trans-Acetyldicarbonyl(η^5-cyclopentadienyl)[tris(3,5-dimethylphenyl)–phosphane]molybdenum(II)

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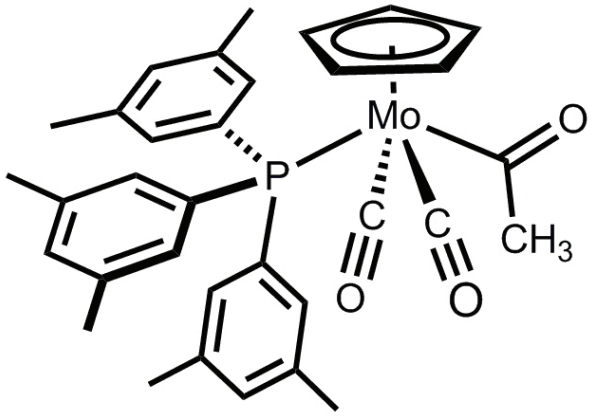
Abstract

The title compound, $[Mo(C_5H_5)(C_2H_3O)(C_{24}H_{27}P)(CO)_2]$, was prepared by reaction of $[Mo(C_5H_5)(CO)_3(CH_3)]$ with tris-(3,5-dimethylphenyl)phosphine. The complex exhibits a four-legged piano-stool geometry with *trans*-disposed acetyl and phosphine ligands. The molecular geometry is nearly identical to that of the triphenylphosphine derivative, but introduction of methyl groups on the aromatic phosphine substituents significantly impacts supramolecular organization. Non-classical C—H···O interactions involving the acetyl carbonyl group lead to a chain motif along [010], and another set of C—H···O close contacts join molecules related by an inversion center at (-x, 1-y, 1-z).



Structure description

Synthesis of the title complex, $[Mo(C_5H_5)(C_2H_3O)(C_{24}H_{27}P)(CO)_2]$ (1), has not previously been reported, though several analogous complexes have been reported. The most closely related complex for which structural information is available contains a triphenylphosphine ligand (Churchill & Fennessey, 1968).

Complex (1) exhibits a four-legged 'piano stool' geometry common for cyclopentadienyl (Cp) complexes of molybdenum (Fig. 1). The acetyl and phosphine ligands are *trans*-disposed and the acetyl ligand is oriented with the oxygen atom *syn* to the Cp ring, which is consistent with the majority of related crystal structures, the only exception being the recently reported tri(2-furyl)phosphine derivative (Whited *et al.*, 2013). The Mo—Cp centroid distance is 2.016 (1) Å. The Mo1—P1 bond length (2.4708 (7) Å) is nearly identical within error to that of the triphenylphosphine derivative and only slightly longer than those of methyldiphenyl (2.462 (2) Å) and dimethylphenyl (2.4535 (9) Å) analogues (Whited *et al.*, 2012; Whited *et al.*, 2014). The C1–Mo1–P1 angle (132.89 (8)°) is also quite similar to the triphenylphosphine complex, indicating that the added bulk of six *meta*-methyl groups does not markedly change the steric profile of the phosphine ligand near the metal center.

Although the presence of *meta*-methyl groups does not change the local structure, the supramolecular organization differs substantially from the triphenylphosphine derivative. Whereas the triphenylphosphine complex is joined into

sheets in the solid state by close contacts between the acetyl oxygen and the *meta*- and *para*-hydrogen atoms of the phosphine phenyl rings, such contacts are precluded for (1) by the presence of *meta*-methyl groups. However, the acetyl oxygen atom (O1) still plays an important role for complex (1), since C—H···O intermolecular hydrogen-bonding interactions between O1 of the acetyl carbonyl on one complex and H33*B* from a methyl group of a 3,5-dimethylphenyl phosphine substituent (C33···O1^{ii} 3.349 (4) Å, Table 3) of a neighboring complex organize the molecules into chains parallel to [010] (Fig. 2). Additional C16—H16*B*···O3 close contacts (C16···O3^{i} 3.234 (4) Å, Table 3) link molecules related by an inversion center at (-x, 1-y, 1-z).

Synthesis and crystallization

$CpMo(CO)_3(CH_3)$. This compound was prepared by a modification of the method used by Gladysz *et al.* (1979), as previously reported by Whited and Hofmeister (2014).

