

Crystal Structures of Phosphine-Supported (η^5 -cyclopentadienyl)molybdenum(II) Propionyl Complexes

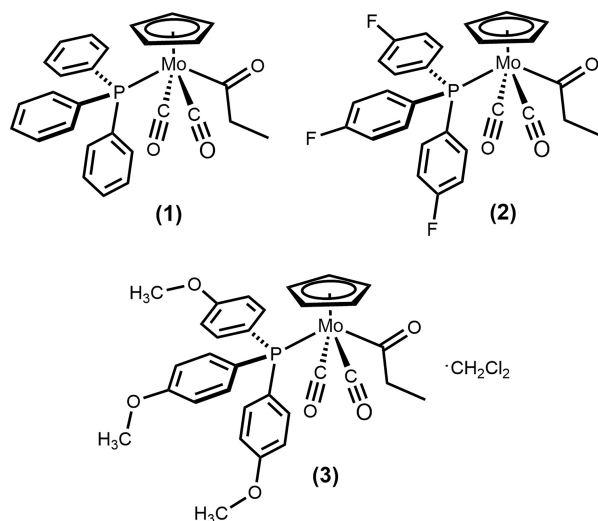
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Abstract

Three cyclopentadienylmolybdenum(II) propionyl complexes featuring triarylphosphine ligands with different *para* substituents (triphenylphosphine (1), tris(4-fluorophenyl)phosphine (2), and tris(4-methoxyphenyl)phosphine (3)) have been prepared from the corresponding ethyl complexes via phosphine-induced migratory insertion. These complexes exhibit four-legged piano-stool geometries with molecular structures quite similar to each other and to related acetyl complexes. The extended structures of the three complexes differ somewhat, with the *para* substituent of the triarylphosphine of (2) (fluoro) or (3) (methoxy) engaging in non-classical C—H $\cdots$ F or C—H $\cdots$ O hydrogen-bonding interactions.



1. Chemical context

Cyclopentadienylmolybdenum(II) complexes featuring carbonyl ligands commonly adopt 'four-legged piano-stool' geometries (Barnett & Slocum, 1972; Kubacek *et al.*, 1982), and those featuring alkyl co-ligands readily undergo migratory insertion to afford Mo(II) acyl complexes upon exposure to phosphines (Barnett & Treichel, 1967; Butler *et al.*, 1967). The effect of changing phosphine substituents on this reaction is well-established, with bulkier phosphines enhancing the rates of subsequent deinsertion from the acyl complexes, a net decarbonylation (Barnett, 1969; Barnett & Pollmann, 1974). Most complexes of the type Mo(C₅H₅)(CO)₂(PR₃)(COR) feature acetyl ligands, though there are limited examples of other acyl complexes that have been structurally characterized (Michelini-Rodriguez *et al.*, 1993; Murshid *et al.*, 2016).

We have previously reported synthetic details and solid-state structures for a number of Mo(II) acetyl complexes of the type described above (Whited & Hofmeister, 2014; Whited *et al.*, 2012, 2014), examining the effect of changing

phosphine substituents on local and supramolecular features. Consistent with reports on decarbonylation reactivity, we have found that the primary impact on molecular structure is observed in the Mo—P bond lengths, with some changes in P—Mo—C bond angles as a result of sterics. Use of tri(2-furyl)phosphine, which features heteroatoms as potential hydrogen-bond acceptors, leads to an unusual structure with the acetyl oriented down, away from the cyclopentadienyl ring rather than up toward it as observed in other cases (Whited *et al.* 2013), and a similar effect was observed by incorporation of a Lewis-acidic manganese unit to interact with the acetyl ligand (Adatia *et al.*, 1986). Recent use of other potentially hydrogen-bonding phosphine ligands did not lead to the same solid-state effect (Anstey *et al.* 2020).

We were interested in extending earlier studies to higher-order alkyl groups at molybdenum, and in this report we describe the synthesis and solid-state structures of related Mo(II) propionyl complexes derived from an ethyl precursor and supported by triarylphosphine ligands differing in their *para* substitution (—H, —F, and —OCH₃). Although substitution of phosphine aryl groups with electron-withdrawing or -donating groups minimally affects local structure, the supramolecular organization is substantially affected by non-classical hydrogen-bonding to the fluoro and methoxy groups in (2) and (3), respectively.

2. Structural commentary

The molecular structures of (1), (2), and (3) are illustrated in Figs. 1–3. All complexes exhibit an overall structure common for CpMo acetyl complexes of this type, with *trans*-disposed carbonyl ligands. As previously observed for most related acetyl complexes, the acyl C=O points up toward the Cp ring. In the case of (1), this phenomenon could be rationalized by presence of short C4—H4A···O1 (2.672 Å) and C4—H4B···O2 (2.639 Å) contacts involving the carbonyl ligands that are enabled when the acyl points up. However, the variation of the Mo1—C3—C4—C5 torsion angle across the series (175.31 (12)° for (1), 172.61 (18)° for (2), and 137.17 (10)° for (3)) argues against the general importance of such an interaction.

Selected geometric parameters for (1), (2), and (3) are presented in Tables 1–3. Complex (2) crystallized with two nearly equivalent molecules in the asymmetric unit, so geometric parameters are presented for both. In general, the three complexes are nearly identical, as might be expected based on the dominant role of sterics in determining structure and the fact that the steric profiles of the three triarylphosphine ligands are identical. The Mo—P bond length in (2) [2.4692 (4) Å (avg)] is slightly shorter than in (1) or (3) [2.4816 (4) Å and 2.4745 (3) Å, respectively], which may be related to stronger π -backbonding to the tris(4-fluorophenyl)phosphine ligand. Stronger backbonding is supported by the observation by infrared spectroscopy of slightly higher-energy carbonyl stretching vibrations for (2) [$\nu(\text{CO})_{\text{avg}} = 1897 \text{ cm}^{-1}$] compared with (1) and (3) [$\nu(\text{CO})_{\text{avg}} = 1893 \text{ cm}^{-1}$ for (1), 1890 cm^{-1} for (3)]. Geometric parameters for all complexes are quite similar to those for the related triphenylphosphine-supported CpMo acetyl complex (Churchill & Fennessey, 1968).

3. Supramolecular features

In spite of the similarities among (1), (2), and (3) in their molecular structures, the *para* substituent of the triarylphosphine ligand (H for (1), F for (2), OCH₃ for (3)) plays an important role in determining the extended structure. The extended structure of (1) is dominated by non-classical C—H···O interactions involving its carbonyl ligands. A short C—H···O interaction between O2 and H12 of a phenyl ring (2.36 Å) joins (1) into centrosymmetrical dimers that are organized into chains along [010] by intermolecular C15—H15···Cg4 (2.952 Å, where Cg4 represents the centroid of the C23—C28 ring) and intramolecular O2—Cg4 (3.295 Å) interactions (Fig. 4). These chains are linked into sheets parallel to (10–1) through another set of non-classical C—H···O interactions (2.60 Å) between O1 of the other carbonyl ligand and H6 from a cyclopentadienyl ligand (Fig. 5).

The tris(4-fluorophenyl)phosphine-supported derivative (3) features two nearly equivalent molecules in the asymmetric unit exhibiting a non-classical C—H $\cdots$ O interaction between O6 of a propionyl ligand and H15 from a phenyl ring (2.59 Å) and a C—H $\cdots$ F close contact between F3 and H53 (2.60 Å). These pairs of molecules are joined into chains along [001] by C34—H34 $\cdots$ O1 interactions (2.38 Å) (Fig. 6). The molecules are further organized parallel to (010) by C—H $\cdots$ F close contacts between F4 and H49 (2.55 Å) and C—H $\cdots$ O interactions between O3 and H55 (2.53 Å) (Fig. 7), then further joined along [010] by C—F $\cdots$ π interactions (F5—Cg4 = 3.17 Å, where Cg4 represents the centroid of the C23–C38 ring).

Like complex (1), complex (3) is joined into centrosymmetrical dimers by a C—H $\cdots$ O interaction involving a carbonyl ligand (C8—H8 $\cdots$ O1, Table 6), and these are linked into chains along [110] through an additional C—H $\cdots$ O interaction between the propionyl oxygen and a cyclopentadienyl ligand (Fig. 8). Additional C31—H31B $\cdots$ O5 interactions along [110], involving a methoxy group from the phosphine ligand, join the molecules into a network parallel to (001). This further set of interactions involving methoxy groups, as well as important close contacts involving the dichloromethane solvent, are depicted in Fig. 9.

4. Database survey

The current version of the Cambridge Structural Database (Version 5.41, updated August 2020; Allen, 2002) has fourteen entries corresponding to molybdenum acyl complexes of the general form Mo(C₅H₅)(CO)₂(PR₃)(COR). The *trans*-dicarbonyl structure, as observed for (1)–(3), is preferred except in cases where the phosphine and acyl ligands are covalently linked, forcing them to be *cis* (Adams *et al.*, 1991; Mercier *et al.*, 1993; Yan *et al.*, 2009).

5. Synthesis and crystallization

CpMo(CO)₃(CH₂CH₃). This compound was prepared by modification of the method used of Gladysz *et al.* (1979), as previously reported by Whited & Hofmeister (2014) and Anstey *et al.* (2020). In a 20-ml scintillation vial equipped with a flea-sized stir bar, [CpMo(CO)₃]₂ (0.1908 g, 0.39 mmol) was dissolved in THF (10 ml). Sodium triethylborohydride (0.87 mL of 1.0-M solution in THF, 0.87 mmol) was added dropwise by syringe with vigorous stirring, leading to an immediate color change from purple to green-yellow with evolution of H₂ gas. The reaction was allowed to proceed with stirring for 20 min, and an excess of iodoethane (0.098 mL, 1.2 mmol) was added dropwise with stirring and the reaction was allowed to proceed for 6 h. Volatiles were removed *in vacuo* to afford a yellow-brown film that was stored at –35 °C for 1 week. The solid was extracted with pentane (4 x 10 ml) and filtered through a 1-cm pad of activated alumina to afford a yellow solution, and removal of solvent *in vacuo* afforded **CpMo(CO)₃(CH₂CH₃)** as a pure yellow powder (0.131 g, 61%). ¹H NMR (400 MHz, CDCl₃): δ 5.28 (s, 5H, Cp ring), 1.72 (q, ³J_{HH} = 7.4 Hz, 2H, –CH₂CH₃), 1.45 (t, ³J_{HH} = 7.4 Hz, 3H, –CH₂CH₃). ¹³C{¹H} NMR (101 MHz, CDCl₃): δ 239.9 (Mo–CO), 227.8 (Mo–CO), 93.0 (Cp ring), 20.4 (Mo–CH₂CH₃)–3.7 (Mo–CH₂CH₃). IR (CH₂Cl₂, NaCl, cm^{–1}) ν (CO): 2015, 1921 (split).

CpMo(CO)₂(PPh₃)(COCH₂CH₃) (1). In an inert-atmosphere glove box, CpMo(CO)₃(CH₂CH₃) (0.0803 g, 0.293 mmol) and triphenylphosphine (0.115 g, 0.440 mmol, 1.5 equiv) were dissolved in acetonitrile (5 ml) in a 20-ml scintillation vial equipped with a flea-sized stir bar. The mixture was stirred for 1 week, during which time a bright yellow precipitate formed. The yellow solid was isolated by filtration and washed with pentane (2 x 5 ml), then dried *in vacuo* to afford pure **1**. Yellow crystals of **1** suitable for X-ray diffraction were obtained from a concentrated dichloromethane solution by vapor cross diffusion with pentane at –35 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.50–7.30 (m, 15H, PPh₃), 5.00 (d, *J* = 1.2 Hz, 5H, C₅H₅), 3.03 (q, ³J_{HH} = 7.2 Hz, 2H, C(O)CH₂CH₃), 0.90 (t, ³J_{HH} = 7.2 Hz, 3H, C(O)CH₂CH₃). ¹³C{¹H} NMR (101 MHz, CDCl₃): δ 267.7 (d, ²J_{CP} = 11 Hz, Mo–COEt), 238.8 (d, ²J_{CP} = 24 Hz, Mo–CO), 135.7 (d, ¹J_{CP} = 44 Hz, *ipso*-C of PPh₃), 133.2 (d, ²J_{CP} = 11 Hz, *ortho*-C of PPh₃), 130.5 (d, ⁴J_{CP} = 2 Hz, *para*-C of

PPh_3), 128.6 (d, $^3J_{\text{CP}} = 11$ Hz, *meta*-C of PPh_3), 96.7 (Cp ring), 58.1 (Mo– COCH_2CH_3), 10.1 (Mo– COCH_2CH_3).

$^{31}\text{P}\{^1\text{H}\}$ NMR (162 MHz, CDCl_3): δ 68.4 (s). IR (CH_2Cl_2 , NaCl, cm^{-1}) $\nu(\text{CO})$: 1935, 1851, 1614 (acetyl).

$\text{CpMo}(\text{CO})_2(\text{P}(\text{4-FPh})_3)(\text{COCH}_2\text{CH}_3)$ (2). In an inert-atmosphere glove box, $\text{CpMo}(\text{CO})_3(\text{CH}_2\text{CH}_3)$ (0.0997 g, 0.36 mmol) and tris(4-fluorophenyl)phosphine (0.17 g, 0.55 mmol, 1.5 equiv) were dissolved in acetonitrile (5 ml) in a 20-ml scintillation vial equipped with a flea-sized stir bar. The mixture was stirred for 1 week, causing a color change to orange, but without formation of any precipitate. Solvent was removed *in vacuo*, causing precipitation of a yellow solid that was isolated by filtration and washed with pentane (2 x 3 ml) to afford the desired product **2** (0.12 g, 56%). Yellow crystals of **2** suitable for X-ray diffraction were obtained from a concentrated dichloromethane solution by vapor cross diffusion with pentane at -35°C . ^1H NMR (400 MHz, CDCl_3): δ 7.41–7.30 (br m, 6H, *ortho*-C–H of phosphine), 7.14 (td, $^3J_{\text{HH}} \approx ^3J_{\text{HF}} = 8.6$ Hz, $^4J_{\text{HP}} = 1.5$ Hz, 6H, *meta*-C–H of phosphine), 4.90 (d, $J = 1.2$ Hz, 5H, C_5H_5), 2.99 (q, $^3J_{\text{HH}} = 7.2$ Hz, 2H, $\text{C}(\text{O})\text{CH}_2\text{CH}_3$), 0.90 (t, $^3J_{\text{HH}} = 7.2$ Hz, 3H, $\text{C}(\text{O})\text{CH}_2\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3): δ 265.4 (d, $^2J_{\text{CP}} = 11$ Hz, Mo–COEt), 238.2 (d, $^2J_{\text{CP}} = 24$ Hz, Mo–CO), 164.0 (dd, $^1J_{\text{CF}} = 253$ Hz, $^4J_{\text{CP}} = 2$ Hz, C–F of phosphine), 135.0 (dd, $^2J_{\text{CP}} = 13$ Hz, $^3J_{\text{CF}} = 8$ Hz, *ortho*-C of phosphine), 131.3 (dd, $^1J_{\text{CP}} = 46$ Hz, $^4J_{\text{CF}} = 4$ Hz, *ipso*-C of phosphine), 116.0 (dd, $^2J_{\text{CF}} = 21$ Hz, $^3J_{\text{CP}} = 11$ Hz, *meta*-C of phosphine), 96.5 (Cp ring), 58.2 (Mo– COCH_2CH_3), 10.9 (Mo– COCH_2CH_3). $^{31}\text{P}\{^1\text{H}\}$ NMR (162 MHz, CDCl_3): δ 68.5 (s). IR (CH_2Cl_2 , NaCl, cm^{-1}) $\nu(\text{CO})$: 1938, 1856, 1620 (acetyl).

$\text{CpMo}(\text{CO})_2(\text{P}(\text{4-MeOPh})_3)(\text{COCH}_2\text{CH}_3)$ (3). In an inert-atmosphere glove box, $\text{CpMo}(\text{CO})_3(\text{CH}_2\text{CH}_3)$ (0.113 g, 0.41 mmol) and tris(4-methoxyphenyl)phosphine (0.218 g, 0.61 mmol, 1.5 equiv) were dissolved in acetonitrile (5 ml) in a 20-ml scintillation vial equipped with a flea-sized stir bar. The mixture was stirred for 1 week, causing precipitation of **3** as a pure yellow powder that was isolated by filtration. Crystals of **3** suitable for X-ray diffraction were obtained from a concentrated dichloromethane solution by vapor cross diffusion with pentane at -35°C . ^1H NMR (400 MHz, CDCl_3): δ 7.37–7.23 (br m, 6H, *ortho*-C–H of phosphine), 7.14 (dd, $^3J_{\text{HH}} = 8.8$ Hz, $^4J_{\text{HP}} = 1.7$ Hz, *meta*-C–H of phosphine), 4.99 (d, $J = 1.2$ Hz, 5H, C_5H_5), 3.03 (q, $^3J_{\text{HH}} = 7.2$ Hz, 2H, $\text{C}(\text{O})\text{CH}_2\text{CH}_3$), 0.89 (t, $^3J_{\text{HH}} = 7.2$ Hz, 3H, $\text{C}(\text{O})\text{CH}_2\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3): δ 268.6 (d, $^2J_{\text{CP}} = 11$ Hz, Mo–COEt), 239.2 (d, $^2J_{\text{CP}} = 24$ Hz, Mo–CO), 161.1 (C–OCH₃ of phosphine), 134.6 (d, $^2J_{\text{CP}} = 12$ Hz, *ortho*-C of phosphine), 127.4 (d, $^1J_{\text{CP}} = 50$ Hz, *ipso*-C of phosphine), 114.0 (d, $^3J_{\text{CP}} = 11$ Hz, *meta*-C of phosphine), 96.6 (Cp ring), 58.0 (Mo– COCH_2CH_3), 10.1 (Mo– COCH_2CH_3). $^{31}\text{P}\{^1\text{H}\}$ NMR (162 MHz, CDCl_3): δ 62.3 (s). IR (CH_2Cl_2 , NaCl, cm^{-1}) $\nu(\text{CO})$: 1933, 1847, 1605 (acetyl).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 7. H atoms were placed in calculated positions and refined in the riding-model approximation with distances of C–H = 0.95, 0.98, 0.99, and 1.00 Å for the phenyl, methyl, methylene, and cyclopentadienyl groups, respectively, and with $U_{\text{iso}}(\text{H}) = k \times U_{\text{eq}}(\text{C})$, $k = 1.2$ for cyclopentadienyl, phenyl, and methylene groups and 1.5 for methyl groups. Methyl group H atoms were allowed to rotate in order to find the best rotameric conformation.

