57.61 (8)	C10—C11—H11B	109.5
C7—Mo1—P1	131.14 (7)	C10—C11—H11C	109.5
C7—Mo1—C5	58.24 (8)	H11A—C11—H11B	109.5
C7—Mo1—C6	35.51 (8)	H11A—C11—H11C	109.5
C7—Mo1—C8	35.20 (9)	H11B—C11—H11C	109.5
C7—Mo1—C9	58.20 (9)	C13—C12—P1	118.36 (16)
C8—Mo1—P1	96.21 (7)	C17—C12—P1	123.11 (16)
C8—Mo1—C5	57.94 (8)	C17—C12—C13	118.5 (2)
C8—Mo1—C6	58.45 (9)	C12—C13—H13	119.5
C8—Mo1—C9	35.03 (8)	C14—C13—C12	120.9 (2)
C9—Mo1—P1	84.34 (6)	C14—C13—H13	119.5
C9—Mo1—C5	34.29 (8)	C13—C14—H14	120.0
C10—P1—Mo1	117.10 (7)	C15—C14—C13	120.0 (2)

C10—P1—C18	103.97 (10)	C15—C14—H14	120.0
C12—P1—Mo1	112.60 (7)	C14—C15—H15	120.1
C12—P1—C10	100.72 (10)	C16—C15—C14	119.8 (2)
C12—P1—C18	102.90 (9)	C16—C15—H15	120.1
C18—P1—Mo1	117.35 (7)	C15—C16—H16	119.8
O1—C1—Mo1	175.79 (19)	C15—C16—C17	120.4 (2)
O2—C2—Mo1	176.56 (19)	C17—C16—H16	119.8
O3—C3—Mo1	120.13 (17)	C12—C17—C16	120.3 (2)
O3—C3—C4	116.7 (2)	C12—C17—H17	119.8
C4—C3—Mo1	123.19 (16)	C16—C17—H17	119.8
C3—C4—H4A	109.5	C19—C18—P1	118.76 (16)
C3—C4—H4B	109.5	C19—C18—C23	118.97 (19)
C3—C4—H4C	109.5	C23—C18—P1	122.26 (17)
H4A—C4—H4B	109.5	C18—C19—H19	119.8
H4A—C4—H4C	109.5	C18—C19—C20	120.4 (2)
H4B—C4—H4C	109.5	C20—C19—H19	119.8
Mo1—C5—H5	122.7	C19—C20—H20	119.9
C6—C5—Mo1	70.86 (12)	C21—C20—C19	120.2 (2)
C6—C5—H5	126.1	C21—C20—H20	119.9
C9—C5—Mo1	72.01 (13)	C20—C21—H21	120.2
C9—C5—H5	126.1	C22—C21—C20	119.7 (2)
C9—C5—C6	107.7 (2)	C22—C21—H21	120.2
Mo1—C6—H6	119.9	C21—C22—H22	119.8
C5—C6—Mo1	74.56 (13)	C21—C22—C23	120.3 (2)
C5—C6—H6	125.9	C23—C22—H22	119.8
C5—C6—C7	108.3 (2)	C18—C23—C22	120.4 (2)
C7—C6—Mo1	71.43 (12)	C18—C23—H23	119.8
C7—C6—H6	125.9	C22—C23—H23	119.8
Mo1—C7—H7	119.5		
Mo1—P1—C10—C11	-60.67 (18)	C9—C5—C6—C7	-1.0 (2)
Mo1—P1—C12—C13	-62.49 (18)	C10—P1—C12—C13	63.03 (19)
Mo1—P1—C12—C17	114.86 (18)	C10—P1—C12—C17	-119.61 (19)
Mo1—P1—C18—C19	-28.34 (19)	C10—P1—C18—C19	-159.42 (17)
Mo1—P1—C18—C23	153.15 (17)	C10—P1—C18—C23	22.1 (2)
Mo1—C5—C6—C7	-63.95 (15)	C12—P1—C10—C11	176.90 (17)
Mo1—C5—C9—C8	63.24 (15)	C12—P1—C18—C19	95.88 (18)
Mo1—C6—C7—C8	-65.44 (15)	C12—P1—C18—C23	-82.6 (2)
Mo1—C7—C8—C9	-65.34 (15)	C12—C13—C14—C15	-0.3 (4)
Mo1—C8—C9—C5	-64.74 (16)	C13—C12—C17—C16	-0.6 (3)
P1—C12—C13—C14	178.29 (18)	C13—C14—C15—C16	-0.5 (4)
P1—C12—C17—C16	-177.99 (18)	C14—C15—C16—C17	0.6 (4)
P1—C18—C19—C20	-178.29 (17)	C15—C16—C17—C12	-0.1 (4)
P1—C18—C23—C22	179.50 (18)	C17—C12—C13—C14	0.8 (3)
C5—C6—C7—Mo1	66.00 (15)	C18—P1—C10—C11	70.56 (18)
C5—C6—C7—C8	0.6 (2)	C18—P1—C12—C13	170.22 (17)
C6—C5—C9—Mo1	-62.23 (15)	C18—P1—C12—C17	-12.4 (2)
C6—C5—C9—C8	1.0 (2)	C18—C19—C20—C21	-1.2 (3)

C6—C7—C8—Mo1	65.40 (15)	C19—C18—C23—C22	1.0 (3)
C6—C7—C8—C9	0.1 (2)	C19—C20—C21—C22	0.9 (3)
C7—C8—C9—Mo1	64.07 (15)	C20—C21—C22—C23	0.3 (4)
C7—C8—C9—C5	-0.7 (2)	C21—C22—C23—C18	-1.3 (4)
C9—C5—C6—Mo1	62.98 (15)	C23—C18—C19—C20	0.3 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C8—H8...O2 ⁱ	0.93	2.63	3.414 (3)	142
C13—H13...O2 ⁱ	0.93	2.71	3.282 (3)	121
C22—H22...O3 ⁱⁱ	0.93	2.66	3.316 (3)	128

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $x, y+1, z$.