$CpMo(CO)_2(P(3,5\text{-}Me_2C_6H_3)_3)(COCH_3)$ (1). In an inert-atmosphere glove box, $CpMo(CO)_3(CH_3)$ (68.3 mg, 0.263 mmol) was dissolved in 5 ml acetonitrile. In a separate vial, tris(3,5-dimethylphenyl)phosphine (152 mg, 0.437 mmol) was dissolved in 5 ml acetonitrile. The vials were combined and the resulting solution was stirred for 1 week. Solvent was removed *in vacuo*, leaving an orange oil that was washed with pentane (2 *x* 3 ml), extracted into benzene (3 ml), filtered, and lyophilized to afford the desired product in pure form as a yellow powder, as confirmed by IR and NMR (^{1}H, ^{13}C, and ^{31}P) spectral analyses. Crystalline material was obtained as yellow-orange prisms by chilling a concentrated solution of (1) in diethyl ether at 233 K.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. 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 methyl groups, respectively, and with U_{iso}(H) = k×U_{eq}(C), k = 1.2 for phenyl and cyclopentadienyl groups and 1.5 for methyl groups. Methyl group H atoms were allowed to rotate in order to find the best rotameric conformation. The maximum and minimum electron densities in the final difference Fourier map are located 0.85 and 0.72 Å, respectively, from atom Mo1.

A small number of low-angle reflections (11) were rejected from this high-quality data set due to the arrangement of the instrument with a conservatively sized beam stop and a fixed-position detector. The large number of reflections in this data set (and the Fourier-transform relationship of intensities to atoms) ensures that no particular bias was thereby introduced into this routine structure determination.

Table 1

Experimental details

Crystal data	
Chemical formula	$C_{33}H_{35}MoO_3P$
M_r	606.52
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	173
a, b, c (Å)	10.9903 (11), 11.2364 (11), 14.1608 (14)
α, β, γ (°)	89.737 (8), 78.229 (6), 60.997 (7)
V (Å^3)	1488.2 (3)
Z	2
Radiation type	Mo Kα
μ (mm^{-1})	0.53

Crystal size (mm)	0.37 × 0.29 × 0.19
Data collection	
Diffractometer	Rigaku XtaLAB mini diffractometer
Absorption correction	Multi-scan REQAB (Rigaku, 1998)
T_{min}, T_{max}	0.732, 0.905
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15778, 6824, 5758
R_{int}	0.037
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.088, 1.05
No. of reflections	6824
No. of parameters	350
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.48, −0.51

Computer programs: *CrystalClear*-SM Expert 2.0 r13 (Rigaku, 2011), *SIR2008* (Burla *et al.*, 2007), *SHELXL* (Sheldrick, 2015), *Mercury* 3.9 (Macrae *et al.*, 2008), *publCIF* 1.9.19_c (Westrip, 2010).

Table 2

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

Mo1—P1	2.4708 (7)	Mo1—C3	1.970 (3)
Mo1—C1	2.270 (3)	Mo1—C4	1.966 (3)
C1—Mo1—P1	132.89 (8)	C4—Mo1—P1	78.21 (7)
C3—Mo1—P1	81.30 (7)	C4—Mo1—C1	70.1 (1)
C3—Mo1—C1	77.3 (1)	C4—Mo1—C3	108.9 (1)

Table 3

Hydrogen-bond geometry (Å, º) for (1)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16*B*···O3^{i}	0.98	2.65	3.234 (4)	119
C33—H33*B*···O1^{ii}	0.98	2.55	3.349 (4)	139

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (ii) *x*, *y*+1, *z*.

Acknowledgements

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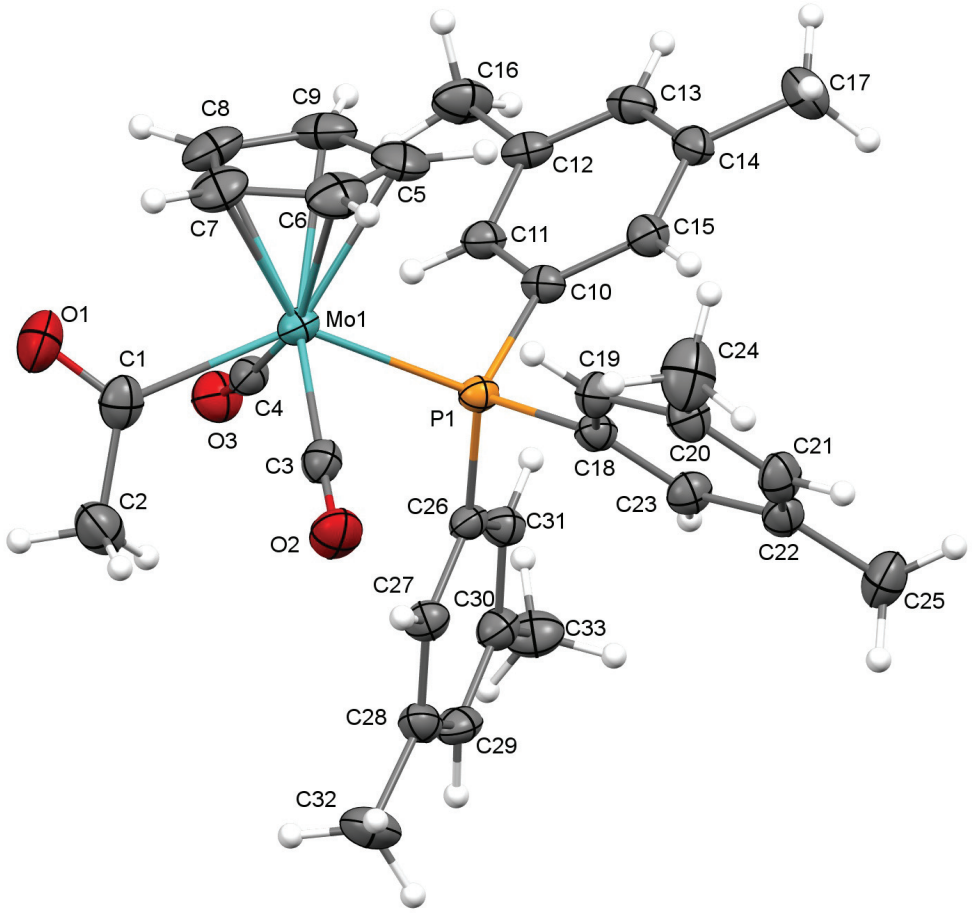