A small number of intense low-angle reflections (three for (1); seven for (2); five for (3)) are missing from these high-quality data sets due to the arrangement of the instrument with a conservatively sized beam stop. The large number of reflections in the data sets (and the Fourier-transform relationship of intensities to atoms) ensures that no particular bias has been introduced.

The structure of (3) exhibited modest disorder in the position of Cl1 of the dichloromethane solvent, which was modeled with two sites showing approximately equivalent occupancies (0.53210 for Cl1A, 0.4769 for Cl1B).

Table 1

Selected geometric parameters (Å, °) for (1)

Mo1—P1	2.4816 (4)	Mo1—C2	1.9662 (15)
Mo1—C1	1.9640 (13)	Mo1—C3	2.2794 (14)
C1—Mo1—C2	107.83 (6)	C1—Mo1—P1	79.34 (4)
C1—Mo1—C3	74.37 (5)	C2—Mo1—P1	78.28 (4)
C2—Mo1—C3	72.47 (5)	C3—Mo1—P1	131.76 (4)
Mo1—C3—C4—C5	175.31 (12)		

Table 2

Hydrogen-bond geometry (Å, °) for (1)

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C12—H12 $\cdots$ O2 ⁱ	0.95	2.36	3.1188 (18)	137
C6—H6 $\cdots$ O1 ⁱⁱ	1.00	2.60	3.545 (2)	158

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x, -y+1, -z$.**Table 3**

Selected geometric parameters (Å, °) for (2)

Mo1—P1	2.4730 (6)	Mo1—C2	1.965 (2)
Mo1—C1	1.966 (2)	Mo1—C3	2.265 (2)
C1—Mo1—C2	109.77 (10)	C1—Mo1—P1	80.12 (7)
C1—Mo1—C3	77.34 (9)	C2—Mo1—P1	79.50 (7)
C2—Mo1—C3	70.93 (9)	C3—Mo1—P1	133.55 (6)
Mo1—C3—C4—C5	172.61 (18)		

Table 4

Hydrogen-bond geometry (Å, °) for (2)

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C15—H15 $\cdots$ O6	0.95	2.59	3.371 (4)	139
C49—H49 $\cdots$ F4 ⁱ	0.95	2.55	3.344 (3)	141
C55—H55 $\cdots$ O3 ⁱⁱ	0.95	2.53	3.450 (3)	164
C34—H34 $\cdots$ O1 ⁱⁱⁱ	1.00	2.38	3.237 (3)	143

Symmetry codes: (i) $x+1, y, z$; (ii) $x-1, y, z$; (iii) $x, -y+1/2, z+1/2$.**Table 5**

Selected geometric parameters (Å, °) for (3)

Mo1—P1	2.4745 (3)	Mo1—C2	1.9658 (12)
Mo1—C1	1.9675 (12)	Mo1—C3	2.2564 (11)
C1—Mo1—C2	106.36 (5)	C1—Mo1—P1	79.97 (3)

C1—Mo1—C3	72.49 (4)	C2—Mo1—P1	79.79 (3)
C2—Mo1—C3	74.79 (4)	C3—Mo1—P1	134.90 (3)
Mo1—C3—C4—C5	137.17 (10)		

Table 6

Hydrogen-bond geometry (Å, °) for (3)

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C31—H31B $\cdots$ O5 ⁱ	0.98	2.58	3.4880 (16)	155
C6—H6 $\cdots$ O3 ⁱⁱ	1.00	2.57	3.4555 (16)	148
C8—H8 $\cdots$ O1 ⁱⁱⁱ	1.00	2.45	3.2714 (16)	139
C32—H32C $\cdots$ O2 ^{iv}	0.99	2.54	3.418 (2)	148

Symmetry codes: (i) $x+1, y+1, z$; (ii) $-x+1, -y, -z$; (iii) $-x+1, -y+1, -z$; (iv) $x-1, y, z$.**Table 7**

Experimental details

	(1)	(2)	(3)
Crystal data			
Chemical formula	C ₂₈ H ₂₅ MoO ₃ P	C ₂₈ H ₂₂ F ₃ MoO ₃ P	C ₃₁ H ₃₁ MoO ₆ P·CH ₂ Cl ₂
<i>M</i> _r	536.39	590.36	711.39
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	170	170	170
<i>a</i> , <i>b</i> , <i>c</i> (Å)	9.1719 (5), 11.7493 (7), 12.6049 (7)	11.7991 (4), 18.6907 (8), 22.4744 (8)	10.5308 (6), 12.1305 (7), 13.6154 (8)
α , β , γ (°)	113.083 (2), 99.148 (2), 99.380 (2)	90, 97.256 (2), 90	97.660 (2), 104.759 (2), 107.081 (2)
<i>V</i> (Å ³)	1195.14 (12)	4916.7 (3)	1566.43 (16)
<i>Z</i>	2	8	2
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.64	0.65	0.68
Crystal size (mm)	0.16 $\times$ 0.16 $\times$ 0.06	0.05 $\times$ 0.05 $\times$ 0.05	0.23 $\times$ 0.21 $\times$ 0.12
Data collection			
Diffractionmeter	Bruker D8 QUEST ECO	Bruker D8 QUEST ECO	Bruker D8 QUEST ECO
Absorption correction	Multi-scan (Krause <i>et al.</i> , 2015)	Multi-scan (Krause <i>et al.</i> , 2015)	Multi-scan (Krause <i>et al.</i> , 2015)
<i>T</i> _{min} , <i>T</i> _{max}	0.89, 0.96	0.86, 0.97	0.84, 0.92
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	49481, 5939, 5674	73758, 10047, 8547	81820, 9578, 9053
<i>R</i> _{int}	0.025	0.042	0.029
(sin θ / λ) _{max} (Å ⁻¹)	0.667	0.625	0.714
Refinement			
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.019, 0.051, 1.07	0.029, 0.063, 1.07	0.020, 0.054, 1.06
No. of reflections	5939	10047	9578
No. of parameters	299	651	393

H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\max}, \Delta\rho_{\min}$ ($\text{e } \text{\AA}^{-3}$)	0.34, −0.40	0.37, −0.39	0.40, −0.48

Computer programs: BIS V6.2.14 (Bruker, 2019), SAINT V8.40B (Bruker, 2019), SHELXT 2018/2 (Sheldrick, 2015), SHELXL2018/3 (Sheldrick, 2015), Mercury 4.3.1 (Macrae *et al.*, 2008), publCIF 1.9.21_c (Westrip, 2010).

Acknowledgements

Carleton College is acknowledged for partial funding to support purchase of the Bruker D8 Quest ECO diffractometer used to perform this work.

Funding information

Funding for this research was provided by: National Science Foundation, Directorate for Mathematical and Physical Sciences (award No. CHE-1552591 to M. T. Whited); Camille and Henry Dreyfus Foundation (award No. TH-16-035 to M. T. Whited).

References

- Adams, H., Bailey, N. A., Day, A. N., Morris, M. J. & Harrison, M. M. (1991). *J. Organomet. Chem.* **407**, 247–258.
- Adatia, T., Henrick, K., Horton, A. D., Mays, M. J. & McPartin, M. (1986). *J. Chem. Soc. Chem. Commun.*, 1206–1208.
- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
- Anstey, M. R., Bost, J. L., Grumman, A. S., Kennedy, N. D. & Whited, M. T. (2020). *Acta Cryst.* **E76**, 547–551.
- Barnett, K. W. (1969). *Inorg. Chem.* **8**, 2009–2011.
- Barnett, K. W. & Pollmann, T. G. (1974). *J. Organomet. Chem.* **69**, 413–421.
- Barnett, K. W. & Slocum, D. W. (1972). *J. Organomet. Chem.* **44**, 1–37.
- Barnett, K. W. & Treichel, P. M. (1967). *Inorg. Chem.* **6**, 294–299.
- Bruker (2019). *APEX3*. Bruker Nano, Inc., Madison, WI, USA.
- Bruker (2019). *BIS*. Bruker Nano, Inc., Madison, WI, USA.
- Bruker (2019). *SAINT*. Bruker Nano, Inc., Madison, WI, USA.
- Butler, I. S., Basolo, F. & Pearson, R. G. (1967). *Inorg. Chem.* **6**, 2074–2079.
- Churchill, M. R. & Fennessey, J. P. (1968). *Inorg. Chem.* **7**, 953–959.
- Gladysz, J. A., Williams, G. M., Tam, W., Johnson, D. L., Parker, D. W. & Selover, J. C. (1979). *Inorg. Chem.* **18**, 553–558.
- Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.
- Kubacek, P., Hoffmann, R. & Zdenek, H. (1982). *Organometallics* **1**, 180–188.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Eglington, P. R., McCabe, P., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Mercier, F., Ricard, L. & Mathey, F. (1993). *Organometallics*, **12**, 98–103.
- Michellini-Rodriguez, I., Romero, A. L., Kapoor, R. N., Cervantes-Lee, F. & Pannell, K. H. (1993). *Organometallics* **12**, 1221–1224.
- Murshid, N., Rahman, M. A. & Wang, X. (2016). *J. Organomet. Chem.* **819**, 109–114.

Sheldrick, G. M. (2015). *Acta Cryst.* **A71**, 3–8.

Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

Whited, M. T., Bakker-Arkema, J. G., Greenwald, J. E., Morrill, L. A. & Janzen, D. E. (2013). *Acta Cryst.* **E69**, m475–m476.

Whited, M. T., Boerma, J. W., McClellan, M. J., Padilla, C. E. & Janzen, D. E. (2012). *Acta Cryst.* **E68**, m1158–m1159.

Whited, M. T. & Hofmeister, G. E. (2014). *J. Chem. Educ.* **91**, 1050–1053.

Whited, M. T., Hofmeister, G. E., Hodges, C. J., Jensen, L. J., Keyes, S. H., Ngamnithiporn, A. & Janzen, D. E. (2014). *Acta Cryst.* **E70**, 216–220.

Yan, X., Yu, B., Tang, N., Wang, L. & Xi, C. (2009). *Organometallics*, **28**, 6827–6830.

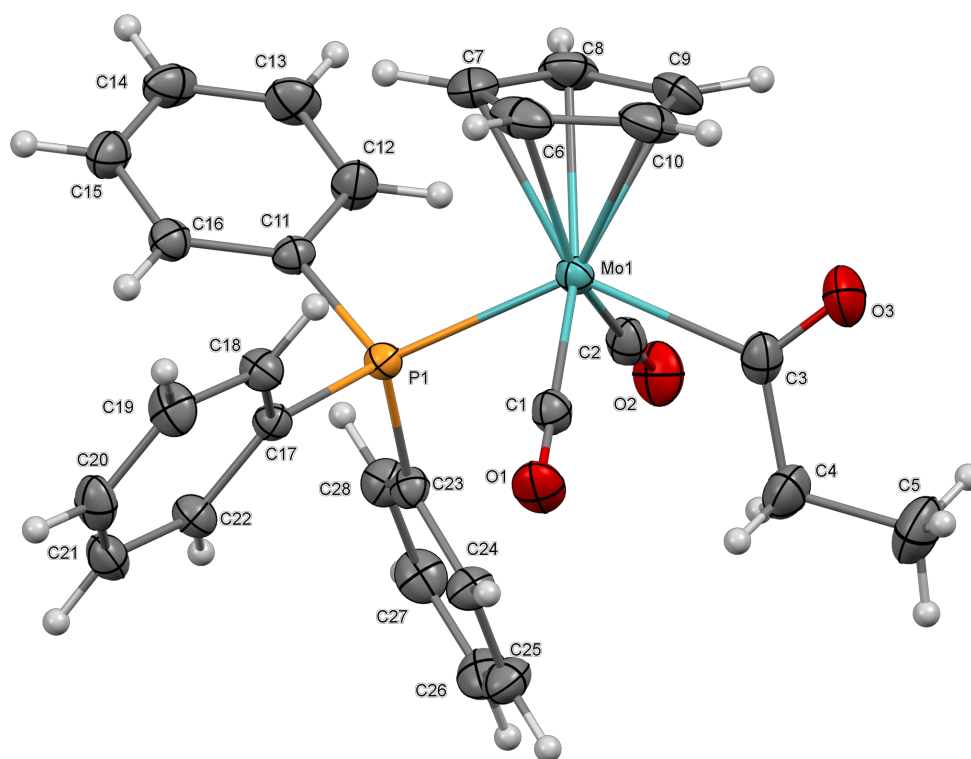
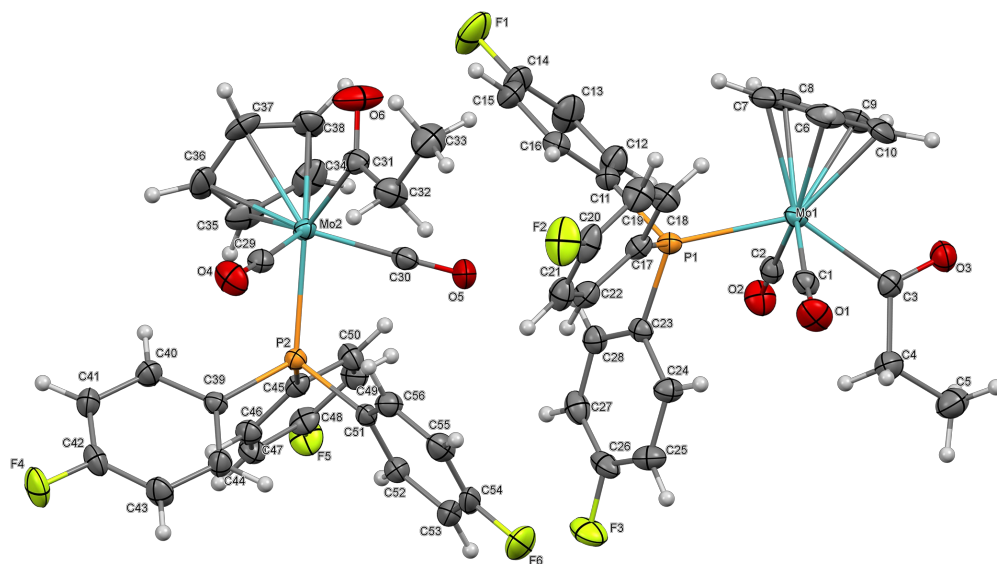


Figure 1

Molecular structure of (1) with ellipsoids at 50% probability.

**Figure 2**

Molecular structure of (2) with ellipsoids at 50% probability.

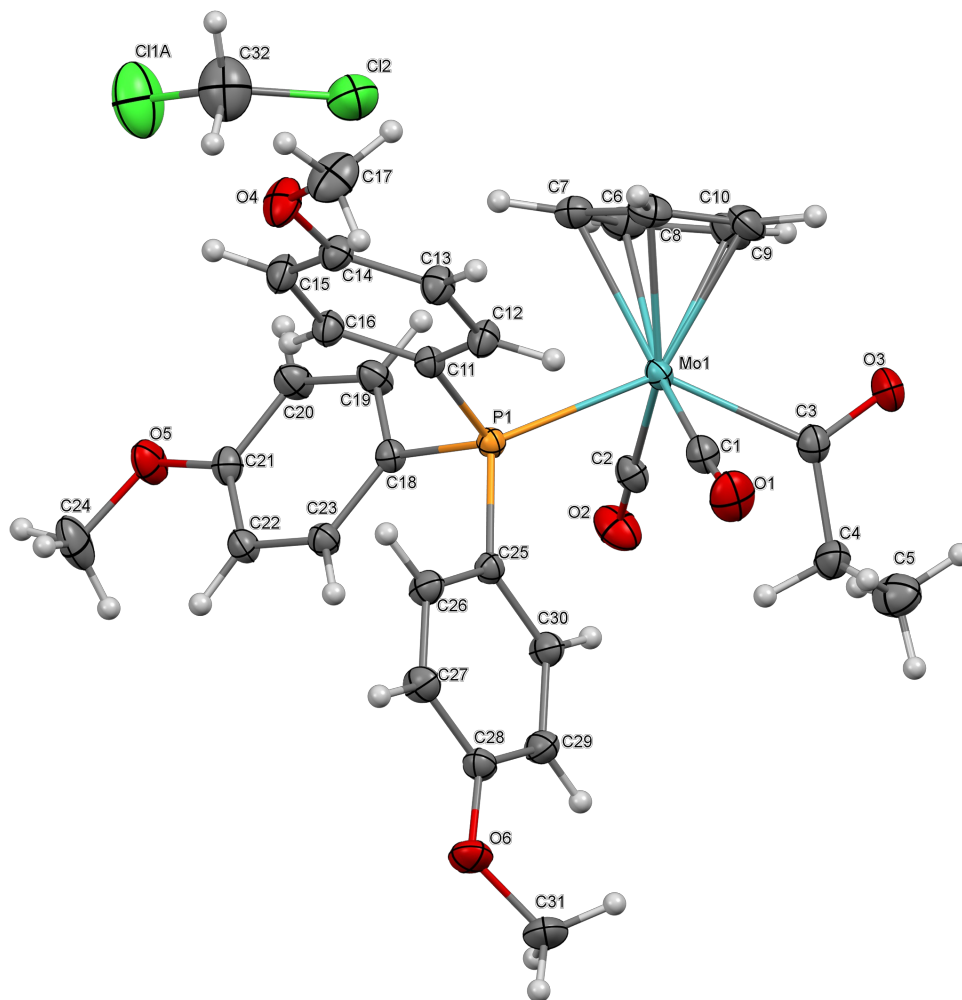


Figure 3
Molecular structure of (3) with ellipsoids at 50% probability.

