

# *trans*-Acetyldicarbonyl( $\eta^5$ -cyclopentadienyl)-[tris(3,5-dimethylphenyl)phosphane- $\kappa P$ ]-molybdenum(II)

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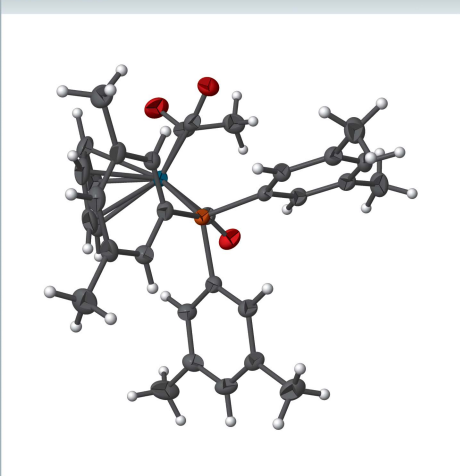
Keywords: crystal structure; phosphine; piano-stool complex; cyclopentadienyl; divalent molybdenum.

CCDC reference: 1526390

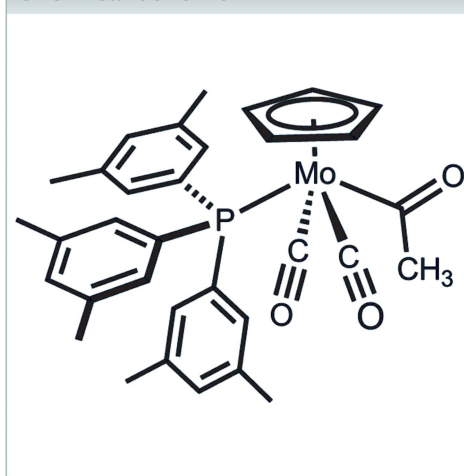
Structural data: full structural data are available from iucrdata.iucr.org

The title compound,  $[\text{Mo}(\text{C}_5\text{H}_5)(\text{C}_2\text{H}_3\text{O})(\text{C}_{24}\text{H}_{27}\text{P})(\text{CO})_2]$ , was prepared by reaction of  $[\text{Mo}(\text{C}_5\text{H}_5)(\text{CO})_3(\text{CH}_3)]$  with tris(3,5-dimethylphenyl)phosphane. The complex exhibits a four-legged piano-stool geometry with *trans*-disposed acetyl and phosphane ligands. The molecular geometry is nearly identical to that of the triphenylphosphane derivative, but introduction of methyl groups on the aromatic phosphane substituents significantly impacts supramolecular organization. In the crystal, non-classical C—H $\cdots$ O interactions involving the acetyl carbonyl group lead to a chain motif along [010], and another set of C—H $\cdots$ O close contacts join inversion-related molecules.

## 3D view



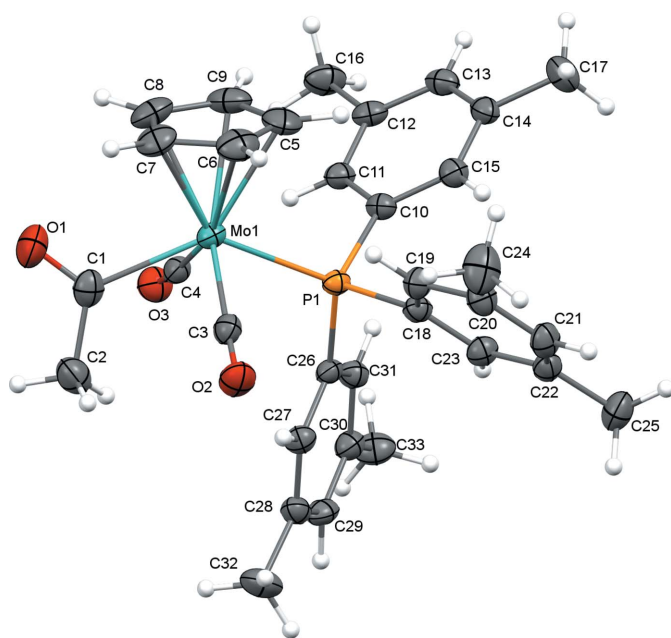
## Chemical scheme



## Structure description

The synthesis of the title complex,  $[\text{Mo}(\text{C}_5\text{H}_5)(\text{C}_2\text{H}_3\text{O})(\text{C}_{24}\text{H}_{27}\text{P})(\text{CO})_2]$ , (1), has not been reported previously, though several analogous complexes are known. The most closely related complex for which structural information is available contains a triphenylphosphane ligand (Churchill & Fennessey, 1968).