Figure 1

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

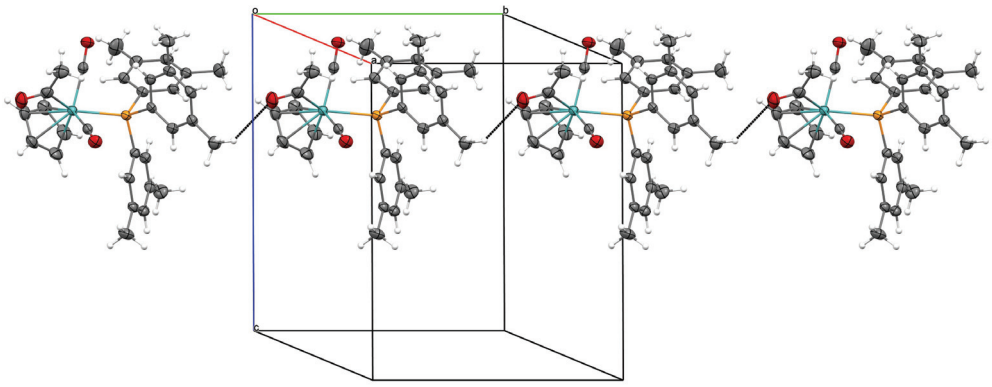

Figure 2

Crystal packing of (1) viewed perpendicular to (100) showing chains along [010]. Dashed lines indicate intermolecular C—H···O hydrogen-bonding interactions.

full crystallographic data

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Computing details

Data collection: *CrystalClear*-SM Expert 2.0 r13 (Rigaku, 2011); cell refinement: *CrystalClear*-SM Expert 2.0 r13 (Rigaku, 2011); data reduction: *CrystalClear*-SM Expert 2.0 r13 (Rigaku, 2011); program(s) used to solve structure: *SIR2008* (Burla *et al.*, 2007); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015); molecular graphics: *Mercury* 3.9 (Macrae *et al.*, 2008); software used to prepare material for publication: *publCIF* 1.9.19_c (Westrip, 2010).

trans-Acetyldicarbonyl-(η^5-cyclopentadienyl)-\ (tris(3,5-dimethylphenyl)phosphine)-molybdenum(II)

Crystal data

$C_{33}H_{35}MoO_3P$
$M_r = 606.52$
Triclinic, $P\overline{1}$
$a = 10.9903$ (11) Å
$b = 11.2364$ (11) Å
$c = 14.1608$ (14) Å
$\alpha = 89.737$ (8)°
$\beta = 78.229$ (6)°
$\gamma = 60.997$ (7)°
$V = 1488.2$ (3) Å^3
$Z = 2$
$F(000) = 628$
$D_x = 1.354$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
Cell parameters from 14088 reflections
$\theta = 3.3$–$27.7°$
$\mu = 0.53$ mm^{-1}
$T = 173$ K
Prism, yellow
0.37 × 0.29 × 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)
$T_{min} = 0.732$, $T_{max} = 0.905$
15778 measured reflections
6824 independent reflections
5758 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$
$h = -14 \rightarrow 14$
$k = -14 \rightarrow 14$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.088$
$S = 1.05$
6824 reflections
350 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.031P)^2 + 1.3336P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.51$ 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.

Refinement. Refinement was performed using all reflections. The weighted R-factor (wR) and goodness of fit (S) are based on F^2. R-factor (gt) are based on F. The threshold expression of $F^2 > 2.0$ sigma(F^2) is used only for calculating R-factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²) for (1)