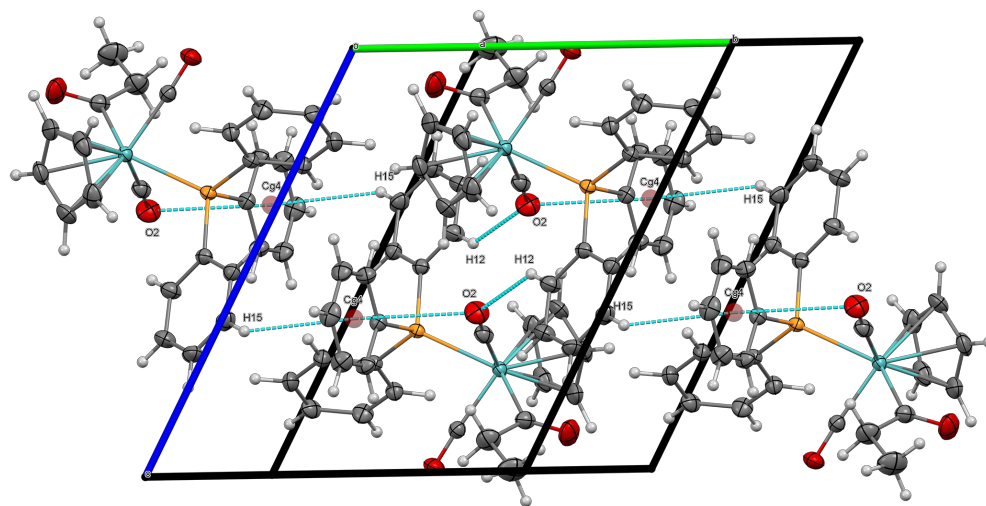


Figure 4

Chains of (1) along [010], viewed along [3–10].

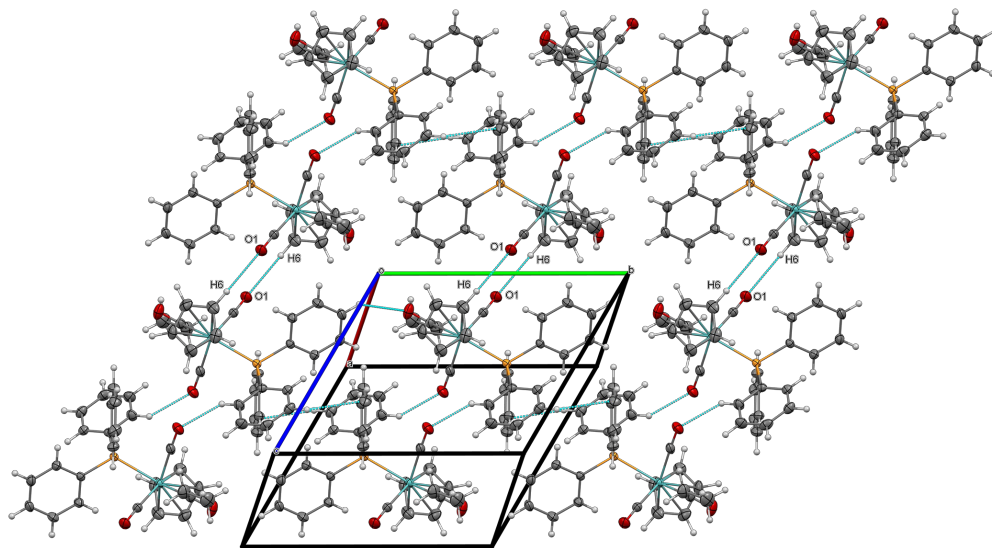


Figure 5

Sheets of (1) formed by C—H $\cdots$ O interactions, viewed perpendicular to (10–1).

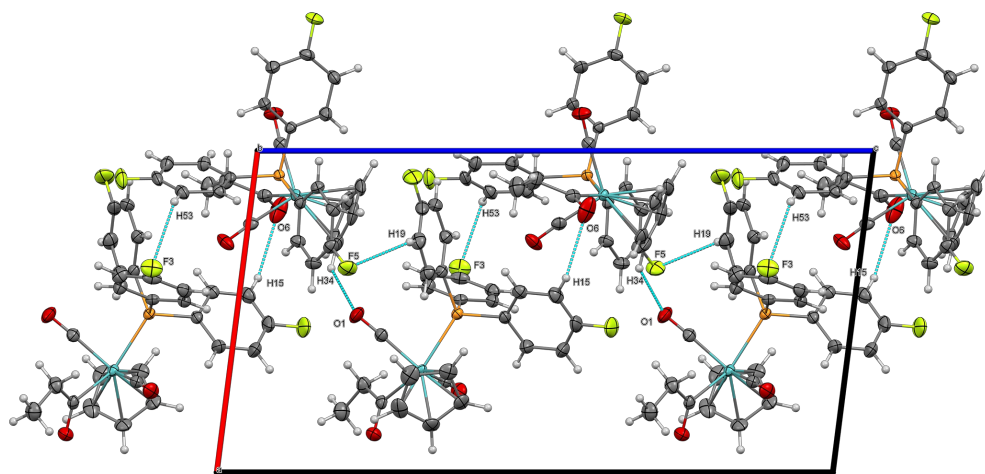


Figure 6

Chains along [001] of the two nearly identical molecules of (2) in the asymmetric unit, with their C—H $\cdots$ O and C—H $\cdots$ F interactions, viewed along [010].

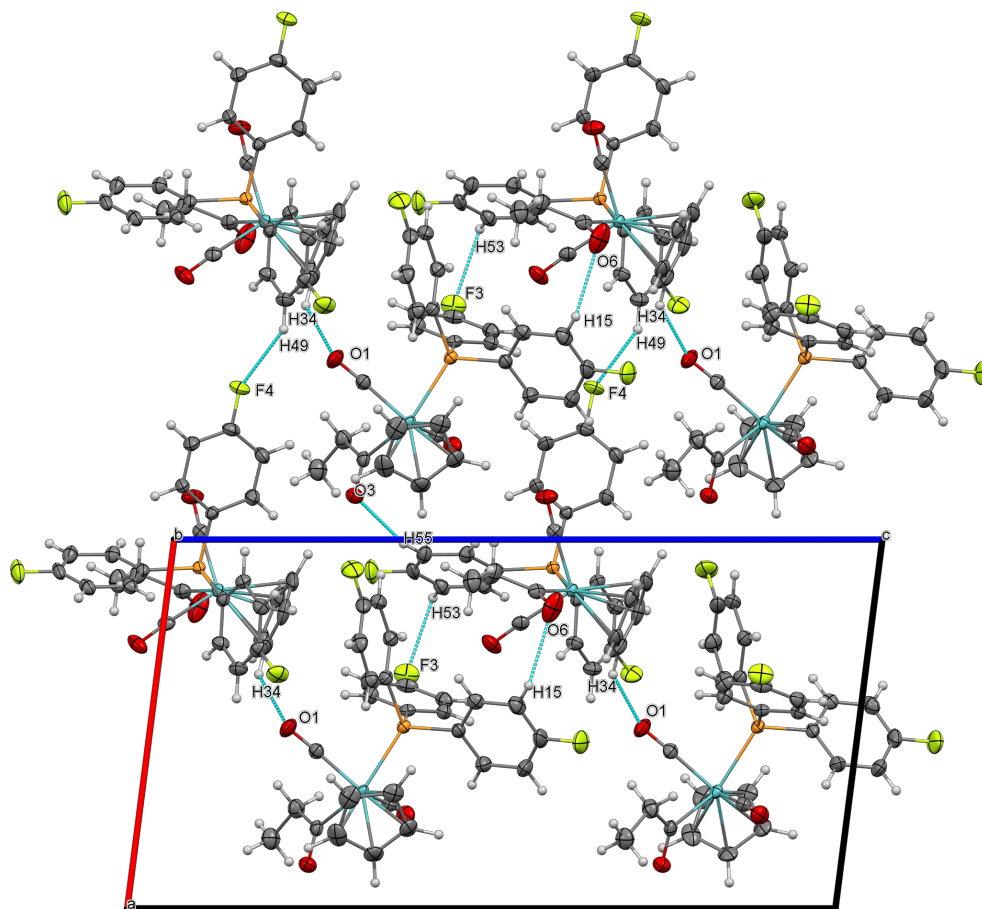


Figure 7

Sheets of (2) parallel to (010), viewed perpendicular to (010).

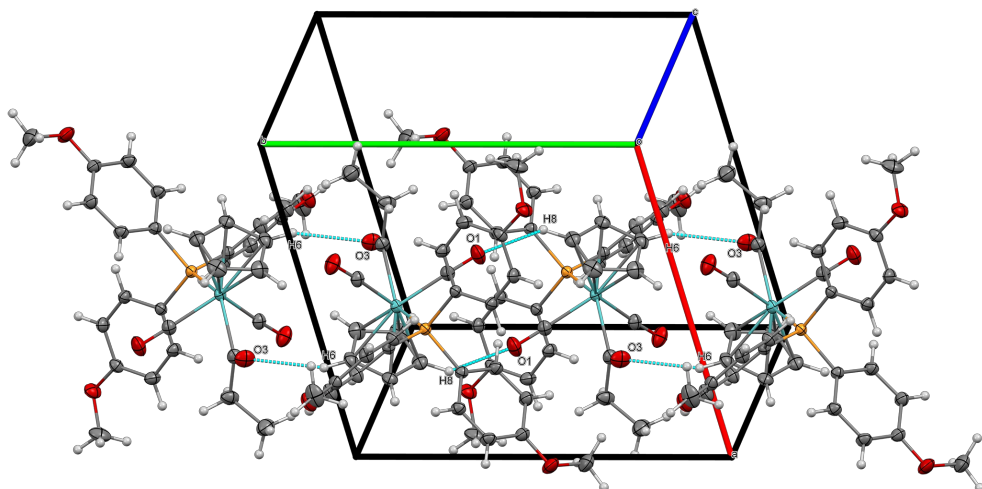


Figure 8

Chains of (3) along [010], viewed perpendicular to (001).

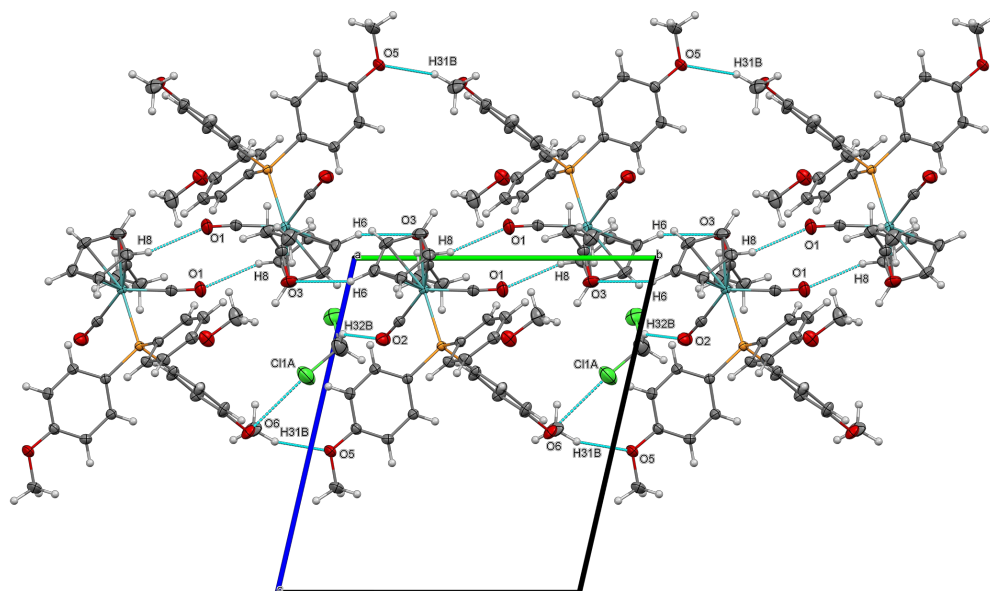


Figure 9

Network of complex (3) formed by interactions featuring methoxy groups and dichloromethane solvent, viewed along [100].

supporting information

Crystal Structures of Phosphine-Supported (η^5 -cyclopentadienyl)molybdenum(II) Propionyl Complexes

Matthew T. Whited,* Margaret A. Ball, Alison Block, Benjamin A. Brewster, LouLou Ferrer, Helen J. Jin-Lee, Colby J. King, Jamie D. North, Inger L. Shelton and David G. Wilson

Computing details

For all compounds, data collection: BIS V6.2.14 (Bruker, 2019); cell refinement: *SAINT* V8.40B (Bruker, 2019); data reduction: *SAINT* V8.40B (Bruker, 2019); program(s) used to solve structure: SHELXT 2018/2 (Sheldrick, 2015); program(s) used to refine structure: *SHELXL* 2018/3 (Sheldrick, 2015); molecular graphics: *Mercury* 4.3.1 (Macrae *et al.*, 2008); software used to prepare material for publication: *publCIF* 1.9.21_c (Westrip, 2010).

(1)

Crystal data

$C_{28}H_{25}MoO_3P$	$Z = 2$
$M_r = 536.39$	$F(000) = 548$
Triclinic, $P\bar{1}$	$D_x = 1.491 \text{ Mg m}^{-3}$
$a = 9.1719 (5) \text{ \AA}$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$b = 11.7493 (7) \text{ \AA}$	Cell parameters from 9085 reflections
$c = 12.6049 (7) \text{ \AA}$	$\theta = 2.6\text{--}33.1^\circ$
$\alpha = 113.083 (2)^\circ$	$\mu = 0.64 \text{ mm}^{-1}$
$\beta = 99.148 (2)^\circ$	$T = 170 \text{ K}$
$\gamma = 99.380 (2)^\circ$	Prism, yellow
$V = 1195.14 (12) \text{ \AA}^3$	$0.16 \times 0.16 \times 0.06 \text{ mm}$

Data collection

Bruker D8 QUEST ECO	$T_{\min} = 0.89, T_{\max} = 0.96$
diffractometer	49481 measured reflections
Radiation source: sealed tube, Siemens	5939 independent reflections
KFFMO2K-90C	5674 reflections with $I > 2\sigma(I)$
Curved Graphite monochromator	$R_{\text{int}} = 0.025$
Detector resolution: $7.3910 \text{ pixels mm}^{-1}$	$\theta_{\max} = 28.3^\circ, \theta_{\min} = 2.3^\circ$
φ and ω scans	$h = -12 \rightarrow 12$
Absorption correction: multi-scan	$k = -15 \rightarrow 15$
(Krause <i>et al.</i> , 2015)	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.019$	H-atom parameters constrained
$wR(F^2) = 0.051$	$w = 1/[\sigma^2(F_o^2) + (0.022P)^2 + 0.6108P]$
$S = 1.07$	where $P = (F_o^2 + 2F_c^2)/3$
5939 reflections	$(\Delta/\sigma)_{\max} = 0.001$
299 parameters	$\Delta\rho_{\max} = 0.34 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.40 \text{ e \AA}^{-3}$
Primary atom site location: dual	

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$) for (1)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Mo1	0.21851 (2)	0.45813 (2)	0.23960 (2)	0.01978 (4)
P1	0.32246 (3)	0.69322 (3)	0.33370 (3)	0.01786 (6)
O1	0.22653 (14)	0.50413 (11)	0.01296 (9)	0.0343 (2)
O2	0.54687 (13)	0.48265 (11)	0.37404 (11)	0.0391 (3)
O3	0.23712 (16)	0.18616 (11)	0.08762 (12)	0.0459 (3)
C1	0.22950 (15)	0.48927 (12)	0.09861 (11)	0.0233 (2)
C2	0.42735 (17)	0.47555 (13)	0.32222 (12)	0.0267 (3)
C3	0.30515 (18)	0.29556 (13)	0.11888 (12)	0.0288 (3)
C4	0.4478 (2)	0.32080 (16)	0.07449 (15)	0.0390 (4)
H4A	0.428632	0.366718	0.024555	0.047*
H4B	0.533049	0.37787	0.144096	0.047*
C5	0.4970 (2)	0.20261 (19)	0.00328 (17)	0.0504 (5)
H5A	0.420288	0.151328	−0.072025	0.076*
H5B	0.507534	0.15204	0.048824	0.076*
H5C	0.595154	0.227972	−0.013045	0.076*
C6	−0.05044 (17)	0.42011 (17)	0.21381 (16)	0.0377 (4)
H6	−0.121988	0.444525	0.162697	0.045*
C7	0.01630 (18)	0.49182 (16)	0.33645 (15)	0.0356 (3)
H7	0.00108	0.576933	0.387033	0.043*
C8	0.09701 (19)	0.41922 (16)	0.37808 (14)	0.0348 (3)
H8	0.146453	0.442892	0.463005	0.042*
C9	0.08355 (19)	0.30236 (15)	0.28055 (15)	0.0354 (3)
H9	0.118582	0.227614	0.284712	0.042*
C10	−0.00792 (19)	0.30284 (16)	0.17850 (15)	0.0369 (3)
H10	−0.047308	0.228557	0.098522	0.044*
C11	0.29484 (14)	0.77386 (12)	0.48373 (11)	0.0209 (2)
C12	0.32519 (18)	0.71890 (14)	0.56225 (12)	0.0296 (3)
H12	0.360821	0.643324	0.537306	0.035*
C13	0.30390 (19)	0.77344 (15)	0.67645 (13)	0.0337 (3)
H13	0.325727	0.735596	0.729406	0.04*
C14	0.25087 (18)	0.88295 (15)	0.71309 (13)	0.0329 (3)
H14	0.235238	0.919963	0.790907	0.039*
C15	0.22073 (17)	0.93831 (14)	0.63604 (13)	0.0309 (3)
H15	0.1844	1.013525	0.661221	0.037*
C16	0.24327 (15)	0.88454 (12)	0.52163 (12)	0.0238 (2)
H16	0.223316	0.923696	0.46953	0.029*
C17	0.25504 (14)	0.78737 (12)	0.25929 (11)	0.0190 (2)
C18	0.10476 (15)	0.74751 (13)	0.19178 (12)	0.0240 (2)
H18	0.041175	0.669342	0.180761	0.029*
C19	0.04765 (17)	0.82174 (14)	0.14056 (13)	0.0302 (3)
H19	−0.055523	0.795183	0.096435	0.036*
C20	0.14075 (18)	0.93438 (14)	0.15365 (13)	0.0305 (3)