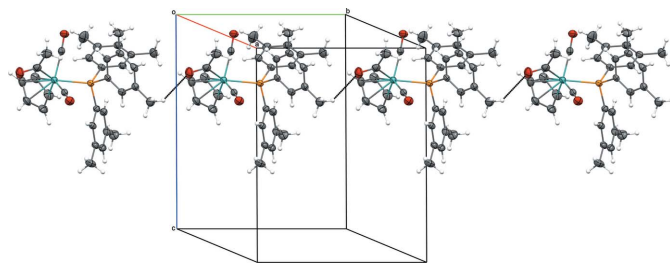
Complex (1) exhibits a four-legged ‘piano-stool’ geometry, common for cyclopentadienyl (Cp) complexes of molybdenum (Fig. 1, Table 1). The acetyl and phosphane ligands are *trans*-disposed and the acetyl ligand is oriented with the O atom *syn* to the Cp ring, which is consistent with the majority of related crystal structures, the only exception being the recently reported tri(2-furyl)phosphane 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 triphenylphosphane derivative and only



**Figure 1**  
The molecular structure of (1), with displacement ellipsoids drawn at the 50% probability level.

slightly longer than those of methylphenyl [2.462 (2) Å] and dimethylphenyl [2.4535 (9) Å] analogues (Whited *et al.*, 2012, 2014). The C1–Mo1–P1 angle [132.89 (8)°] is also quite similar to the triphenylphosphane complex, indicating that the added bulk of six *meta*-methyl groups does not markedly change the steric profile of the phosphane ligand near the metal atom.

Although the presence of *meta*-methyl groups does not change the local structure, the supramolecular organization differs substantially from the triphenylphosphane derivative. Whereas the triphenylphosphane complex is joined into sheets in the solid state by close contacts between the acetyl O and the *meta* and *para* H atoms of the phosphane phenyl rings, such contacts are precluded for (1) by the presence of *meta*-methyl groups. However, the acetyl O1 atom still plays an important role for complex (1), since intermolecular C–H···O hydrogen-bonding interactions between atom O1 of the acetyl carbonyl group on one complex and atom H33B from a methyl group of a 3,5-dimethylphenyl phosphane



**Figure 2**  
The crystal packing of (1), viewed approximately perpendicular to (100), showing chains along [010]. Dashed lines indicate intermolecular C–H···O hydrogen-bonding interactions.

**Table 1**  
Selected geometric parameters (Å, °).

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 2**  
Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
C16–H16B···O3 <sup>i</sup>	0.98	2.65	3.234 (4)	119
C33–H33B···O1 <sup>ii</sup>	0.98	2.55	3.349 (4)	139

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

substituent [C33···O1<sup>ii</sup> = 3.349 (4) Å; Table 2] of a neighboring complex organize the molecules into chains parallel to [010] (Fig. 2). Additional C16–H16B···O3 close contacts [C16···O3<sup>i</sup> = 3.234 (4) Å; Table 2] link molecules related by an inversion center at  $(-x, -y + 1, -z + 1)$ .

### Synthesis and crystallization

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

**CpMo(CO)<sub>2</sub>(P(3,5-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>)<sub>3</sub>)(COCH<sub>3</sub>) (1)**. In an inert atmosphere glove-box, CpMo(CO)<sub>3</sub>(CH<sub>3</sub>) (68.3 mg, 0.263 mmol) was dissolved in 5 ml acetonitrile. In a separate vial, tris(3,5-dimethylphenyl)phosphane (152 mg, 0.437 mmol) was dissolved in 5 ml acetonitrile. The vials were combined and the resulting solution was stirred for 1 week. The solvent was removed *in vacuo*, leaving an orange oil that was washed with pentane (2 × 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 (<sup>1</sup>H, <sup>13</sup>C, and <sup>31</sup>P) spectroscopic 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 3. The maximum and minimum electron densities in the final difference Fourier map are located 0.85 and 0.72 Å<sup>3</sup>, 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 3**  
Experimental details.