	x	*y*	*z*	U_{iso}*/U_{eq}
Mo1	0.27802 (2)	0.14889 (2)	0.26221 (2)	0.02292 (7)
P1	0.33171 (7)	0.33783 (6)	0.26900 (4)	0.02146 (14)
O1	0.0668 (2)	0.0454 (2)	0.26196 (17)	0.0477 (6)
O2	0.2898 (2)	0.2042 (2)	0.04515 (14)	0.0389 (5)
O3	−0.0005 (2)	0.3708 (2)	0.40356 (14)	0.0357 (5)
C1	0.0865 (3)	0.1385 (3)	0.2354 (2)	0.0327 (6)
C2	−0.0206 (3)	0.2456 (3)	0.1860 (2)	0.0420 (7)
H2A	−0.1086	0.2400	0.1995	0.050*
H2B	0.0194	0.2296	0.1158	0.050*
H2C	−0.0419	0.3367	0.2105	0.050*
C3	0.2786 (3)	0.1917 (3)	0.12713 (19)	0.0273 (5)
C4	0.1015 (3)	0.2928 (3)	0.34792 (19)	0.0258 (5)
C5	0.5057 (3)	0.0070 (3)	0.2952 (2)	0.0429 (8)
H5	0.5817	0.0325	0.2927	0.051*
C6	0.4944 (3)	−0.0642 (3)	0.2187 (2)	0.0413 (7)
H6	0.5607	−0.0983	0.1530	0.050*
C7	0.3782 (3)	−0.0882 (3)	0.2546 (2)	0.0394 (7)
H7	0.3506	−0.1446	0.2189	0.047*
C8	0.3202 (4)	−0.0328 (3)	0.3537 (2)	0.0404 (7)
H8	0.2456	−0.0445	0.3998	0.049*
C9	0.3994 (4)	0.0253 (3)	0.3786 (2)	0.0425 (8)
H9	0.3891	0.0638	0.4451	0.051*
C10	0.3950 (3)	0.3425 (2)	0.37818 (18)	0.0241 (5)
C11	0.3086 (3)	0.3584 (3)	0.47017 (18)	0.0259 (5)
H11	0.2142	0.3742	0.4755	0.031*
C12	0.3586 (3)	0.3515 (3)	0.55382 (19)	0.0280 (6)
C13	0.4973 (3)	0.3276 (3)	0.54430 (19)	0.0296 (6)
H13	0.5321	0.3233	0.6011	0.036*
C14	0.5866 (3)	0.3099 (3)	0.4544 (2)	0.0282 (6)
C15	0.5342 (3)	0.3176 (3)	0.37144 (19)	0.0267 (5)
H15	0.5940	0.3058	0.3094	0.032*
C16	0.2665 (3)	0.3669 (3)	0.6530 (2)	0.0403 (7)
H16A	0.3106	0.2816	0.6827	0.048*
H16B	0.1714	0.3871	0.6466	0.048*
H16C	0.2578	0.4420	0.6939	0.048*
C17	0.7376 (3)	0.2816 (3)	0.4459 (2)	0.0409 (7)
H17A	0.7686	0.2462	0.5051	0.049*
H17B	0.7411	0.3665	0.4370	0.049*
H17C	0.8012	0.2136	0.3900	0.049*
C18	0.4725 (3)	0.3360 (3)	0.17228 (18)	0.0245 (5)

C19	0.5724 (3)	0.2154 (3)	0.11316 (18)	0.0276 (6)
H19	0.5634	0.1357	0.1199	0.033*
C20	0.6860 (3)	0.2102 (3)	0.0439 (2)	0.0315 (6)
C21	0.6961 (3)	0.3284 (3)	0.03509 (19)	0.0319 (6)
H21	0.7722	0.3259	−0.0125	0.038*
C22	0.5991 (3)	0.4498 (3)	0.09314 (19)	0.0283 (6)
C23	0.4860 (3)	0.4527 (3)	0.16183 (18)	0.0273 (5)
H23	0.4176	0.5354	0.2019	0.033*
C24	0.7946 (4)	0.0788 (3)	−0.0194 (3)	0.0508 (9)
H24A	0.8642	0.0189	0.0169	0.061*
H24B	0.8442	0.0995	−0.0773	0.061*
H24C	0.7460	0.0328	−0.0390	0.061*
C25	0.6142 (4)	0.5759 (3)	0.0831 (2)	0.0414 (7)
H25A	0.5736	0.6224	0.0294	0.050*
H25B	0.7156	0.5494	0.0699	0.050*
H25C	0.5632	0.6378	0.1434	0.050*
C26	0.1824 (3)	0.5099 (2)	0.26811 (17)	0.0221 (5)
C27	0.1200 (3)	0.5347 (3)	0.18874 (18)	0.0263 (5)
H27	0.1532	0.4618	0.1395	0.032*
C28	0.0104 (3)	0.6634 (3)	0.17984 (19)	0.0280 (6)
C29	−0.0354 (3)	0.7686 (3)	0.2525 (2)	0.0302 (6)
H29	−0.1083	0.8577	0.2463	0.036*
C30	0.0217 (3)	0.7479 (3)	0.33391 (19)	0.0279 (6)
C31	0.1322 (3)	0.6169 (3)	0.34102 (18)	0.0258 (5)
H31	0.1732	0.6007	0.3959	0.031*
C32	−0.0603 (3)	0.6872 (3)	0.0953 (2)	0.0428 (7)
H32A	−0.1484	0.6821	0.1149	0.051*
H32B	0.0049	0.6171	0.0410	0.051*
H32C	−0.0834	0.7780	0.0753	0.051*
C33	−0.0352 (3)	0.8636 (3)	0.4132 (2)	0.0395 (7)
H33A	−0.1374	0.9247	0.4180	0.047*
H33B	0.0160	0.9150	0.3980	0.047*
H33C	−0.0210	0.8260	0.4752	0.047*