H20	0.101594	0.984707	0.118267	0.037*
C21	0.29082 (18)	0.97327 (13)	0.21836 (13)	0.0287 (3)
H21	0.355199	1.049602	0.226151	0.034*
C22	0.34774 (15)	0.90100 (12)	0.27206 (12)	0.0242 (3)
H22	0.450298	0.929118	0.317696	0.029*
C23	0.52909 (14)	0.73720 (12)	0.35437 (11)	0.0207 (2)
C24	0.58606 (15)	0.69609 (13)	0.25280 (12)	0.0258 (3)
H24	0.5177	0.656619	0.176174	0.031*
C25	0.74111 (16)	0.71250 (15)	0.26317 (14)	0.0319 (3)
H25	0.779018	0.68406	0.1938	0.038*
C26	0.84183 (16)	0.77070 (15)	0.37523 (15)	0.0331 (3)
H26	0.948349	0.781057	0.382243	0.04*
C27	0.78743 (16)	0.81325 (14)	0.47583 (13)	0.0310 (3)
H27	0.856515	0.853859	0.552162	0.037*
C28	0.63068 (16)	0.79681 (13)	0.46591 (12)	0.0264 (3)
H28	0.593438	0.826442	0.53553	0.032*

Atomic displacement parameters ($\text{\AA}^2$) for (1)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.02476 (6)	0.01872 (6)	0.01856 (6)	0.00571 (4)	0.00643 (4)	0.01018 (4)
P1	0.01821 (14)	0.01985 (14)	0.01678 (14)	0.00582 (11)	0.00409 (11)	0.00879 (11)
O1	0.0494 (6)	0.0349 (6)	0.0221 (5)	0.0107 (5)	0.0090 (4)	0.0155 (4)
O2	0.0397 (6)	0.0413 (6)	0.0407 (6)	0.0187 (5)	0.0014 (5)	0.0220 (5)
O3	0.0611 (8)	0.0230 (5)	0.0525 (7)	0.0117 (5)	0.0214 (6)	0.0118 (5)
C1	0.0263 (6)	0.0203 (6)	0.0214 (6)	0.0053 (5)	0.0048 (5)	0.0078 (5)
C2	0.0368 (7)	0.0246 (6)	0.0237 (6)	0.0131 (5)	0.0088 (5)	0.0129 (5)
C3	0.0399 (8)	0.0254 (6)	0.0231 (6)	0.0133 (6)	0.0073 (6)	0.0105 (5)
C4	0.0481 (9)	0.0354 (8)	0.0361 (8)	0.0188 (7)	0.0199 (7)	0.0112 (7)
C5	0.0631 (12)	0.0475 (10)	0.0411 (9)	0.0299 (9)	0.0218 (9)	0.0097 (8)
C6	0.0248 (7)	0.0488 (9)	0.0495 (9)	0.0041 (6)	0.0104 (6)	0.0327 (8)
C7	0.0338 (8)	0.0335 (8)	0.0460 (9)	0.0091 (6)	0.0235 (7)	0.0183 (7)
C8	0.0409 (8)	0.0411 (8)	0.0281 (7)	0.0056 (7)	0.0165 (6)	0.0194 (6)
C9	0.0462 (9)	0.0290 (7)	0.0416 (8)	0.0071 (6)	0.0193 (7)	0.0236 (7)
C10	0.0378 (8)	0.0338 (8)	0.0336 (8)	−0.0071 (6)	0.0109 (6)	0.0142 (6)
C11	0.0198 (5)	0.0229 (6)	0.0189 (5)	0.0043 (5)	0.0048 (4)	0.0083 (5)
C12	0.0393 (8)	0.0309 (7)	0.0231 (6)	0.0137 (6)	0.0095 (6)	0.0136 (6)
C13	0.0438 (8)	0.0366 (8)	0.0220 (6)	0.0064 (6)	0.0086 (6)	0.0149 (6)
C14	0.0375 (8)	0.0317 (7)	0.0217 (6)	0.0000 (6)	0.0117 (6)	0.0052 (5)
C15	0.0332 (7)	0.0243 (6)	0.0296 (7)	0.0057 (5)	0.0120 (6)	0.0050 (5)
C16	0.0231 (6)	0.0221 (6)	0.0238 (6)	0.0034 (5)	0.0054 (5)	0.0085 (5)
C17	0.0209 (5)	0.0210 (6)	0.0176 (5)	0.0069 (4)	0.0057 (4)	0.0096 (5)
C18	0.0217 (6)	0.0248 (6)	0.0265 (6)	0.0047 (5)	0.0041 (5)	0.0130 (5)
C19	0.0272 (7)	0.0330 (7)	0.0308 (7)	0.0092 (6)	−0.0002 (5)	0.0163 (6)
C20	0.0430 (8)	0.0266 (7)	0.0270 (7)	0.0144 (6)	0.0060 (6)	0.0153 (6)
C21	0.0382 (7)	0.0215 (6)	0.0279 (7)	0.0054 (5)	0.0083 (6)	0.0127 (5)
C22	0.0244 (6)	0.0226 (6)	0.0245 (6)	0.0033 (5)	0.0041 (5)	0.0107 (5)
C23	0.0192 (5)	0.0212 (6)	0.0223 (6)	0.0064 (4)	0.0043 (5)	0.0096 (5)
C24	0.0218 (6)	0.0286 (7)	0.0229 (6)	0.0043 (5)	0.0043 (5)	0.0080 (5)
C25	0.0255 (7)	0.0342 (7)	0.0335 (7)	0.0090 (6)	0.0119 (6)	0.0097 (6)
C26	0.0187 (6)	0.0348 (7)	0.0442 (8)	0.0079 (5)	0.0050 (6)	0.0158 (7)

C27	0.0250 (6)	0.0325 (7)	0.0302 (7)	0.0037 (5)	−0.0029 (5)	0.0132 (6)
C28	0.0254 (6)	0.0281 (6)	0.0231 (6)	0.0063 (5)	0.0029 (5)	0.0096 (5)

Geometric parameters (Å, °) for (I)

Mo1—P1	2.4816 (4)	C11—C12	1.3951 (18)
Mo1—C1	1.9640 (13)	C12—C13	1.388 (2)
Mo1—C2	1.9662 (15)	C12—H12	0.95
Mo1—C3	2.2794 (14)	C13—C14	1.384 (2)
Mo1—C9	2.3138 (14)	C13—H13	0.95
Mo1—C10	2.3216 (15)	C14—C15	1.382 (2)
Mo1—C8	2.3612 (14)	C14—H14	0.95
Mo1—C6	2.3795 (15)	C15—C16	1.3945 (19)
Mo1—C7	2.3805 (15)	C15—H15	0.95
P1—C17	1.8270 (12)	C16—H16	0.95
P1—C23	1.8289 (13)	C17—C18	1.3956 (17)
P1—C11	1.8341 (13)	C17—C22	1.3961 (18)
O1—C1	1.1563 (17)	C18—C19	1.3910 (18)
O2—C2	1.1555 (18)	C18—H18	0.95
O3—C3	1.2083 (19)	C19—C20	1.387 (2)
C3—C4	1.533 (2)	C19—H19	0.95
C4—C5	1.514 (2)	C20—C21	1.384 (2)
C4—H4A	0.99	C20—H20	0.95
C4—H4B	0.99	C21—C22	1.3909 (19)
C5—H5A	0.98	C21—H21	0.95
C5—H5B	0.98	C22—H22	0.95
C5—H5C	0.98	C23—C28	1.3907 (18)
C6—C7	1.408 (2)	C23—C24	1.3984 (18)
C6—C10	1.414 (3)	C24—C25	1.3814 (19)
C6—H6	1.0	C24—H24	0.95
C7—C8	1.409 (2)	C25—C26	1.392 (2)
C7—H7	1.0	C25—H25	0.95
C8—C9	1.409 (2)	C26—C27	1.376 (2)
C8—H8	1.0	C26—H26	0.95
C9—C10	1.422 (2)	C27—C28	1.398 (2)
C9—H9	1.0	C27—H27	0.95
C10—H10	1.0	C28—H28	0.95
C11—C16	1.3886 (18)		
C1—Mo1—C2	107.83 (6)	C7—C8—Mo1	73.47 (8)
C1—Mo1—C3	74.37 (5)	C9—C8—Mo1	70.63 (8)
C2—Mo1—C3	72.47 (5)	C7—C8—H8	125.9
C1—Mo1—C9	136.85 (6)	C9—C8—H8	125.9
C2—Mo1—C9	100.67 (6)	Mo1—C8—H8	125.9
C3—Mo1—C9	84.47 (6)	C8—C9—C10	107.75 (14)
C1—Mo1—C10	103.82 (6)	C8—C9—Mo1	74.31 (8)
C2—Mo1—C10	133.28 (6)	C10—C9—Mo1	72.43 (8)
C3—Mo1—C10	84.30 (6)	C8—C9—H9	125.8
C9—Mo1—C10	35.73 (6)	C10—C9—H9	125.8
C1—Mo1—C8	155.48 (6)	Mo1—C9—H9	125.8
C2—Mo1—C8	96.57 (6)	C6—C10—C9	108.10 (15)

C3—Mo1—C8	116.63 (6)	C6—C10—Mo1	74.76 (9)
C9—Mo1—C8	35.06 (6)	C9—C10—Mo1	71.83 (9)
C10—Mo1—C8	58.45 (6)	C6—C10—H10	125.7
C1—Mo1—C6	97.82 (6)	C9—C10—H10	125.7
C2—Mo1—C6	154.34 (6)	Mo1—C10—H10	125.7
C3—Mo1—C6	116.12 (6)	C16—C11—C12	118.96 (12)
C9—Mo1—C6	58.55 (6)	C16—C11—P1	123.40 (10)
C10—Mo1—C6	34.97 (6)	C12—C11—P1	117.64 (10)
C8—Mo1—C6	57.79 (6)	C13—C12—C11	120.73 (14)
C1—Mo1—C7	123.06 (6)	C13—C12—H12	119.6
C2—Mo1—C7	123.54 (6)	C11—C12—H12	119.6
C3—Mo1—C7	140.20 (6)	C14—C13—C12	119.95 (14)
C9—Mo1—C7	58.03 (6)	C14—C13—H13	120.0
C10—Mo1—C7	57.85 (6)	C12—C13—H13	120.0
C8—Mo1—C7	34.56 (6)	C15—C14—C13	119.77 (13)
C6—Mo1—C7	34.42 (6)	C15—C14—H14	120.1
C1—Mo1—P1	79.34 (4)	C13—C14—H14	120.1
C2—Mo1—P1	78.28 (4)	C14—C15—C16	120.48 (14)
C3—Mo1—P1	131.76 (4)	C14—C15—H15	119.8
C9—Mo1—P1	139.01 (4)	C16—C15—H15	119.8
C10—Mo1—P1	141.74 (5)	C11—C16—C15	120.10 (13)
C8—Mo1—P1	103.97 (4)	C11—C16—H16	119.9
C6—Mo1—P1	106.91 (4)	C15—C16—H16	119.9
C7—Mo1—P1	88.01 (4)	C18—C17—C22	118.98 (12)
C17—P1—C23	102.95 (6)	C18—C17—P1	119.14 (9)
C17—P1—C11	102.96 (6)	C22—C17—P1	121.83 (10)
C23—P1—C11	103.95 (6)	C19—C18—C17	120.29 (12)
C17—P1—Mo1	119.32 (4)	C19—C18—H18	119.9
C23—P1—Mo1	111.87 (4)	C17—C18—H18	119.9
C11—P1—Mo1	114.09 (4)	C20—C19—C18	120.26 (13)
O1—C1—Mo1	175.81 (12)	C20—C19—H19	119.9
O2—C2—Mo1	176.43 (13)	C18—C19—H19	119.9
O3—C3—C4	118.46 (14)	C21—C20—C19	119.83 (13)
O3—C3—Mo1	119.78 (12)	C21—C20—H20	120.1
C4—C3—Mo1	121.76 (10)	C19—C20—H20	120.1
C5—C4—C3	115.02 (15)	C20—C21—C22	120.23 (13)
C5—C4—H4A	108.5	C20—C21—H21	119.9
C3—C4—H4A	108.5	C22—C21—H21	119.9
C5—C4—H4B	108.5	C21—C22—C17	120.39 (12)
C3—C4—H4B	108.5	C21—C22—H22	119.8
H4A—C4—H4B	107.5	C17—C22—H22	119.8
C4—C5—H5A	109.5	C28—C23—C24	119.14 (12)
C4—C5—H5B	109.5	C28—C23—P1	122.88 (10)
H5A—C5—H5B	109.5	C24—C23—P1	117.61 (10)
C4—C5—H5C	109.5	C25—C24—C23	120.42 (13)
H5A—C5—H5C	109.5	C25—C24—H24	119.8
H5B—C5—H5C	109.5	C23—C24—H24	119.8
C7—C6—C10	107.44 (14)	C24—C25—C26	120.02 (14)
C7—C6—Mo1	72.83 (9)	C24—C25—H25	120.0
C10—C6—Mo1	70.27 (9)	C26—C25—H25	120.0
C7—C6—H6	126.2	C27—C26—C25	120.14 (13)

C10—C6—H6	126.2	C27—C26—H26	119.9
Mo1—C6—H6	126.2	C25—C26—H26	119.9
C6—C7—C8	108.81 (15)	C26—C27—C28	120.10 (13)
C6—C7—Mo1	72.75 (9)	C26—C27—H27	120.0
C8—C7—Mo1	71.97 (8)	C28—C27—H27	120.0
C6—C7—H7	125.5	C23—C28—C27	120.16 (13)
C8—C7—H7	125.5	C23—C28—H28	119.9
Mo1—C7—H7	125.5	C27—C28—H28	119.9
C7—C8—C9	107.89 (14)		
O3—C3—C4—C5	−5.3 (2)	C14—C15—C16—C11	0.7 (2)
Mo1—C3—C4—C5	175.31 (12)	C23—P1—C17—C18	157.21 (10)
C10—C6—C7—C8	−1.24 (17)	C11—P1—C17—C18	−94.93 (11)
Mo1—C6—C7—C8	−63.38 (11)	Mo1—P1—C17—C18	32.63 (12)
C10—C6—C7—Mo1	62.13 (10)	C23—P1—C17—C22	−25.50 (12)
C6—C7—C8—C9	1.22 (17)	C11—P1—C17—C22	82.36 (11)
Mo1—C7—C8—C9	−62.66 (10)	Mo1—P1—C17—C22	−150.08 (9)
C6—C7—C8—Mo1	63.88 (11)	C22—C17—C18—C19	−1.5 (2)
C7—C8—C9—C10	−0.72 (17)	P1—C17—C18—C19	175.84 (11)
Mo1—C8—C9—C10	−65.22 (10)	C17—C18—C19—C20	1.6 (2)
C7—C8—C9—Mo1	64.51 (11)	C18—C19—C20—C21	−0.3 (2)
C7—C6—C10—C9	0.79 (17)	C19—C20—C21—C22	−1.2 (2)
Mo1—C6—C10—C9	64.59 (11)	C20—C21—C22—C17	1.3 (2)
C7—C6—C10—Mo1	−63.80 (10)	C18—C17—C22—C21	0.1 (2)
C8—C9—C10—C6	−0.05 (17)	P1—C17—C22—C21	−177.21 (10)
Mo1—C9—C10—C6	−66.52 (10)	C17—P1—C23—C28	118.22 (11)
C8—C9—C10—Mo1	66.48 (11)	C11—P1—C23—C28	11.11 (13)
C17—P1—C11—C16	−3.65 (12)	Mo1—P1—C23—C28	−112.46 (11)
C23—P1—C11—C16	103.45 (12)	C17—P1—C23—C24	−68.86 (11)
Mo1—P1—C11—C16	−134.44 (10)	C11—P1—C23—C24	−175.97 (10)
C17—P1—C11—C12	175.23 (11)	Mo1—P1—C23—C24	60.46 (11)
C23—P1—C11—C12	−77.66 (12)	C28—C23—C24—C25	1.1 (2)
Mo1—P1—C11—C12	44.44 (12)	P1—C23—C24—C25	−172.08 (11)
C16—C11—C12—C13	0.2 (2)	C23—C24—C25—C26	−0.2 (2)
P1—C11—C12—C13	−178.74 (12)	C24—C25—C26—C27	−0.7 (2)
C11—C12—C13—C14	0.5 (2)	C25—C26—C27—C28	0.8 (2)
C12—C13—C14—C15	−0.6 (2)	C24—C23—C28—C27	−1.1 (2)
C13—C14—C15—C16	0.0 (2)	P1—C23—C28—C27	171.76 (11)
C12—C11—C16—C15	−0.8 (2)	C26—C27—C28—C23	0.1 (2)
P1—C11—C16—C15	178.09 (10)		

Hydrogen-bond geometry (Å, °) for (I)

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C12—H12 $\cdots$ O2 ⁱ	0.95	2.36	3.1188 (18)	137
C6—H6 $\cdots$ O1 ⁱⁱ	1.00	2.60	3.545 (2)	158

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

(2)

Crystal data

$\text{C}_{28}\text{H}_{22}\text{F}_3\text{MoO}_3\text{P}$
 $M_r = 590.36$
 Monoclinic, $P2_1/c$
 $a = 11.7991$ (4) Å
 $b = 18.6907$ (8) Å
 $c = 22.4744$ (8) Å
 $\beta = 97.256$ (2)°
 $V = 4916.7$ (3) Å³
 $Z = 8$