Crystal data	
Chemical formula	[Mo(C <sub>5</sub> H <sub>5</sub> )(C <sub>2</sub> H <sub>3</sub> O)(C <sub>24</sub> H <sub>27</sub> P)- (CO) <sub>2</sub> ]
<i>M<sub>r</sub></i>	606.52
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	10.9903 (11), 11.2364 (11), 14.1608 (14)
$\alpha$ , $\beta$ , $\gamma$ (°)	89.737 (8), 78.229 (6), 60.997 (7)
<i>V</i> (Å <sup>3</sup> )	1488.2 (3)
<i>Z</i>	2
Radiation type	Mo <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	0.53
Crystal size (mm)	0.37 × 0.29 × 0.19
Data collection	
Diffractometer	Rigaku XtaLAB mini
Absorption correction	Multi-scan ( <i>REQAB</i> ; Rigaku, 1998)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.732, 0.905
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	15778, 6824, 5758
<i>R<sub>int</sub></i> ( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.037 0.649
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	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 <sup>-3</sup> )	0.48, -0.51

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

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Single Crystal X-ray Diffractometer for a Regional PUI Molecular Structure Facility'. Additional support was provided by the NSF in the form of a CAREER award to MTW (CHE-1552591) and by Carleton College.

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## References

- Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G., Siliqi, D. & Spagna, R. (2007). *J. Appl. Cryst.* **40**, 609–613.
- Churchill, M. R. & Fennessey, J. P. (1968). *Inorg. Chem.* **7**, 553–558.
- Gladysz, J. A., Williams, G. M., Tam, W., Johnson, D. L., Parker, D. W. & Selover, J. C. (1979). *Inorg. Chem.* **18**, 553–558.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Rigaku (1998). *REQAB*. Rigaku Corporation, Tokyo, Japan.
- Rigaku (2011). *CrystalClear*. Rigaku Americas, The Woodlands, Texas, USA, and Rigaku Corporation, Tokyo, Japan.
- 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. T., Keyes, S. H., Ngamnthiporn, A. & Janzen, D. E. (2014). *Acta Cryst.* **E70**, 216–220.

## full crystallographic data

*IUCrData* (2017). 2, x170042 [https://doi.org/10.1107/S2414314617000426]

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*trans*-Acetyldicarbonyl( $\eta^5$ -cyclopentadienyl)[tris(3,5-dimethylphenyl)phosphane- $\kappa P$ ]molybdenum(II)

*Crystal data*

[Mo(C<sub>5</sub>H<sub>5</sub>)(C<sub>2</sub>H<sub>3</sub>O)(C<sub>24</sub>H<sub>27</sub>P)(CO)<sub>2</sub>]

$M_r = 606.52$

Triclinic,  $P\bar{1}$

$a = 10.9903$  (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<sup>3</sup>

$Z = 2$

$F(000) = 628$

$D_x = 1.354$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71075$  Å

Cell parameters from 14088 reflections

$\theta = 3.3$ – $27.7$ °

$\mu = 0.53$  mm<sup>-1</sup>

$T = 173$  K

Prism, yellow

$0.37 \times 0.29 \times 0.19$  mm

*Data collection*

Rigaku XtaLAB mini  
diffractometer

Detector resolution: 6.849 pixels mm<sup>-1</sup>

$\omega$  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  $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 27.5$ °,  $\theta_{\min} = 3.2$ °

$h = -14 \rightarrow 14$

$k = -14 \rightarrow 14$

$l = -18 \rightarrow 18$

*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<sup>-3</sup>

$\Delta\rho_{\min} = -0.51$  e Å<sup>-3</sup>

*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.** H atoms were placed in calculated positions and refined using 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  $U_{\text{iso}}(\text{H}) = kU_{\text{eq}}(\text{C})$ , with  $k = 1.2$  for phenyl and cyclopentadienyl groups and 1.5 for methyl groups. Methyl groups were allowed to rotate in order to find the best rotameric conformation.

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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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, °)*

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, °)*

<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>
C16—H16 <i>B</i> $\cdots$ O3 <sup>i</sup>	0.98	2.65	3.234 (4)	119
C33—H33 <i>B</i> $\cdots$ O1 <sup>ii</sup>	0.98	2.55	3.349 (4)	139

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