Atomic displacement parameters (Å²) for (1)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.02448 (12)	0.01994 (11)	0.02207 (12)	−0.00976 (9)	−0.00405 (8)	0.00436 (8)
P1	0.0215 (3)	0.0217 (3)	0.0190 (3)	−0.0094 (3)	−0.0038 (2)	0.0036 (2)
O1	0.0512 (14)	0.0469 (13)	0.0568 (14)	−0.0352 (12)	−0.0071 (11)	0.0083 (11)
O2	0.0509 (13)	0.0478 (12)	0.0263 (10)	−0.0300 (11)	−0.0108 (9)	0.0082 (9)
O3	0.0292 (11)	0.0358 (11)	0.0338 (11)	−0.0120 (9)	−0.0011 (9)	0.0007 (9)
C1	0.0333 (15)	0.0391 (16)	0.0260 (14)	−0.0210 (13)	0.0010 (11)	−0.0017 (12)
C2	0.0354 (17)	0.055 (2)	0.0395 (17)	−0.0237 (16)	−0.0120 (13)	0.0066 (15)
C3	0.0295 (14)	0.0251 (13)	0.0300 (14)	−0.0157 (12)	−0.0066 (11)	0.0035 (11)
C4	0.0300 (14)	0.0263 (13)	0.0258 (13)	−0.0170 (12)	−0.0080 (11)	0.0070 (11)
C5	0.0326 (16)	0.0328 (16)	0.057 (2)	−0.0073 (13)	−0.0216 (15)	0.0153 (15)
C6	0.0319 (16)	0.0295 (15)	0.0421 (17)	−0.0024 (13)	−0.0011 (13)	0.0046 (13)
C7	0.0456 (18)	0.0208 (13)	0.0447 (17)	−0.0114 (13)	−0.0094 (14)	0.0049 (12)
C8	0.0504 (19)	0.0268 (14)	0.0364 (16)	−0.0150 (14)	−0.0055 (14)	0.0130 (12)
C9	0.052 (2)	0.0304 (15)	0.0389 (17)	−0.0114 (15)	−0.0229 (15)	0.0136 (13)

C10	0.0260 (13)	0.0220 (12)	0.0229 (12)	−0.0099 (11)	−0.0080 (10)	0.0033 (10)
C11	0.0240 (13)	0.0247 (13)	0.0254 (13)	−0.0092 (11)	−0.0059 (10)	0.0065 (10)
C12	0.0325 (14)	0.0215 (12)	0.0248 (13)	−0.0090 (11)	−0.0078 (11)	0.0063 (10)
C13	0.0353 (15)	0.0246 (13)	0.0296 (14)	−0.0122 (12)	−0.0159 (12)	0.0052 (11)
C14	0.0270 (14)	0.0240 (13)	0.0361 (15)	−0.0121 (11)	−0.0138 (11)	0.0065 (11)
C15	0.0257 (13)	0.0260 (13)	0.0265 (13)	−0.0118 (11)	−0.0047 (10)	0.0028 (10)
C16	0.0406 (17)	0.0460 (18)	0.0256 (14)	−0.0154 (15)	−0.0059 (13)	0.0105 (13)
C17	0.0331 (16)	0.0484 (18)	0.0490 (18)	−0.0223 (15)	−0.0192 (14)	0.0087 (15)
C18	0.0238 (13)	0.0267 (13)	0.0218 (12)	−0.0115 (11)	−0.0059 (10)	0.0060 (10)
C19	0.0262 (13)	0.0298 (14)	0.0271 (13)	−0.0150 (12)	−0.0035 (11)	0.0028 (11)
C20	0.0268 (14)	0.0345 (15)	0.0306 (14)	−0.0145 (12)	−0.0030 (11)	−0.0016 (12)
C21	0.0307 (15)	0.0427 (16)	0.0247 (13)	−0.0220 (13)	−0.0009 (11)	0.0027 (12)
C22	0.0318 (14)	0.0356 (15)	0.0242 (13)	−0.0211 (13)	−0.0083 (11)	0.0073 (11)
C23	0.0284 (14)	0.0289 (13)	0.0221 (12)	−0.0135 (12)	−0.0026 (10)	0.0022 (10)
C24	0.0416 (19)	0.0464 (19)	0.053 (2)	−0.0213 (16)	0.0121 (15)	−0.0146 (16)
C25	0.0488 (19)	0.0436 (17)	0.0413 (17)	−0.0320 (16)	−0.0053 (14)	0.0080 (14)
C26	0.0222 (12)	0.0206 (12)	0.0223 (12)	−0.0103 (10)	−0.0037 (10)	0.0067 (10)
C27	0.0290 (14)	0.0255 (13)	0.0228 (12)	−0.0125 (11)	−0.0048 (10)	0.0027 (10)
C28	0.0257 (13)	0.0348 (14)	0.0245 (13)	−0.0151 (12)	−0.0079 (11)	0.0093 (11)
C29	0.0265 (14)	0.0227 (13)	0.0342 (15)	−0.0071 (11)	−0.0060 (11)	0.0101 (11)
C30	0.0281 (14)	0.0244 (13)	0.0291 (14)	−0.0123 (11)	−0.0042 (11)	0.0049 (11)
C31	0.0251 (13)	0.0272 (13)	0.0231 (13)	−0.0114 (11)	−0.0055 (10)	0.0044 (10)
C32	0.0422 (18)	0.0450 (18)	0.0384 (17)	−0.0151 (15)	−0.0212 (14)	0.0134 (14)
C33	0.0422 (18)	0.0265 (14)	0.0377 (16)	−0.0084 (13)	−0.0077 (13)	0.0017 (12)