$F(000) = 2384$
 $D_x = 1.595$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 9718 reflections
 $\theta = 2.3\text{--}30.5^\circ$
 $\mu = 0.65$ mm⁻¹
 $T = 170$ K
 Prism, yellow
 $0.05 \times 0.05 \times 0.05$ mm

Data collection

Bruker D8 QUEST ECO
 diffractometer
 Radiation source: sealed tube, Siemens
 KFFMO2K-90C
 Curved Graphite monochromator
 Detector resolution: 7.3910 pixels mm⁻¹
 φ and ω scans
 Absorption correction: multi-scan
 (Krause *et al.*, 2015)

$T_{\min} = 0.86$, $T_{\max} = 0.97$
 73758 measured reflections
 10047 independent reflections
 8547 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$
 $\theta_{\max} = 26.4^\circ$, $\theta_{\min} = 2.3^\circ$
 $h = -14 \rightarrow 14$
 $k = -23 \rightarrow 23$
 $l = -28 \rightarrow 27$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.063$
 $S = 1.07$
 10047 reflections
 651 parameters
 0 restraints
 Primary atom site location: dual

Secondary atom site location: difference Fourier map
 Hydrogen site location: inferred from neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0211P)^2 + 4.4865P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 0.37$ e Å⁻³
 $\Delta\rho_{\min} = -0.39$ e Å⁻³

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²) for (2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Mo1	0.68359 (2)	0.16744 (2)	0.31097 (2)	0.01937 (5)
P1	0.50728 (5)	0.18602 (3)	0.35791 (3)	0.01993 (12)
F1	0.54908 (16)	0.08511 (12)	0.61078 (7)	0.0637 (6)
F2	0.08121 (14)	0.03649 (10)	0.25717 (8)	0.0533 (5)
F3	0.36509 (14)	0.49064 (7)	0.35239 (8)	0.0441 (4)
O1	0.51071 (16)	0.18266 (10)	0.19490 (8)	0.0399 (5)
O2	0.74633 (16)	0.31199 (10)	0.37628 (8)	0.0361 (4)
O3	0.88132 (15)	0.22239 (10)	0.24695 (8)	0.0333 (4)
C1	0.5734 (2)	0.18193 (12)	0.23876 (11)	0.0256 (5)
C2	0.71837 (19)	0.25955 (13)	0.35133 (10)	0.0243 (5)
C3	0.7827 (2)	0.23663 (13)	0.25283 (10)	0.0248 (5)

C4	0.7294 (2)	0.30324 (14)	0.22084 (12)	0.0346 (6)
H4A	0.66605	0.28786	0.190403	0.041*
H4B	0.696117	0.333204	0.250547	0.041*
C5	0.8111 (3)	0.34882 (15)	0.19021 (13)	0.0409 (7)
H5A	0.84251	0.32037	0.159538	0.061*
H5B	0.873425	0.365392	0.219977	0.061*
H5C	0.77008	0.3902	0.171316	0.061*
C6	0.6987 (3)	0.04365 (14)	0.29401 (13)	0.0414 (7)
H6	0.647429	0.014715	0.264404	0.05*
C7	0.6872 (2)	0.05233 (13)	0.35502 (13)	0.0345 (6)
H7	0.625273	0.031156	0.376022	0.041*
C8	0.7818 (2)	0.09086 (14)	0.38260 (12)	0.0345 (6)
H8	0.799791	0.100797	0.426502	0.041*
C9	0.8523 (2)	0.10673 (15)	0.33829 (12)	0.0368 (6)
H9	0.930101	0.128742	0.345501	0.044*
C10	0.8008 (3)	0.07731 (15)	0.28325 (13)	0.0421 (7)
H10	0.835835	0.075189	0.245032	0.05*
C11	0.5180 (2)	0.15457 (12)	0.43586 (10)	0.0236 (5)
C12	0.6089 (2)	0.17724 (15)	0.47718 (12)	0.0350 (6)
H12	0.664562	0.208646	0.464597	0.042*
C13	0.6194 (2)	0.15470 (18)	0.53629 (12)	0.0435 (7)
H13	0.680534	0.171027	0.564616	0.052*
C14	0.5390 (2)	0.10810 (17)	0.55288 (12)	0.0408 (7)
C15	0.4497 (2)	0.08329 (14)	0.51394 (12)	0.0354 (6)
H15	0.395996	0.050678	0.526812	0.043*
C16	0.4392 (2)	0.10706 (13)	0.45480 (11)	0.0278 (5)
H16	0.377406	0.090595	0.426987	0.033*
C17	0.3749 (2)	0.14313 (12)	0.32379 (10)	0.0238 (5)
C18	0.3798 (2)	0.07402 (13)	0.30120 (11)	0.0312 (6)
H18	0.451868	0.051639	0.300493	0.037*
C19	0.2808 (2)	0.03741 (15)	0.27971 (12)	0.0363 (6)
H19	0.284022	−0.010424	0.265731	0.044*
C20	0.1781 (2)	0.07191 (16)	0.27914 (12)	0.0360 (6)
C21	0.1688 (2)	0.14034 (15)	0.29969 (11)	0.0321 (6)
H21	0.096469	0.163162	0.297831	0.039*
C22	0.2682 (2)	0.17545 (13)	0.32338 (11)	0.0275 (5)
H22	0.263447	0.222164	0.33953	0.033*
C23	0.46301 (18)	0.27942 (12)	0.36021 (10)	0.0212 (5)
C24	0.4234 (2)	0.31163 (12)	0.30539 (11)	0.0270 (5)
H24	0.421085	0.284722	0.269401	0.032*
C25	0.3877 (2)	0.38197 (13)	0.30282 (12)	0.0318 (6)
H25	0.358032	0.403262	0.265723	0.038*
C26	0.3961 (2)	0.42019 (12)	0.35498 (13)	0.0308 (6)
C27	0.4348 (2)	0.39122 (13)	0.40983 (12)	0.0306 (6)
H27	0.439161	0.419233	0.445318	0.037*
C28	0.4673 (2)	0.31977 (13)	0.41208 (11)	0.0255 (5)
H28	0.492908	0.298322	0.449712	0.031*
Mo2	0.13778 (2)	0.20503 (2)	0.57103 (2)	0.01918 (5)
P2	0.07626 (5)	0.32692 (3)	0.53987 (3)	0.01888 (12)
F4	−0.41095 (12)	0.38483 (9)	0.56536 (7)	0.0443 (4)
F5	0.36489 (13)	0.55112 (8)	0.67019 (7)	0.0405 (4)

F6	0.08738 (15)	0.40659 (9)	0.28469 (7)	0.0483 (4)
O4	−0.11576 (15)	0.17823 (10)	0.51826 (9)	0.0389 (4)
O5	0.28370 (16)	0.23246 (10)	0.46697 (9)	0.0408 (5)
O6	0.1843 (2)	0.04766 (10)	0.54648 (9)	0.0584 (6)
C29	−0.0209 (2)	0.18824 (12)	0.53604 (11)	0.0250 (5)
C30	0.2268 (2)	0.22200 (12)	0.50419 (11)	0.0261 (5)
C31	0.1403 (2)	0.09868 (12)	0.52100 (11)	0.0265 (5)
C32	0.0920 (3)	0.08959 (15)	0.45594 (12)	0.0392 (7)
H32A	0.008542	0.097943	0.452191	0.047*
H32B	0.125178	0.127064	0.43224	0.047*
C33	0.1125 (3)	0.01792 (15)	0.42855 (13)	0.0441 (7)
H33A	0.073311	0.016208	0.387439	0.066*
H33B	0.082777	−0.02003	0.452393	0.066*
H33C	0.194657	0.010948	0.428005	0.066*
C34	0.2910 (2)	0.21959 (17)	0.64802 (12)	0.0412 (7)
H34	0.365723	0.243055	0.64373	0.049*
C35	0.1968 (3)	0.25274 (16)	0.66799 (11)	0.0433 (7)
H35	0.192359	0.30424	0.679536	0.052*
C36	0.1135 (3)	0.20086 (17)	0.67425 (11)	0.0408 (7)
H36	0.040367	0.208562	0.691483	0.049*
C37	0.1563 (2)	0.13425 (15)	0.65645 (11)	0.0373 (6)
H37	0.120101	0.086396	0.660455	0.045*
C38	0.2670 (2)	0.14677 (15)	0.64067 (11)	0.0358 (6)
H38	0.322423	0.109088	0.631453	0.043*
C39	−0.07161 (19)	0.35126 (12)	0.54894 (10)	0.0202 (5)
C40	−0.1144 (2)	0.33622 (13)	0.60274 (11)	0.0293 (5)
H40	−0.064987	0.31691	0.63546	0.035*
C41	−0.2281 (2)	0.34908 (14)	0.60907 (11)	0.0314 (6)
H41	−0.256614	0.339932	0.646011	0.038*
C42	−0.2982 (2)	0.37531 (13)	0.56070 (12)	0.0290 (5)
C43	−0.2598 (2)	0.39207 (14)	0.50748 (11)	0.0314 (6)
H43	−0.309978	0.411534	0.475153	0.038*
C44	−0.1453 (2)	0.37987 (13)	0.50190 (11)	0.0264 (5)
H44	−0.11709	0.391315	0.465296	0.032*
C45	0.16211 (19)	0.39779 (12)	0.57976 (10)	0.0221 (5)
C46	0.1136 (2)	0.45612 (12)	0.60505 (10)	0.0243 (5)
H46	0.032784	0.460151	0.601914	0.029*
C47	0.1819 (2)	0.50884 (13)	0.63496 (11)	0.0279 (5)
H47	0.148881	0.548962	0.652204	0.034*
C48	0.2979 (2)	0.50132 (13)	0.63887 (11)	0.0285 (5)
C49	0.3500 (2)	0.44497 (13)	0.61377 (11)	0.0298 (6)
H49	0.430847	0.44186	0.616714	0.036*
C50	0.2813 (2)	0.39324 (13)	0.58426 (11)	0.0274 (5)
H50	0.315441	0.35379	0.566651	0.033*
C51	0.08310 (18)	0.35142 (12)	0.46138 (10)	0.0204 (5)
C52	0.1267 (2)	0.41698 (12)	0.44605 (11)	0.0250 (5)
H52	0.155534	0.449472	0.476796	0.03*
C53	0.1287 (2)	0.43575 (14)	0.38613 (11)	0.0297 (5)
H53	0.158918	0.480455	0.375637	0.036*
C54	0.0860 (2)	0.38815 (15)	0.34296 (11)	0.0316 (6)
C55	0.0410 (2)	0.32290 (14)	0.35584 (11)	0.0322 (6)

H55	0.011264	0.291174	0.324697	0.039*
C56	0.0404 (2)	0.30477 (13)	0.41563 (11)	0.0269 (5)
H56	0.010257	0.259799	0.425552	0.032*

Atomic displacement parameters ($\text{\AA}^2$) for (2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.02038 (10)	0.01882 (10)	0.01827 (10)	0.00364 (8)	−0.00004 (7)	−0.00050 (7)
P1	0.0203 (3)	0.0190 (3)	0.0200 (3)	0.0007 (2)	0.0005 (2)	0.0010 (2)
F1	0.0570 (12)	0.1040 (16)	0.0297 (9)	−0.0035 (11)	0.0034 (8)	0.0313 (10)
F2	0.0360 (9)	0.0682 (12)	0.0554 (11)	−0.0274 (9)	0.0049 (8)	−0.0178 (9)
F3	0.0454 (10)	0.0205 (7)	0.0669 (11)	0.0049 (7)	0.0087 (8)	−0.0037 (7)
O1	0.0374 (11)	0.0506 (12)	0.0284 (10)	0.0065 (9)	−0.0093 (8)	−0.0035 (9)
O2	0.0375 (10)	0.0331 (10)	0.0377 (10)	−0.0074 (8)	0.0049 (8)	−0.0128 (8)
O3	0.0276 (9)	0.0392 (10)	0.0341 (10)	0.0025 (8)	0.0079 (8)	0.0000 (8)
C1	0.0268 (12)	0.0247 (12)	0.0254 (12)	0.0038 (10)	0.0037 (10)	−0.0031 (10)
C2	0.0199 (11)	0.0290 (13)	0.0243 (12)	−0.0001 (10)	0.0042 (9)	0.0002 (10)
C3	0.0287 (13)	0.0271 (12)	0.0183 (11)	−0.0015 (10)	0.0015 (10)	−0.0039 (9)
C4	0.0399 (15)	0.0330 (14)	0.0316 (14)	0.0057 (12)	0.0075 (12)	0.0039 (11)
C5	0.0494 (17)	0.0356 (15)	0.0381 (16)	−0.0021 (13)	0.0073 (13)	0.0064 (12)
C6	0.0561 (19)	0.0191 (12)	0.0470 (17)	0.0114 (12)	−0.0009 (14)	−0.0039 (12)
C7	0.0371 (15)	0.0207 (12)	0.0456 (16)	0.0074 (11)	0.0049 (12)	0.0096 (11)
C8	0.0336 (14)	0.0354 (14)	0.0326 (14)	0.0134 (12)	−0.0024 (11)	0.0123 (12)
C9	0.0254 (13)	0.0428 (15)	0.0415 (16)	0.0161 (12)	0.0012 (12)	0.0133 (13)
C10	0.0544 (18)	0.0326 (14)	0.0414 (16)	0.0265 (14)	0.0146 (14)	0.0023 (12)
C11	0.0242 (12)	0.0254 (12)	0.0216 (11)	0.0050 (10)	0.0042 (9)	0.0033 (9)
C12	0.0266 (13)	0.0496 (16)	0.0284 (13)	−0.0042 (12)	0.0019 (11)	0.0058 (12)
C13	0.0318 (15)	0.071 (2)	0.0255 (14)	−0.0025 (14)	−0.0042 (11)	0.0077 (14)
C14	0.0394 (16)	0.0586 (19)	0.0249 (14)	0.0102 (14)	0.0065 (12)	0.0149 (13)
C15	0.0370 (15)	0.0355 (14)	0.0356 (15)	0.0022 (12)	0.0118 (12)	0.0130 (12)
C16	0.0280 (13)	0.0244 (12)	0.0311 (13)	0.0041 (10)	0.0048 (10)	0.0020 (10)
C17	0.0244 (12)	0.0249 (12)	0.0215 (12)	−0.0048 (10)	0.0007 (9)	0.0028 (9)
C18	0.0315 (14)	0.0293 (13)	0.0322 (14)	−0.0013 (11)	0.0019 (11)	−0.0038 (11)
C19	0.0417 (16)	0.0345 (14)	0.0324 (14)	−0.0108 (12)	0.0038 (12)	−0.0077 (12)
C20	0.0312 (14)	0.0508 (17)	0.0261 (13)	−0.0182 (13)	0.0040 (11)	−0.0026 (12)
C21	0.0244 (13)	0.0432 (15)	0.0292 (14)	−0.0054 (11)	0.0052 (10)	0.0013 (12)
C22	0.0278 (13)	0.0289 (13)	0.0260 (12)	−0.0027 (10)	0.0040 (10)	0.0026 (10)
C23	0.0159 (10)	0.0212 (11)	0.0263 (12)	−0.0006 (9)	0.0014 (9)	0.0002 (9)
C24	0.0317 (13)	0.0225 (12)	0.0260 (12)	0.0020 (10)	0.0004 (10)	−0.0006 (10)
C25	0.0342 (14)	0.0243 (12)	0.0355 (14)	0.0018 (11)	−0.0012 (11)	0.0053 (11)
C26	0.0216 (12)	0.0187 (11)	0.0526 (17)	0.0007 (10)	0.0071 (11)	−0.0025 (11)
C27	0.0273 (13)	0.0311 (13)	0.0345 (14)	−0.0038 (11)	0.0084 (11)	−0.0106 (11)
C28	0.0223 (12)	0.0289 (12)	0.0255 (12)	−0.0004 (10)	0.0039 (10)	−0.0013 (10)
Mo2	0.01832 (10)	0.02103 (10)	0.01787 (10)	0.00077 (8)	0.00110 (7)	0.00333 (8)
P2	0.0180 (3)	0.0205 (3)	0.0182 (3)	0.0014 (2)	0.0026 (2)	0.0011 (2)
F4	0.0207 (7)	0.0672 (11)	0.0468 (10)	0.0075 (7)	0.0113 (7)	−0.0050 (8)
F5	0.0333 (8)	0.0364 (8)	0.0515 (10)	−0.0146 (7)	0.0046 (7)	−0.0119 (7)
F6	0.0641 (11)	0.0604 (11)	0.0221 (8)	0.0108 (9)	0.0129 (8)	0.0110 (7)
O4	0.0235 (10)	0.0388 (11)	0.0529 (12)	−0.0036 (8)	−0.0007 (9)	−0.0067 (9)
O5	0.0381 (11)	0.0421 (11)	0.0470 (12)	0.0118 (9)	0.0244 (9)	0.0136 (9)
O6	0.0987 (19)	0.0270 (10)	0.0434 (12)	0.0110 (11)	−0.0143 (12)	0.0041 (9)

C29	0.0267 (13)	0.0209 (11)	0.0277 (12)	0.0018 (10)	0.0050 (10)	0.0014 (10)
C30	0.0238 (12)	0.0227 (12)	0.0314 (13)	0.0052 (10)	0.0019 (11)	0.0025 (10)
C31	0.0251 (12)	0.0227 (12)	0.0316 (13)	−0.0020 (10)	0.0035 (10)	0.0035 (10)
C32	0.0474 (17)	0.0379 (15)	0.0309 (14)	0.0088 (13)	−0.0011 (12)	−0.0035 (12)
C33	0.0590 (19)	0.0355 (15)	0.0383 (16)	−0.0063 (14)	0.0078 (14)	−0.0056 (13)
C34	0.0361 (15)	0.0618 (19)	0.0225 (13)	−0.0127 (14)	−0.0090 (11)	0.0028 (13)
C35	0.073 (2)	0.0383 (15)	0.0160 (12)	0.0004 (15)	−0.0068 (13)	−0.0004 (11)
C36	0.0420 (16)	0.0634 (19)	0.0181 (12)	0.0140 (15)	0.0072 (11)	0.0112 (13)
C37	0.0483 (17)	0.0379 (15)	0.0239 (13)	−0.0049 (13)	−0.0024 (12)	0.0147 (11)
C38	0.0373 (15)	0.0450 (16)	0.0229 (13)	0.0143 (13)	−0.0048 (11)	0.0066 (11)
C39	0.0201 (11)	0.0202 (11)	0.0200 (11)	0.0019 (9)	0.0009 (9)	−0.0004 (9)
C40	0.0292 (13)	0.0348 (14)	0.0243 (12)	0.0049 (11)	0.0045 (10)	0.0052 (11)
C41	0.0308 (13)	0.0384 (14)	0.0274 (13)	0.0050 (11)	0.0123 (11)	0.0046 (11)
C42	0.0196 (12)	0.0323 (13)	0.0363 (14)	0.0009 (10)	0.0082 (10)	−0.0070 (11)
C43	0.0233 (12)	0.0424 (15)	0.0276 (13)	0.0074 (11)	−0.0004 (10)	0.0001 (11)
C44	0.0233 (12)	0.0347 (13)	0.0216 (12)	0.0027 (10)	0.0044 (10)	0.0017 (10)
C45	0.0236 (12)	0.0235 (11)	0.0189 (11)	−0.0017 (9)	0.0010 (9)	0.0028 (9)
C46	0.0210 (11)	0.0263 (12)	0.0248 (12)	0.0019 (10)	0.0005 (9)	0.0010 (10)
C47	0.0299 (13)	0.0233 (12)	0.0311 (13)	0.0022 (10)	0.0057 (11)	−0.0022 (10)
C48	0.0283 (13)	0.0256 (12)	0.0305 (13)	−0.0086 (10)	−0.0003 (10)	0.0010 (10)
C49	0.0208 (12)	0.0305 (13)	0.0383 (15)	−0.0027 (10)	0.0046 (11)	0.0028 (11)
C50	0.0223 (12)	0.0277 (12)	0.0329 (13)	0.0021 (10)	0.0063 (10)	−0.0027 (10)
C51	0.0187 (11)	0.0242 (11)	0.0183 (11)	0.0051 (9)	0.0029 (9)	0.0011 (9)
C52	0.0250 (12)	0.0252 (12)	0.0254 (12)	0.0026 (10)	0.0053 (10)	0.0015 (10)
C53	0.0299 (13)	0.0314 (13)	0.0295 (13)	0.0052 (11)	0.0109 (11)	0.0076 (11)
C54	0.0319 (14)	0.0434 (15)	0.0215 (12)	0.0143 (12)	0.0115 (10)	0.0088 (11)
C55	0.0360 (14)	0.0364 (14)	0.0232 (12)	0.0096 (12)	0.0001 (11)	−0.0033 (11)
C56	0.0307 (13)	0.0244 (12)	0.0253 (12)	0.0033 (10)	0.0023 (10)	0.0014 (10)

Geometric parameters (Å, °) for (2)

Mo1—P1	2.4730 (6)	Mo2—C29	1.962 (2)
Mo1—C1	1.966 (2)	Mo2—C30	1.964 (3)
Mo1—C2	1.965 (2)	Mo2—C31	2.286 (2)
Mo1—C3	2.265 (2)	Mo2—C38	2.315 (2)
Mo1—C9	2.306 (2)	Mo2—C37	2.319 (2)
Mo1—C10	2.314 (2)	Mo2—C34	2.355 (3)
Mo1—C8	2.348 (2)	Mo2—C36	2.374 (3)
Mo1—C6	2.355 (3)	Mo2—C35	2.376 (3)
Mo1—C7	2.366 (2)	Mo2—P2	2.4654 (6)
P1—C23	1.825 (2)	P2—C45	1.832 (2)
P1—C17	1.835 (2)	P2—C51	1.834 (2)
P1—C11	1.837 (2)	P2—C39	1.839 (2)
F1—C14	1.361 (3)	F4—C42	1.359 (3)
F2—C20	1.359 (3)	F5—C48	1.359 (3)
F3—C26	1.366 (3)	F6—C54	1.356 (3)
O1—C1	1.155 (3)	O4—C29	1.155 (3)
O2—C2	1.156 (3)	O5—C30	1.154 (3)
O3—C3	1.218 (3)	O6—C31	1.196 (3)
C3—C4	1.532 (3)	C31—C32	1.511 (3)
C4—C5	1.515 (4)	C32—C33	1.506 (4)

C4—H4A	0.99	C32—H32A	0.99
C4—H4B	0.99	C32—H32B	0.99
C5—H5A	0.98	C33—H33A	0.98
C5—H5B	0.98	C33—H33B	0.98
C5—H5C	0.98	C33—H33C	0.98
C6—C7	1.404 (4)	C34—C35	1.396 (4)
C6—C10	1.406 (4)	C34—C38	1.396 (4)
C6—H6	1.0	C34—H34	1.0
C7—C8	1.405 (4)	C35—C36	1.400 (4)
C7—H7	1.0	C35—H35	1.0
C8—C9	1.408 (4)	C36—C37	1.420 (4)
C8—H8	1.0	C36—H36	1.0
C9—C10	1.419 (4)	C37—C38	1.416 (4)
C9—H9	1.0	C37—H37	1.0
C10—H10	1.0	C38—H38	1.0
C11—C16	1.391 (3)	C39—C44	1.388 (3)
C11—C12	1.393 (3)	C39—C40	1.397 (3)
C12—C13	1.384 (4)	C40—C41	1.388 (3)
C12—H12	0.95	C40—H40	0.95
C13—C14	1.373 (4)	C41—C42	1.371 (4)
C13—H13	0.95	C41—H41	0.95
C14—C15	1.364 (4)	C42—C43	1.368 (3)
C15—C16	1.392 (3)	C43—C44	1.391 (3)
C15—H15	0.95	C43—H43	0.95
C16—H16	0.95	C44—H44	0.95
C17—C18	1.392 (3)	C45—C46	1.386 (3)
C17—C22	1.395 (3)	C45—C50	1.400 (3)
C18—C19	1.387 (4)	C46—C47	1.391 (3)
C18—H18	0.95	C46—H46	0.95
C19—C20	1.371 (4)	C47—C48	1.367 (3)
C19—H19	0.95	C47—H47	0.95
C20—C21	1.369 (4)	C48—C49	1.376 (3)
C21—C22	1.390 (3)	C49—C50	1.376 (3)
C21—H21	0.95	C49—H49	0.95
C22—H22	0.95	C50—H50	0.95
C23—C28	1.384 (3)	C51—C52	1.389 (3)
C23—C24	1.398 (3)	C51—C56	1.393 (3)
C24—C25	1.379 (3)	C52—C53	1.395 (3)
C24—H24	0.95	C52—H52	0.95
C25—C26	1.366 (4)	C53—C54	1.365 (4)
C25—H25	0.95	C53—H53	0.95
C26—C27	1.371 (4)	C54—C55	1.375 (4)
C27—C28	1.389 (3)	C55—C56	1.387 (3)
C27—H27	0.95	C55—H55	0.95
C28—H28	0.95	C56—H56	0.95
C1—Mo1—C2	109.77 (10)	C29—Mo2—C30	107.12 (10)
C1—Mo1—C3	77.34 (9)	C29—Mo2—C31	74.64 (9)
C2—Mo1—C3	70.93 (9)	C30—Mo2—C31	73.89 (9)
C2—Mo1—C9	100.67 (10)	C29—Mo2—C38	135.20 (10)
C1—Mo1—C9	138.27 (10)	C30—Mo2—C38	102.87 (10)

C3—Mo1—C9	86.76 (9)	C31—Mo2—C38	82.81 (9)
C2—Mo1—C10	131.68 (11)	C29—Mo2—C37	103.05 (10)
C1—Mo1—C10	103.50 (10)	C30—Mo2—C37	136.20 (10)
C3—Mo1—C10	83.74 (10)	C31—Mo2—C37	84.57 (9)
C9—Mo1—C10	35.76 (10)	C38—Mo2—C37	35.59 (10)
C2—Mo1—C8	99.28 (10)	C29—Mo2—C34	156.64 (10)
C1—Mo1—C8	150.20 (10)	C30—Mo2—C34	96.22 (10)
C3—Mo1—C8	119.84 (9)	C31—Mo2—C34	113.90 (10)
C9—Mo1—C8	35.20 (9)	C38—Mo2—C34	34.77 (10)
C10—Mo1—C8	58.73 (10)	C37—Mo2—C34	58.30 (10)
C2—Mo1—C6	156.88 (10)	C29—Mo2—C36	99.19 (10)
C1—Mo1—C6	93.30 (10)	C30—Mo2—C36	153.46 (10)
C3—Mo1—C6	114.34 (10)	C31—Mo2—C36	117.57 (10)
C9—Mo1—C6	58.62 (11)	C38—Mo2—C36	58.30 (10)
C10—Mo1—C6	35.04 (10)	C37—Mo2—C36	35.19 (10)
C8—Mo1—C6	58.07 (10)	C34—Mo2—C36	57.46 (10)
C2—Mo1—C7	127.71 (10)	C29—Mo2—C35	125.58 (11)
C1—Mo1—C7	116.47 (10)	C30—Mo2—C35	121.19 (11)
C3—Mo1—C7	140.99 (9)	C31—Mo2—C35	139.20 (9)
C9—Mo1—C7	58.14 (10)	C38—Mo2—C35	57.64 (10)
C10—Mo1—C7	58.07 (10)	C37—Mo2—C35	57.91 (10)
C8—Mo1—C7	34.68 (9)	C34—Mo2—C35	34.31 (10)
C6—Mo1—C7	34.61 (10)	C36—Mo2—C35	34.28 (10)
C1—Mo1—P1	80.12 (7)	C29—Mo2—P2	78.59 (7)
C2—Mo1—P1	79.50 (7)	C30—Mo2—P2	78.48 (7)
C3—Mo1—P1	133.55 (6)	C31—Mo2—P2	133.28 (6)
C9—Mo1—P1	134.42 (7)	C38—Mo2—P2	140.53 (7)
C10—Mo1—P1	141.31 (8)	C37—Mo2—P2	139.10 (7)
C8—Mo1—P1	99.33 (7)	C34—Mo2—P2	105.85 (8)
C6—Mo1—P1	106.98 (8)	C36—Mo2—P2	103.91 (7)
C7—Mo1—P1	85.46 (7)	C35—Mo2—P2	87.44 (7)
C23—P1—C17	101.43 (10)	C45—P2—C51	101.62 (10)
C23—P1—C11	105.34 (11)	C45—P2—C39	103.92 (10)
C17—P1—C11	102.19 (11)	C51—P2—C39	101.68 (10)
C23—P1—Mo1	113.94 (7)	C45—P2—Mo2	113.86 (7)
C17—P1—Mo1	118.55 (8)	C51—P2—Mo2	117.18 (7)
C11—P1—Mo1	113.67 (8)	C39—P2—Mo2	116.52 (7)
O1—C1—Mo1	172.5 (2)	O4—C29—Mo2	176.6 (2)
O2—C2—Mo1	175.3 (2)	O5—C30—Mo2	176.6 (2)
O3—C3—C4	117.9 (2)	O6—C31—C32	117.5 (2)
O3—C3—Mo1	120.52 (18)	O6—C31—Mo2	119.32 (19)
C4—C3—Mo1	121.58 (17)	C32—C31—Mo2	123.17 (17)
C5—C4—C3	115.1 (2)	C33—C32—C31	115.8 (2)
C5—C4—H4A	108.5	C33—C32—H32A	108.3
C3—C4—H4A	108.5	C31—C32—H32A	108.3
C5—C4—H4B	108.5	C33—C32—H32B	108.3
C3—C4—H4B	108.5	C31—C32—H32B	108.3
H4A—C4—H4B	107.5	H32A—C32—H32B	107.4
C4—C5—H5A	109.5	C32—C33—H33A	109.5
C4—C5—H5B	109.5	C32—C33—H33B	109.5
H5A—C5—H5B	109.5	H33A—C33—H33B	109.5

C4—C5—H5C	109.5	C32—C33—H33C	109.5
H5A—C5—H5C	109.5	H33A—C33—H33C	109.5
H5B—C5—H5C	109.5	H33B—C33—H33C	109.5
C7—C6—C10	107.9 (3)	C35—C34—C38	108.2 (3)
C7—C6—Mo1	73.13 (14)	C35—C34—Mo2	73.66 (16)
C10—C6—Mo1	70.88 (15)	C38—C34—Mo2	71.04 (14)
C7—C6—H6	125.9	C35—C34—H34	125.7
C10—C6—H6	125.9	C38—C34—H34	125.7
Mo1—C6—H6	125.9	Mo2—C34—H34	125.7
C6—C7—C8	108.7 (3)	C34—C35—C36	108.8 (3)
C6—C7—Mo1	72.26 (14)	C34—C35—Mo2	72.03 (15)
C8—C7—Mo1	71.94 (14)	C36—C35—Mo2	72.80 (15)
C6—C7—H7	125.6	C34—C35—H35	125.5
C8—C7—H7	125.6	C36—C35—H35	125.5
Mo1—C7—H7	125.6	Mo2—C35—H35	125.5
C7—C8—C9	107.7 (3)	C35—C36—C37	107.5 (3)
C7—C8—Mo1	73.38 (14)	C35—C36—Mo2	72.92 (15)
C9—C8—Mo1	70.80 (14)	C37—C36—Mo2	70.28 (14)
C7—C8—H8	126.0	C35—C36—H36	126.2
C9—C8—H8	126.0	C37—C36—H36	126.2
Mo1—C8—H8	126.0	Mo2—C36—H36	126.2
C8—C9—C10	108.0 (3)	C38—C37—C36	107.3 (3)
C8—C9—Mo1	74.01 (14)	C38—C37—Mo2	72.04 (14)
C10—C9—Mo1	72.42 (14)	C36—C37—Mo2	74.53 (14)
C8—C9—H9	125.8	C38—C37—H37	126.0
C10—C9—H9	125.8	C36—C37—H37	126.0
Mo1—C9—H9	125.8	Mo2—C37—H37	126.0
C6—C10—C9	107.8 (3)	C34—C38—C37	108.1 (3)
C6—C10—Mo1	74.08 (15)	C34—C38—Mo2	74.19 (15)
C9—C10—Mo1	71.82 (14)	C37—C38—Mo2	72.36 (14)
C6—C10—H10	125.9	C34—C38—H38	125.7
C9—C10—H10	125.9	C37—C38—H38	125.7
Mo1—C10—H10	125.9	Mo2—C38—H38	125.7
C16—C11—C12	118.6 (2)	C44—C39—C40	118.5 (2)
C16—C11—P1	121.88 (18)	C44—C39—P2	121.51 (17)
C12—C11—P1	119.53 (18)	C40—C39—P2	119.86 (17)
C13—C12—C11	121.0 (2)	C41—C40—C39	120.9 (2)
C13—C12—H12	119.5	C41—C40—H40	119.5
C11—C12—H12	119.5	C39—C40—H40	119.5
C14—C13—C12	118.2 (3)	C42—C41—C40	118.4 (2)
C14—C13—H13	120.9	C42—C41—H41	120.8
C12—C13—H13	120.9	C40—C41—H41	120.8
F1—C14—C15	118.2 (3)	F4—C42—C43	118.2 (2)
F1—C14—C13	118.7 (3)	F4—C42—C41	119.0 (2)
C15—C14—C13	123.1 (2)	C43—C42—C41	122.8 (2)
C14—C15—C16	118.2 (2)	C42—C43—C44	118.3 (2)
C14—C15—H15	120.9	C42—C43—H43	120.8
C16—C15—H15	120.9	C44—C43—H43	120.8
C11—C16—C15	121.0 (2)	C39—C44—C43	121.1 (2)
C11—C16—H16	119.5	C39—C44—H44	119.5
C15—C16—H16	119.5	C43—C44—H44	119.5