Geometric parameters (Å, º) for (1)

Mo1—P1	2.4708 (7)	C16—H16A	0.9800
Mo1—C1	2.270 (3)	C16—H16B	0.9800
Mo1—C3	1.970 (3)	C16—H16C	0.9800
Mo1—C4	1.966 (3)	C17—H17A	0.9800
Mo1—C5	2.371 (3)	C17—H17B	0.9800
Mo1—C6	2.379 (3)	C17—H17C	0.9800
Mo1—C7	2.330 (3)	C18—C19	1.391 (4)
Mo1—C8	2.316 (3)	C18—C23	1.395 (4)
Mo1—C9	2.346 (3)	C19—H19	0.9500
P1—C10	1.831 (3)	C19—C20	1.398 (4)
P1—C18	1.837 (3)	C20—C21	1.389 (4)
P1—C26	1.834 (2)	C20—C24	1.508 (4)
O1—C1	1.211 (3)	C21—H21	0.9500
O2—C3	1.157 (3)	C21—C22	1.384 (4)
O3—C4	1.157 (3)	C22—C23	1.400 (4)
C1—C2	1.510 (4)	C22—C25	1.507 (4)
C2—H2A	0.9800	C23—H23	0.9500
C2—H2B	0.9800	C24—H24A	0.9800
C2—H2C	0.9800	C24—H24B	0.9800
C5—H5	1.0000	C24—H24C	0.9800
C5—C6	1.410 (5)	C25—H25A	0.9800
C5—C9	1.417 (5)	C25—H25B	0.9800
C6—H6	1.0000	C25—H25C	0.9800
C6—C7	1.424 (4)	C26—C27	1.392 (3)

C7—H7	1.0000	C26—C31	1.396 (3)
C7—C8	1.422 (4)	C27—H27	0.9500
C8—H8	1.0000	C27—C28	1.389 (4)
C8—C9	1.410 (5)	C28—C29	1.390 (4)
C9—H9	1.0000	C28—C32	1.511 (4)
C10—C11	1.401 (3)	C29—H29	0.9500
C10—C15	1.397 (4)	C29—C30	1.388 (4)
C11—H11	0.9500	C30—C31	1.403 (4)
C11—C12	1.391 (4)	C30—C33	1.510 (4)
C12—C13	1.391 (4)	C31—H31	0.9500
C12—C16	1.510 (4)	C32—H32A	0.9800
C13—H13	0.9500	C32—H32B	0.9800
C13—C14	1.387 (4)	C32—H32C	0.9800
C14—C15	1.396 (4)	C33—H33A	0.9800
C14—C17	1.509 (4)	C33—H33B	0.9800
C15—H15	0.9500	C33—H33C	0.9800