C18—C17—C22	118.6 (2)	C46—C45—C50	118.8 (2)
C18—C17—P1	119.04 (18)	C46—C45—P2	122.54 (18)
C22—C17—P1	122.22 (18)	C50—C45—P2	118.68 (18)
C19—C18—C17	120.9 (2)	C45—C46—C47	120.7 (2)
C19—C18—H18	119.6	C45—C46—H46	119.6
C17—C18—H18	119.6	C47—C46—H46	119.6
C20—C19—C18	118.4 (2)	C48—C47—C46	118.2 (2)
C20—C19—H19	120.8	C48—C47—H47	120.9
C18—C19—H19	120.8	C46—C47—H47	120.9
F2—C20—C21	118.6 (3)	F5—C48—C47	118.4 (2)
F2—C20—C19	118.4 (3)	F5—C48—C49	118.4 (2)
C21—C20—C19	123.0 (2)	C47—C48—C49	123.2 (2)
C20—C21—C22	118.1 (2)	C48—C49—C50	118.0 (2)
C20—C21—H21	121.0	C48—C49—H49	121.0
C22—C21—H21	121.0	C50—C49—H49	121.0
C21—C22—C17	121.0 (2)	C49—C50—C45	121.1 (2)
C21—C22—H22	119.5	C49—C50—H50	119.4
C17—C22—H22	119.5	C45—C50—H50	119.4
C28—C23—C24	118.7 (2)	C52—C51—C56	118.7 (2)
C28—C23—P1	124.40 (18)	C52—C51—P2	121.43 (17)
C24—C23—P1	116.89 (17)	C56—C51—P2	119.85 (17)
C25—C24—C23	120.9 (2)	C51—C52—C53	120.9 (2)
C25—C24—H24	119.5	C51—C52—H52	119.6
C23—C24—H24	119.5	C53—C52—H52	119.6
C26—C25—C24	118.3 (2)	C54—C53—C52	118.2 (2)
C26—C25—H25	120.9	C54—C53—H53	120.9
C24—C25—H25	120.9	C52—C53—H53	120.9
C25—C26—F3	118.5 (2)	F6—C54—C53	118.2 (2)
C25—C26—C27	123.0 (2)	F6—C54—C55	118.7 (2)
F3—C26—C27	118.4 (2)	C53—C54—C55	123.1 (2)
C26—C27—C28	118.1 (2)	C54—C55—C56	118.0 (2)
C26—C27—H27	120.9	C54—C55—H55	121.0
C28—C27—H27	120.9	C56—C55—H55	121.0
C23—C28—C27	120.8 (2)	C55—C56—C51	121.1 (2)
C23—C28—H28	119.6	C55—C56—H56	119.4
C27—C28—H28	119.6	C51—C56—H56	119.4
O3—C3—C4—C5	−6.1 (3)	O6—C31—C32—C33	−4.5 (4)
Mo1—C3—C4—C5	172.61 (18)	Mo2—C31—C32—C33	173.63 (19)
C10—C6—C7—C8	−0.4 (3)	C38—C34—C35—C36	0.9 (3)
Mo1—C6—C7—C8	−63.15 (17)	Mo2—C34—C35—C36	63.96 (19)
C10—C6—C7—Mo1	62.78 (18)	C38—C34—C35—Mo2	−63.05 (18)
C6—C7—C8—C9	0.4 (3)	C34—C35—C36—C37	−1.3 (3)
Mo1—C7—C8—C9	−62.91 (17)	Mo2—C35—C36—C37	62.17 (18)
C6—C7—C8—Mo1	63.36 (18)	C34—C35—C36—Mo2	−63.47 (18)
C7—C8—C9—C10	−0.4 (3)	C35—C36—C37—C38	1.2 (3)
Mo1—C8—C9—C10	−64.96 (17)	Mo2—C36—C37—C38	65.08 (17)
C7—C8—C9—Mo1	64.60 (17)	C35—C36—C37—Mo2	−63.90 (18)
C7—C6—C10—C9	0.1 (3)	C35—C34—C38—C37	−0.2 (3)
Mo1—C6—C10—C9	64.39 (17)	Mo2—C34—C38—C37	−64.91 (17)
C7—C6—C10—Mo1	−64.24 (18)	C35—C34—C38—Mo2	64.75 (19)

C8—C9—C10—C6	0.1 (3)	C36—C37—C38—C34	−0.6 (3)
Mo1—C9—C10—C6	−65.88 (18)	Mo2—C37—C38—C34	66.12 (18)
C8—C9—C10—Mo1	66.01 (18)	C36—C37—C38—Mo2	−66.75 (17)
C23—P1—C11—C16	108.3 (2)	C45—P2—C39—C44	105.6 (2)
C17—P1—C11—C16	2.7 (2)	C51—P2—C39—C44	0.4 (2)
Mo1—P1—C11—C16	−126.24 (18)	Mo2—P2—C39—C44	−128.27 (18)
C23—P1—C11—C12	−73.3 (2)	C45—P2—C39—C40	−79.0 (2)
C17—P1—C11—C12	−178.9 (2)	C51—P2—C39—C40	175.70 (19)
Mo1—P1—C11—C12	52.1 (2)	Mo2—P2—C39—C40	47.1 (2)
C16—C11—C12—C13	−1.7 (4)	C44—C39—C40—C41	0.4 (4)
P1—C11—C12—C13	179.8 (2)	P2—C39—C40—C41	−175.1 (2)
C11—C12—C13—C14	1.4 (4)	C39—C40—C41—C42	1.6 (4)
C12—C13—C14—F1	179.4 (3)	C40—C41—C42—F4	176.8 (2)
C12—C13—C14—C15	−0.1 (5)	C40—C41—C42—C43	−2.8 (4)
F1—C14—C15—C16	179.8 (2)	F4—C42—C43—C44	−177.7 (2)
C13—C14—C15—C16	−0.7 (4)	C41—C42—C43—C44	1.9 (4)
C12—C11—C16—C15	0.9 (4)	C40—C39—C44—C43	−1.3 (4)
P1—C11—C16—C15	179.29 (19)	P2—C39—C44—C43	174.10 (19)
C14—C15—C16—C11	0.3 (4)	C42—C43—C44—C39	0.2 (4)
C23—P1—C17—C18	164.88 (19)	C51—P2—C45—C46	101.2 (2)
C11—P1—C17—C18	−86.5 (2)	C39—P2—C45—C46	−4.1 (2)
Mo1—P1—C17—C18	39.3 (2)	Mo2—P2—C45—C46	−131.89 (18)
C23—P1—C17—C22	−19.6 (2)	C51—P2—C45—C50	−78.1 (2)
C11—P1—C17—C22	89.0 (2)	C39—P2—C45—C50	176.65 (19)
Mo1—P1—C17—C22	−145.17 (17)	Mo2—P2—C45—C50	48.9 (2)
C22—C17—C18—C19	−1.2 (4)	C50—C45—C46—C47	−0.7 (3)
P1—C17—C18—C19	174.5 (2)	P2—C45—C46—C47	−179.96 (18)
C17—C18—C19—C20	2.4 (4)	C45—C46—C47—C48	−0.2 (4)
C18—C19—C20—F2	178.9 (2)	C46—C47—C48—F5	−177.3 (2)
C18—C19—C20—C21	−1.1 (4)	C46—C47—C48—C49	1.2 (4)
F2—C20—C21—C22	178.5 (2)	F5—C48—C49—C50	177.4 (2)
C19—C20—C21—C22	−1.5 (4)	C47—C48—C49—C50	−1.2 (4)
C20—C21—C22—C17	2.8 (4)	C48—C49—C50—C45	0.1 (4)
C18—C17—C22—C21	−1.5 (4)	C46—C45—C50—C49	0.8 (4)
P1—C17—C22—C21	−176.99 (19)	P2—C45—C50—C49	−179.95 (19)
C17—P1—C23—C28	121.0 (2)	C45—P2—C51—C52	−10.9 (2)
C11—P1—C23—C28	14.8 (2)	C39—P2—C51—C52	96.2 (2)
Mo1—P1—C23—C28	−110.42 (19)	Mo2—P2—C51—C52	−135.61 (17)
C17—P1—C23—C24	−59.7 (2)	C45—P2—C51—C56	171.36 (19)
C11—P1—C23—C24	−165.84 (18)	C39—P2—C51—C56	−81.6 (2)
Mo1—P1—C23—C24	68.90 (19)	Mo2—P2—C51—C56	46.6 (2)
C28—C23—C24—C25	−0.9 (4)	C56—C51—C52—C53	−0.5 (3)
P1—C23—C24—C25	179.73 (19)	P2—C51—C52—C53	−178.30 (18)
C23—C24—C25—C26	2.5 (4)	C51—C52—C53—C54	0.4 (4)
C24—C25—C26—F3	177.5 (2)	C52—C53—C54—F6	179.7 (2)
C24—C25—C26—C27	−2.2 (4)	C52—C53—C54—C55	0.2 (4)
C25—C26—C27—C28	0.4 (4)	F6—C54—C55—C56	179.9 (2)
F3—C26—C27—C28	−179.3 (2)	C53—C54—C55—C56	−0.6 (4)
C24—C23—C28—C27	−1.0 (3)	C54—C55—C56—C51	0.5 (4)
P1—C23—C28—C27	178.31 (18)	C52—C51—C56—C55	0.1 (3)
C26—C27—C28—C23	1.3 (4)	P2—C51—C56—C55	177.88 (19)

Hydrogen-bond geometry (Å, °) for (2)

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C15—H15 $\cdots$ O6	0.95	2.59	3.371 (4)	139
C49—H49 $\cdots$ F4 ⁱ	0.95	2.55	3.344 (3)	141
C55—H55 $\cdots$ O3 ⁱⁱ	0.95	2.53	3.450 (3)	164
C34—H34 $\cdots$ O1 ⁱⁱⁱ	1.00	2.38	3.237 (3)	143

Symmetry codes: (i) $x+1, y, z$; (ii) $x-1, y, z$; (iii) $x, -y+1/2, z+1/2$.

(3)*Crystal data*

$\text{C}_{31}\text{H}_{31}\text{MoO}_6\text{P}\cdot\text{CH}_2\text{Cl}_2$

$M_r = 711.39$

Triclinic, $P\bar{1}$

$a = 10.5308$ (6) Å

$b = 12.1305$ (7) Å

$c = 13.6154$ (8) Å

$\alpha = 97.660$ (2)°

$\beta = 104.759$ (2)°

$\gamma = 107.081$ (2)°

$V = 1566.43$ (16) Å³

$Z = 2$

$F(000) = 728$

$D_x = 1.508$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9554 reflections

$\theta = 3.0\text{--}36.3^\circ$

$\mu = 0.68$ mm⁻¹

$T = 170$ K

Block, pale yellow

$0.23 \times 0.21 \times 0.12$ mm

Data collection

Bruker D8 QUEST ECO

diffractometer

Radiation source: sealed tube, Siemens

KFFMO2K-90C

Curved Graphite monochromator

Detector resolution: 7.3910 pixels mm⁻¹

ϕ and ω scans

Absorption correction: multi-scan

(Krause *et al.*, 2015)

$T_{\min} = 0.84$, $T_{\max} = 0.92$

81820 measured reflections

9578 independent reflections

9053 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$

$\theta_{\max} = 30.5^\circ$, $\theta_{\min} = 2.3^\circ$

$h = -15 \rightarrow 15$

$k = -17 \rightarrow 17$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.020$

$wR(F^2) = 0.054$

$S = 1.06$

9578 reflections

393 parameters

0 restraints

Primary atom site location: dual

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0226P)^2 + 0.7135P]$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.40$ e Å⁻³

$\Delta\rho_{\min} = -0.48$ e Å⁻³

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²) for (3)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Mo1	0.52042 (2)	0.25393 (2)	0.09427 (2)	0.01619 (3)	

P1	0.51816 (3)	0.35557 (2)	0.26360 (2)	0.01580 (5)
O1	0.68635 (12)	0.51311 (8)	0.09342 (8)	0.0368 (2)
O2	0.71687 (11)	0.15626 (9)	0.24292 (8)	0.0355 (2)
O3	0.65766 (10)	0.19845 (9)	−0.07044 (7)	0.0330 (2)
O4	0.07178 (10)	0.57231 (9)	0.24125 (8)	0.0335 (2)
O5	0.41490 (10)	0.06671 (8)	0.57748 (7)	0.03008 (19)
O6	1.00779 (9)	0.78117 (8)	0.52048 (7)	0.03004 (19)
C1	0.62823 (12)	0.41791 (10)	0.09761 (9)	0.0227 (2)
C2	0.64709 (12)	0.19679 (10)	0.19028 (8)	0.0220 (2)
C3	0.68821 (12)	0.24759 (10)	0.02074 (9)	0.0233 (2)
C4	0.84445 (13)	0.30859 (12)	0.07936 (10)	0.0301 (2)
H4A	0.878597	0.383496	0.057367	0.036*
H4B	0.857453	0.328655	0.154986	0.036*
C5	0.93197 (15)	0.23248 (14)	0.06042 (13)	0.0385 (3)
H5A	0.909437	0.164404	0.092354	0.058*
H5B	1.031399	0.27963	0.091479	0.058*
H5C	0.911323	0.204489	−0.01474	0.058*
C6	0.32457 (13)	0.07480 (10)	0.03752 (10)	0.0266 (2)
H6	0.313597	0.0047	0.069965	0.032*
C7	0.27424 (12)	0.16854 (10)	0.05911 (9)	0.0246 (2)
H7	0.222345	0.176265	0.110302	0.03*
C8	0.29947 (13)	0.24425 (11)	−0.00983 (10)	0.0272 (2)
H8	0.266202	0.312821	−0.017144	0.033*
C9	0.36793 (14)	0.19845 (12)	−0.07342 (9)	0.0309 (3)
H9	0.388632	0.227539	−0.13493	0.037*
C10	0.38374 (14)	0.09369 (11)	−0.04378 (10)	0.0306 (3)
H10	0.418501	0.037449	−0.080607	0.037*
C11	0.37443 (11)	0.41395 (9)	0.25087 (8)	0.01780 (18)
C12	0.35998 (12)	0.49063 (10)	0.18422 (9)	0.0226 (2)
H12	0.419737	0.505677	0.142103	0.027*
C13	0.26014 (12)	0.54555 (11)	0.17798 (9)	0.0237 (2)
H13	0.252561	0.598157	0.132747	0.028*
C14	0.17141 (12)	0.52242 (10)	0.23893 (9)	0.0227 (2)
C15	0.18237 (12)	0.44423 (11)	0.30402 (9)	0.0248 (2)
H15	0.120263	0.426933	0.344156	0.03*
C16	0.28358 (12)	0.39144 (10)	0.31053 (9)	0.02147 (19)
H16	0.291252	0.339271	0.356177	0.026*
C17	0.05341 (16)	0.65233 (14)	0.17571 (14)	0.0397 (3)
H17A	0.141582	0.718094	0.192016	0.06*
H17B	−0.018996	0.683173	0.187415	0.06*
H17C	0.025013	0.610782	0.102594	0.06*
C18	0.49824 (11)	0.26789 (9)	0.36153 (8)	0.01745 (17)
C19	0.40076 (12)	0.15263 (10)	0.33211 (9)	0.0235 (2)
H19	0.351772	0.118227	0.26037	0.028*
C20	0.37468 (13)	0.08818 (10)	0.40562 (9)	0.0253 (2)
H20	0.306101	0.011206	0.384356	0.03*
C21	0.44904 (12)	0.13610 (10)	0.51117 (8)	0.02071 (19)
C22	0.54956 (12)	0.24834 (10)	0.54166 (8)	0.02109 (19)
H22	0.602748	0.280472	0.612987	0.025*
C23	0.57210 (11)	0.31379 (10)	0.46696 (8)	0.02006 (19)
H23	0.639338	0.391388	0.488536	0.024*

C24	0.48069 (19)	0.11574 (13)	0.68676 (10)	0.0395 (3)	
H24A	0.443749	0.058545	0.725802	0.059*	
H24B	0.461623	0.188617	0.7061	0.059*	
H24C	0.581792	0.133654	0.703274	0.059*	
C25	0.66885 (11)	0.48601 (9)	0.33776 (8)	0.01716 (17)	
C26	0.65268 (12)	0.58564 (10)	0.39084 (9)	0.0221 (2)	
H26	0.561347	0.586707	0.38549	0.027*	
C27	0.76725 (12)	0.68248 (10)	0.45087 (9)	0.0241 (2)	
H27	0.75409	0.749483	0.485784	0.029*	
C28	0.90179 (12)	0.68197 (10)	0.46023 (8)	0.02111 (19)	
C29	0.92037 (12)	0.58325 (11)	0.40946 (9)	0.0246 (2)	
H29	1.011851	0.581542	0.416595	0.03*	
C30	0.80430 (12)	0.48711 (10)	0.34821 (9)	0.0231 (2)	
H30	0.817702	0.420618	0.312657	0.028*	
C31	1.14412 (14)	0.79549 (14)	0.51248 (11)	0.0366 (3)	
H31A	1.178982	0.737871	0.54421	0.055*	
H31B	1.207379	0.875805	0.548891	0.055*	
H31C	1.139505	0.782624	0.438867	0.055*	
C32	−0.0396 (2)	0.01902 (17)	0.26603 (16)	0.0533 (4)	
H32A	0.017643	0.101022	0.305243	0.064*	0.532 (15)
H32B	−0.133229	0.018994	0.22801	0.064*	0.532 (15)
H32C	−0.117475	0.043473	0.229775	0.064*	0.468 (15)
H32D	0.030037	0.088435	0.319629	0.064*	0.468 (15)
Cl1A	−0.0560 (9)	−0.0707 (4)	0.3518 (4)	0.0661 (11)	0.532 (15)
Cl1B	−0.1046 (5)	−0.0962 (5)	0.3280 (3)	0.0714 (6)	0.468 (15)
Cl2	0.03837 (4)	−0.02605 (4)	0.17571 (4)	0.05013 (10)	

Atomic displacement parameters ($\text{\AA}^2$) for (3)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.01913 (4)	0.01478 (4)	0.01330 (4)	0.00475 (3)	0.00476 (3)	0.00160 (3)
P1	0.01702 (11)	0.01515 (11)	0.01494 (11)	0.00486 (9)	0.00573 (9)	0.00235 (8)
O1	0.0471 (6)	0.0231 (4)	0.0411 (5)	0.0065 (4)	0.0188 (5)	0.0125 (4)
O2	0.0443 (6)	0.0374 (5)	0.0274 (4)	0.0239 (5)	0.0040 (4)	0.0076 (4)
O3	0.0351 (5)	0.0415 (5)	0.0210 (4)	0.0113 (4)	0.0121 (4)	−0.0003 (4)
O4	0.0338 (5)	0.0424 (5)	0.0407 (5)	0.0255 (4)	0.0204 (4)	0.0176 (4)
O5	0.0403 (5)	0.0263 (4)	0.0217 (4)	0.0052 (4)	0.0110 (4)	0.0102 (3)
O6	0.0227 (4)	0.0239 (4)	0.0318 (5)	−0.0006 (3)	0.0043 (3)	−0.0042 (3)
C1	0.0274 (5)	0.0218 (5)	0.0199 (5)	0.0083 (4)	0.0093 (4)	0.0050 (4)
C2	0.0271 (5)	0.0193 (5)	0.0183 (4)	0.0080 (4)	0.0068 (4)	0.0005 (4)
C3	0.0272 (5)	0.0224 (5)	0.0216 (5)	0.0083 (4)	0.0104 (4)	0.0039 (4)
C4	0.0255 (6)	0.0321 (6)	0.0292 (6)	0.0057 (5)	0.0119 (5)	−0.0018 (5)
C5	0.0319 (7)	0.0378 (7)	0.0445 (8)	0.0145 (6)	0.0096 (6)	0.0034 (6)
C6	0.0246 (5)	0.0185 (5)	0.0283 (6)	0.0014 (4)	0.0026 (4)	0.0013 (4)
C7	0.0198 (5)	0.0247 (5)	0.0238 (5)	0.0035 (4)	0.0037 (4)	0.0029 (4)
C8	0.0242 (5)	0.0274 (6)	0.0249 (5)	0.0074 (4)	0.0003 (4)	0.0068 (4)
C9	0.0310 (6)	0.0369 (7)	0.0157 (5)	0.0044 (5)	0.0017 (4)	0.0027 (4)
C10	0.0311 (6)	0.0263 (6)	0.0237 (5)	0.0042 (5)	0.0037 (5)	−0.0077 (4)
C11	0.0175 (4)	0.0184 (4)	0.0171 (4)	0.0056 (4)	0.0060 (3)	0.0027 (3)
C12	0.0228 (5)	0.0263 (5)	0.0234 (5)	0.0101 (4)	0.0114 (4)	0.0094 (4)
C13	0.0248 (5)	0.0258 (5)	0.0250 (5)	0.0113 (4)	0.0102 (4)	0.0097 (4)