C1—Mo1—P1	132.89 (8)	C12—C11—C10	121.1 (2)
C1—Mo1—C5	140.69 (11)	C12—C11—H11	119.5
C1—Mo1—C6	112.85 (11)	C11—C12—C16	121.3 (3)
C1—Mo1—C7	82.86 (11)	C13—C12—C11	118.5 (2)
C1—Mo1—C8	87.38 (11)	C13—C12—C16	120.2 (2)
C1—Mo1—C9	120.95 (11)	C12—C13—H13	119.0
C3—Mo1—P1	81.30 (7)	C14—C13—C12	122.1 (2)
C3—Mo1—C1	77.3 (1)	C14—C13—H13	119.0
C3—Mo1—C5	115.7 (1)	C13—C14—C15	118.5 (2)
C3—Mo1—C6	93.91 (11)	C13—C14—C17	121.1 (2)
C3—Mo1—C7	105.47 (11)	C15—C14—C17	120.4 (3)
C3—Mo1—C8	140.35 (11)	C10—C15—H15	119.5
C3—Mo1—C9	150.13 (12)	C14—C15—C10	121.0 (2)
C4—Mo1—P1	78.21 (7)	C14—C15—H15	119.5
C4—Mo1—C1	70.1 (1)	C12—C16—H16A	109.5
C4—Mo1—C3	108.9 (1)	C12—C16—H16B	109.5
C4—Mo1—C5	129.63 (11)	C12—C16—H16C	109.5
C4—Mo1—C6	156.82 (11)	H16A—C16—H16B	109.5
C4—Mo1—C7	129.38 (11)	H16A—C16—H16C	109.5
C4—Mo1—C8	99.41 (11)	H16B—C16—H16C	109.5
C4—Mo1—C9	99.96 (11)	C14—C17—H17A	109.5
C5—Mo1—P1	86.42 (8)	C14—C17—H17B	109.5
C5—Mo1—C6	34.53 (11)	C14—C17—H17C	109.5
C6—Mo1—P1	110.01 (8)	H17A—C17—H17B	109.5
C7—Mo1—P1	143.68 (8)	H17A—C17—H17C	109.5
C7—Mo1—C5	58.19 (12)	H17B—C17—H17C	109.5
C7—Mo1—C6	35.18 (11)	C19—C18—P1	120.21 (19)
C7—Mo1—C9	58.63 (11)	C19—C18—C23	119.4 (2)
C8—Mo1—P1	132.52 (9)	C23—C18—P1	120.29 (19)
C8—Mo1—C5	58.43 (12)	C18—C19—H19	119.6
C8—Mo1—C6	58.71 (11)	C18—C19—C20	120.8 (2)
C8—Mo1—C7	35.64 (10)	C20—C19—H19	119.6
C8—Mo1—C9	35.21 (11)	C19—C20—C24	120.5 (3)
C9—Mo1—P1	97.80 (8)	C21—C20—C19	118.4 (2)

C9—Mo1—C5	34.96 (11)	C21—C20—C24	121.1 (3)
C9—Mo1—C6	58.10 (11)	C20—C21—H21	118.8
C10—P1—Mo1	110.94 (8)	C22—C21—C20	122.4 (2)
C10—P1—C18	102.31 (12)	C22—C21—H21	118.8
C10—P1—C26	106.53 (11)	C21—C22—C23	118.2 (2)
C18—P1—Mo1	118.64 (8)	C21—C22—C25	121.2 (2)
C26—P1—Mo1	115.73 (8)	C23—C22—C25	120.6 (2)
C26—P1—C18	101.16 (11)	C18—C23—C22	120.9 (2)
O1—C1—Mo1	120.0 (2)	C18—C23—H23	119.6
O1—C1—C2	117.7 (3)	C22—C23—H23	119.6
C2—C1—Mo1	122.3 (2)	C20—C24—H24A	109.5
C1—C2—H2A	109.5	C20—C24—H24B	109.5
C1—C2—H2B	109.5	C20—C24—H24C	109.5
C1—C2—H2C	109.5	H24A—C24—H24B	109.5
H2A—C2—H2B	109.5	H24A—C24—H24C	109.5
H2A—C2—H2C	109.5	H24B—C24—H24C	109.5
H2B—C2—H2C	109.5	C22—C25—H25A	109.5
O2—C3—Mo1	173.4 (2)	C22—C25—H25B	109.5
O3—C4—Mo1	174.9 (2)	C22—C25—H25C	109.5
Mo1—C5—H5	125.6	H25A—C25—H25B	109.5
C6—C5—Mo1	73.04 (17)	H25A—C25—H25C	109.5
C6—C5—H5	125.6	H25B—C25—H25C	109.5
C6—C5—C9	108.5 (3)	C27—C26—P1	117.55 (19)
C9—C5—Mo1	71.54 (17)	C27—C26—C31	119.1 (2)
C9—C5—H5	125.6	C31—C26—P1	123.34 (19)
Mo1—C6—H6	126.1	C26—C27—H27	119.2
C5—C6—Mo1	72.42 (17)	C28—C27—C26	121.6 (2)
C5—C6—H6	126.1	C28—C27—H27	119.2
C5—C6—C7	107.5 (3)	C27—C28—C29	118.1 (2)
C7—C6—Mo1	70.52 (16)	C27—C28—C32	120.9 (3)
C7—C6—H6	126.1	C29—C28—C32	120.9 (2)
Mo1—C7—H7	125.8	C28—C29—H29	118.9
C6—C7—Mo1	74.29 (16)	C30—C29—C28	122.3 (2)
C6—C7—H7	125.8	C30—C29—H29	118.9
C8—C7—Mo1	71.62 (16)	C29—C30—C31	118.4 (2)
C8—C7—C6	108.0 (3)	C29—C30—C33	120.6 (2)
C8—C7—H7	125.8	C31—C30—C33	121.0 (2)
Mo1—C8—H8	125.8	C26—C31—C30	120.6 (2)
C7—C8—Mo1	72.74 (16)	C26—C31—H31	119.7
C7—C8—H8	125.8	C30—C31—H31	119.7
C9—C8—Mo1	73.58 (16)	C28—C32—H32A	109.5
C9—C8—C7	107.9 (3)	C28—C32—H32B	109.5
C9—C8—H8	125.8	C28—C32—H32C	109.5
Mo1—C9—H9	125.8	H32A—C32—H32B	109.5
C5—C9—Mo1	73.50 (16)	H32A—C32—H32C	109.5
C5—C9—H9	125.8	H32B—C32—H32C	109.5
C8—C9—Mo1	71.22 (16)	C30—C33—H33A	109.5
C8—C9—C5	108.1 (3)	C30—C33—H33B	109.5
C8—C9—H9	125.8	C30—C33—H33C	109.5
C11—C10—P1	120.06 (19)	H33A—C33—H33B	109.5
C15—C10—P1	120.89 (19)	H33A—C33—H33C	109.5

C15—C10—C11	118.8 (2)	H33B—C33—H33C	109.5
C10—C11—H11	119.5		
Mo1—P1—C10—C11	−58.8 (2)	C12—C13—C14—C15	0.5 (4)
Mo1—P1—C10—C15	115.2 (2)	C12—C13—C14—C17	−178.9 (3)
Mo1—P1—C18—C19	−17.9 (2)	C13—C14—C15—C10	−0.1 (4)
Mo1—P1—C18—C23	166.56 (18)	C15—C10—C11—C12	0.8 (4)
Mo1—P1—C26—C27	−59.4 (2)	C16—C12—C13—C14	178.5 (3)
Mo1—P1—C26—C31	122.65 (19)	C17—C14—C15—C10	179.4 (2)
Mo1—C5—C6—C7	−62.2 (2)	C18—P1—C10—C11	173.7 (2)
Mo1—C5—C9—C8	63.3 (2)	C18—P1—C10—C15	−12.3 (2)
Mo1—C6—C7—C8	−64.2 (2)	C18—P1—C26—C27	70.2 (2)
Mo1—C7—C8—C9	−65.7 (2)	C18—P1—C26—C31	−107.8 (2)
Mo1—C8—C9—C5	−64.8 (2)	C18—C19—C20—C21	−0.4 (4)
P1—C10—C11—C12	174.91 (19)	C18—C19—C20—C24	179.6 (3)
P1—C10—C15—C14	−174.66 (19)	C19—C18—C23—C22	−0.3 (4)
P1—C18—C19—C20	−175.4 (2)	C19—C20—C21—C22	0.9 (4)
P1—C18—C23—C22	175.2 (2)	C20—C21—C22—C23	−1.1 (4)
P1—C26—C27—C28	−177.2 (2)	C20—C21—C22—C25	178.9 (3)
P1—C26—C31—C30	177.09 (19)	C21—C22—C23—C18	0.8 (4)
C5—C6—C7—Mo1	63.4 (2)	C23—C18—C19—C20	0.2 (4)
C5—C6—C7—C8	−0.8 (3)	C24—C20—C21—C22	−179.1 (3)
C6—C5—C9—Mo1	−64.2 (2)	C25—C22—C23—C18	−179.2 (3)
C6—C5—C9—C8	−0.9 (3)	C26—P1—C10—C11	68.0 (2)
C6—C7—C8—Mo1	65.9 (2)	C26—P1—C10—C15	−118.0 (2)
C6—C7—C8—C9	0.2 (3)	C26—P1—C18—C19	−145.6 (2)
C7—C8—C9—Mo1	65.2 (2)	C26—P1—C18—C23	38.8 (2)
C7—C8—C9—C5	0.4 (3)	C26—C27—C28—C29	0.5 (4)
C9—C5—C6—Mo1	63.2 (2)	C26—C27—C28—C32	−177.4 (3)
C9—C5—C6—C7	1.0 (3)	C27—C26—C31—C30	−0.8 (4)
C10—P1—C18—C19	104.5 (2)	C27—C28—C29—C30	−1.9 (4)
C10—P1—C18—C23	−71.0 (2)	C28—C29—C30—C31	1.8 (4)
C10—P1—C26—C27	176.78 (19)	C28—C29—C30—C33	−177.5 (3)
C10—P1—C26—C31	−1.2 (2)	C29—C30—C31—C26	−0.4 (4)
C10—C11—C12—C13	−0.3 (4)	C31—C26—C27—C28	0.8 (4)
C10—C11—C12—C16	−179.2 (2)	C32—C28—C29—C30	176.1 (3)
C11—C10—C15—C14	−0.6 (4)	C33—C30—C31—C26	178.9 (2)
C11—C12—C13—C14	−0.4 (4)		

Hydrogen-bond geometry (Å, º) for (1)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16*B*···O3^{i}	0.98	2.65	3.234 (4)	119
C33—H33*B*···O1^{ii}	0.98	2.55	3.349 (4)	139

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (ii) *x*, *y*+1, *z*.