C14	0.0203 (5)	0.0247 (5)	0.0244 (5)	0.0098 (4)	0.0076 (4)	0.0038 (4)
C15	0.0238 (5)	0.0309 (6)	0.0254 (5)	0.0115 (4)	0.0133 (4)	0.0091 (4)
C16	0.0219 (5)	0.0245 (5)	0.0206 (5)	0.0084 (4)	0.0093 (4)	0.0075 (4)
C17	0.0368 (7)	0.0399 (7)	0.0557 (9)	0.0245 (6)	0.0182 (7)	0.0213 (7)
C18	0.0190 (4)	0.0173 (4)	0.0168 (4)	0.0065 (4)	0.0064 (3)	0.0038 (3)
C19	0.0270 (5)	0.0197 (5)	0.0178 (5)	0.0029 (4)	0.0035 (4)	0.0027 (4)
C20	0.0289 (6)	0.0189 (5)	0.0222 (5)	0.0017 (4)	0.0056 (4)	0.0044 (4)
C21	0.0249 (5)	0.0210 (5)	0.0202 (5)	0.0098 (4)	0.0099 (4)	0.0073 (4)
C22	0.0241 (5)	0.0226 (5)	0.0157 (4)	0.0074 (4)	0.0059 (4)	0.0035 (4)
C23	0.0213 (5)	0.0188 (4)	0.0181 (4)	0.0046 (4)	0.0062 (4)	0.0029 (4)
C24	0.0615 (10)	0.0355 (7)	0.0211 (6)	0.0107 (7)	0.0169 (6)	0.0103 (5)
C25	0.0182 (4)	0.0158 (4)	0.0169 (4)	0.0044 (3)	0.0069 (3)	0.0020 (3)
C26	0.0199 (5)	0.0195 (5)	0.0263 (5)	0.0069 (4)	0.0085 (4)	−0.0001 (4)
C27	0.0245 (5)	0.0179 (5)	0.0276 (5)	0.0066 (4)	0.0082 (4)	−0.0013 (4)
C28	0.0214 (5)	0.0192 (5)	0.0187 (4)	0.0026 (4)	0.0057 (4)	0.0020 (4)
C29	0.0182 (5)	0.0273 (5)	0.0252 (5)	0.0062 (4)	0.0068 (4)	−0.0010 (4)
C30	0.0206 (5)	0.0224 (5)	0.0244 (5)	0.0077 (4)	0.0073 (4)	−0.0028 (4)
C31	0.0217 (6)	0.0409 (7)	0.0324 (6)	−0.0034 (5)	0.0045 (5)	−0.0004 (5)
C32	0.0701 (12)	0.0490 (10)	0.0572 (11)	0.0328 (9)	0.0283 (9)	0.0210 (8)
Cl1A	0.104 (3)	0.0631 (13)	0.0563 (13)	0.0427 (15)	0.0394 (16)	0.0343 (11)
Cl1B	0.0648 (14)	0.0990 (17)	0.0501 (10)	0.0193 (13)	0.0146 (10)	0.0415 (9)
Cl2	0.03877 (19)	0.0537 (2)	0.0484 (2)	0.00556 (17)	0.01330 (16)	0.00379 (18)

Geometric parameters (Å, °) for (3)

Mo1—P1	2.4745 (3)	C13—C14	1.3942 (16)
Mo1—C1	1.9675 (12)	C13—H13	0.95
Mo1—C2	1.9658 (12)	C14—C15	1.3908 (16)
Mo1—C3	2.2564 (11)	C15—C16	1.3863 (16)
Mo1—C9	2.3092 (12)	C15—H15	0.95
Mo1—C10	2.3210 (12)	C16—H16	0.95
Mo1—C8	2.3562 (12)	C17—H17A	0.98
Mo1—C6	2.3833 (12)	C17—H17B	0.98
Mo1—C7	2.3835 (11)	C17—H17C	0.98
P1—C25	1.8232 (11)	C18—C23	1.3925 (14)
P1—C18	1.8283 (11)	C18—C19	1.4026 (15)
P1—C11	1.8307 (11)	C19—C20	1.3823 (16)
O1—C1	1.1540 (15)	C19—H19	0.95
O2—C2	1.1547 (15)	C20—C21	1.3974 (16)
O3—C3	1.2192 (14)	C20—H20	0.95
O4—C14	1.3612 (14)	C21—C22	1.3876 (15)
O4—C17	1.4246 (17)	C22—C23	1.3959 (15)
O5—C21	1.3576 (13)	C22—H22	0.95
O5—C24	1.4298 (16)	C23—H23	0.95
O6—C28	1.3595 (13)	C24—H24A	0.98
O6—C31	1.4287 (16)	C24—H24B	0.98
C3—C4	1.5329 (17)	C24—H24C	0.98
C4—C5	1.5251 (19)	C25—C30	1.3928 (15)
C4—H4A	0.99	C25—C26	1.4009 (14)
C4—H4B	0.99	C26—C27	1.3830 (15)
C5—H5A	0.98	C26—H26	0.95

C5—H5B	0.98	C27—C28	1.3917 (16)
C5—H5C	0.98	C27—H27	0.95
C6—C10	1.4146 (18)	C28—C29	1.3914 (16)
C6—C7	1.4159 (17)	C29—C30	1.3905 (16)
C6—H6	1.0	C29—H29	0.95
C7—C8	1.4172 (17)	C30—H30	0.95
C7—H7	1.0	C31—H31A	0.98
C8—C9	1.4153 (19)	C31—H31B	0.98
C8—H8	1.0	C31—H31C	0.98
C9—C10	1.426 (2)	C32—Cl1A	1.705 (3)
C9—H9	1.0	C32—Cl2	1.7561 (19)
C10—H10	1.0	C32—Cl1B	1.783 (4)
C11—C12	1.3963 (15)	C32—H32A	0.99
C11—C16	1.3970 (14)	C32—H32B	0.99
C12—C13	1.3920 (15)	C32—H32C	0.99
C12—H12	0.95	C32—H32D	0.99
C1—Mo1—C2	106.36 (5)	C9—C10—Mo1	71.62 (7)
C1—Mo1—C3	72.49 (4)	C6—C10—H10	125.7
C2—Mo1—C3	74.79 (4)	C9—C10—H10	125.7
C2—Mo1—C9	140.57 (5)	Mo1—C10—H10	125.7
C1—Mo1—C9	100.33 (5)	C12—C11—C16	118.13 (10)
C3—Mo1—C9	86.50 (5)	C12—C11—P1	119.07 (8)
C2—Mo1—C10	106.27 (5)	C16—C11—P1	122.64 (8)
C1—Mo1—C10	131.51 (5)	C13—C12—C11	121.61 (10)
C3—Mo1—C10	82.71 (5)	C13—C12—H12	119.2
C9—Mo1—C10	35.86 (5)	C11—C12—H12	119.2
C2—Mo1—C8	153.82 (5)	C12—C13—C14	119.18 (10)
C1—Mo1—C8	99.03 (5)	C12—C13—H13	120.4
C3—Mo1—C8	120.03 (4)	C14—C13—H13	120.4
C9—Mo1—C8	35.30 (5)	O4—C14—C15	115.39 (10)
C10—Mo1—C8	58.80 (5)	O4—C14—C13	124.68 (11)
C2—Mo1—C6	96.95 (5)	C15—C14—C13	119.93 (10)
C1—Mo1—C6	156.61 (5)	C16—C15—C14	120.25 (10)
C3—Mo1—C6	112.97 (4)	C16—C15—H15	119.9
C9—Mo1—C6	58.69 (5)	C14—C15—H15	119.9
C10—Mo1—C6	34.97 (5)	C15—C16—C11	120.87 (10)
C8—Mo1—C6	58.10 (4)	C15—C16—H16	119.6
C2—Mo1—C7	119.76 (5)	C11—C16—H16	119.6
C1—Mo1—C7	127.63 (5)	O4—C17—H17A	109.5
C3—Mo1—C7	140.20 (4)	O4—C17—H17B	109.5
C9—Mo1—C7	58.32 (4)	H17A—C17—H17B	109.5
C10—Mo1—C7	58.06 (4)	O4—C17—H17C	109.5
C8—Mo1—C7	34.79 (4)	H17A—C17—H17C	109.5
C6—Mo1—C7	34.56 (4)	H17B—C17—H17C	109.5
C1—Mo1—P1	79.97 (3)	C23—C18—C19	117.86 (10)
C2—Mo1—P1	79.79 (3)	C23—C18—P1	122.10 (8)
C3—Mo1—P1	134.90 (3)	C19—C18—P1	119.96 (8)
C9—Mo1—P1	133.97 (4)	C20—C19—C18	121.19 (10)
C10—Mo1—P1	140.75 (3)	C20—C19—H19	119.4
C8—Mo1—P1	98.76 (3)	C18—C19—H19	119.4

C6—Mo1—P1	106.54 (3)	C19—C20—C21	120.10 (10)
C7—Mo1—P1	84.85 (3)	C19—C20—H20	120.0
C25—P1—C18	102.11 (5)	C21—C20—H20	120.0
C25—P1—C11	101.79 (5)	O5—C21—C22	124.58 (10)
C18—P1—C11	102.80 (5)	O5—C21—C20	115.76 (10)
C25—P1—Mo1	117.73 (3)	C22—C21—C20	119.66 (10)
C18—P1—Mo1	117.30 (3)	C21—C22—C23	119.65 (10)
C11—P1—Mo1	112.88 (3)	C21—C22—H22	120.2
C14—O4—C17	118.55 (10)	C23—C22—H22	120.2
C21—O5—C24	117.63 (10)	C18—C23—C22	121.48 (10)
C28—O6—C31	117.18 (10)	C18—C23—H23	119.3
O1—C1—Mo1	175.82 (11)	C22—C23—H23	119.3
O2—C2—Mo1	175.76 (10)	O5—C24—H24A	109.5
O3—C3—C4	116.42 (11)	O5—C24—H24B	109.5
O3—C3—Mo1	120.75 (9)	H24A—C24—H24B	109.5
C4—C3—Mo1	122.76 (8)	O5—C24—H24C	109.5
C5—C4—C3	113.12 (11)	H24A—C24—H24C	109.5
C5—C4—H4A	109.0	H24B—C24—H24C	109.5
C3—C4—H4A	109.0	C30—C25—C26	117.80 (10)
C5—C4—H4B	109.0	C30—C25—P1	120.87 (8)
C3—C4—H4B	109.0	C26—C25—P1	121.23 (8)
H4A—C4—H4B	107.8	C27—C26—C25	121.21 (10)
C4—C5—H5A	109.5	C27—C26—H26	119.4
C4—C5—H5B	109.5	C25—C26—H26	119.4
H5A—C5—H5B	109.5	C26—C27—C28	120.13 (10)
C4—C5—H5C	109.5	C26—C27—H27	119.9
H5A—C5—H5C	109.5	C28—C27—H27	119.9
H5B—C5—H5C	109.5	O6—C28—C29	124.40 (10)
C10—C6—C7	107.57 (11)	O6—C28—C27	115.94 (10)
C10—C6—Mo1	70.11 (7)	C29—C28—C27	119.66 (10)
C7—C6—Mo1	72.73 (7)	C30—C29—C28	119.63 (10)
C10—C6—H6	126.1	C30—C29—H29	120.2
C7—C6—H6	126.1	C28—C29—H29	120.2
Mo1—C6—H6	126.1	C29—C30—C25	121.55 (10)
C6—C7—C8	108.64 (11)	C29—C30—H30	119.2
C6—C7—Mo1	72.71 (7)	C25—C30—H30	119.2
C8—C7—Mo1	71.55 (7)	O6—C31—H31A	109.5
C6—C7—H7	125.6	O6—C31—H31B	109.5
C8—C7—H7	125.6	H31A—C31—H31B	109.5
Mo1—C7—H7	125.6	O6—C31—H31C	109.5
C9—C8—C7	107.71 (11)	H31A—C31—H31C	109.5
C9—C8—Mo1	70.54 (7)	H31B—C31—H31C	109.5
C7—C8—Mo1	73.66 (7)	Cl1A—C32—Cl2	112.38 (13)
C9—C8—H8	126.0	Cl2—C32—Cl1B	111.88 (16)
C7—C8—H8	126.0	Cl1A—C32—H32A	109.1
Mo1—C8—H8	126.0	Cl2—C32—H32A	109.1
C8—C9—C10	107.85 (11)	Cl1A—C32—H32B	109.1
C8—C9—Mo1	74.16 (7)	Cl2—C32—H32B	109.1
C10—C9—Mo1	72.52 (7)	H32A—C32—H32B	107.9
C8—C9—H9	125.8	Cl2—C32—H32C	109.2
C10—C9—H9	125.8	Cl1B—C32—H32C	109.2

Mo1—C9—H9	125.8	Cl2—C32—H32D	109.2
C6—C10—C9	108.20 (11)	Cl1B—C32—H32D	109.2
C6—C10—Mo1	74.93 (7)	H32C—C32—H32D	107.9
O3—C3—C4—C5	−45.68 (17)	C11—P1—C18—C23	−94.92 (9)
Mo1—C3—C4—C5	137.17 (10)	Mo1—P1—C18—C23	140.61 (8)
C10—C6—C7—C8	1.13 (13)	C25—P1—C18—C19	−173.09 (9)
Mo1—C6—C7—C8	62.95 (8)	C11—P1—C18—C19	81.66 (10)
C10—C6—C7—Mo1	−61.82 (8)	Mo1—P1—C18—C19	−42.82 (10)
C6—C7—C8—C9	−0.97 (13)	C23—C18—C19—C20	2.47 (17)
Mo1—C7—C8—C9	62.73 (8)	P1—C18—C19—C20	−174.25 (10)
C6—C7—C8—Mo1	−63.70 (8)	C18—C19—C20—C21	−2.02 (19)
C7—C8—C9—C10	0.43 (14)	C24—O5—C21—C22	4.22 (18)
Mo1—C8—C9—C10	65.20 (9)	C24—O5—C21—C20	−175.32 (12)
C7—C8—C9—Mo1	−64.78 (8)	C19—C20—C21—O5	179.25 (11)
C7—C6—C10—C9	−0.86 (14)	C19—C20—C21—C22	−0.32 (18)
Mo1—C6—C10—C9	−64.38 (8)	O5—C21—C22—C23	−177.41 (11)
C7—C6—C10—Mo1	63.52 (8)	C20—C21—C22—C23	2.11 (17)
C8—C9—C10—C6	0.27 (14)	C19—C18—C23—C22	−0.64 (16)
Mo1—C9—C10—C6	66.56 (9)	P1—C18—C23—C22	176.00 (8)
C8—C9—C10—Mo1	−66.29 (9)	C21—C22—C23—C18	−1.63 (17)
C25—P1—C11—C12	71.91 (9)	C18—P1—C25—C30	86.31 (10)
C18—P1—C11—C12	177.41 (9)	C11—P1—C25—C30	−167.65 (9)
Mo1—P1—C11—C12	−55.25 (9)	Mo1—P1—C25—C30	−43.70 (10)
C25—P1—C11—C16	−103.61 (10)	C18—P1—C25—C26	−90.06 (10)
C18—P1—C11—C16	1.89 (10)	C11—P1—C25—C26	15.98 (10)
Mo1—P1—C11—C16	129.22 (8)	Mo1—P1—C25—C26	139.94 (8)
C16—C11—C12—C13	1.21 (17)	C30—C25—C26—C27	0.61 (17)
P1—C11—C12—C13	−174.52 (9)	P1—C25—C26—C27	177.08 (9)
C11—C12—C13—C14	−0.71 (18)	C25—C26—C27—C28	−0.54 (18)
C17—O4—C14—C15	−179.30 (12)	C31—O6—C28—C29	14.43 (17)
C17—O4—C14—C13	1.31 (19)	C31—O6—C28—C27	−165.75 (12)
C12—C13—C14—O4	178.64 (11)	C26—C27—C28—O6	179.75 (11)
C12—C13—C14—C15	−0.72 (18)	C26—C27—C28—C29	−0.42 (18)
O4—C14—C15—C16	−177.78 (11)	O6—C28—C29—C30	−178.90 (11)
C13—C14—C15—C16	1.64 (18)	C27—C28—C29—C30	1.28 (18)
C14—C15—C16—C11	−1.13 (18)	C28—C29—C30—C25	−1.22 (19)
C12—C11—C16—C15	−0.28 (17)	C26—C25—C30—C29	0.27 (17)
P1—C11—C16—C15	175.28 (9)	P1—C25—C30—C29	−176.21 (9)
C25—P1—C18—C23	10.34 (10)		

Hydrogen-bond geometry (Å, °) for (3)

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C31—H31B $\cdots$ O5 ⁱ	0.98	2.58	3.4880 (16)	155
C6—H6 $\cdots$ O3 ⁱⁱ	1.00	2.57	3.4555 (16)	148
C8—H8 $\cdots$ O1 ⁱⁱⁱ	1.00	2.45	3.2714 (16)	139
C32—H32C $\cdots$ O2 ^{iv}	0.99	2.54	3.418 (2)	148

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