Crystal Structures of *trans*-Acetyldicarbonyl(η^5-cyclopentadienyl)(1,3,5-triaza-7-phosphaadamantane)molybdenum(II) and *trans*-Acetyldicarbonyl(η^5-cyclopentadienyl)(3,7-diacetyl-1,3,7-triaza-5-phosphabicyclo[3.3.1]nonane)molybdenum(II)

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Abstract

The title compounds, [Mo(C_5H_5)(PTA)($CO)_2$($COCH_3$)], (1), and [Mo(C_5H_5)(DAPTA)($C_6H_5)_2$))($CO)_2$($COCH_3$)], (2), have been prepared by phosphine-induced migratory insertion from [Mo(C_5H_5)($CO)_3$(CH_3)]. The molecular structures of these complexes are quite similar, exhibiting a four-legged piano-stool geometry with *trans*-disposed carbonyl ligands. The extended structures of complexes (1) and (2) differ substantially. For complex (1), the molybdenum acetyl unit plays a dominant role in organization of the extended structure, joining the molecular into centrosymmetrical dimers through C—H···O interactions with a cyclopentadienyl ligand of a neighboring molecule, and these dimers are linked and into layers parallel to (100) by C—H···O interactions between the molybdenum acetyl and the cyclopentadienyl ligand of another neighbor. The extended structure of (2) is dominated by C—H···O interactions involving the carbonyl groups of acetamide groups of the DAPTA ligand, which join the molecules into centrosymmetrical dimers and linked them in chains along [010]. Additional C—H···O interactions between the molybdenum acetyl oxygen and an acetamide methyl group join the chains into layers parallel to (101).

1. Chemical context

Cyclopentadienylmolybdenum polycarbonyl complexes (Mo(C_5H_5)($CO)_n$) exhibiting "four-legged piano-stool" geometries have been extensively studied for their electronic structure and organometallic reactivity (Kubacek *et al.*, 1982). Specifically, alkyl complexes of the form Mo(C_5H_5)($CO)_3$(*R*) have been shown to undergo carbonyl migratory insertion, affording acyl complexes upon exposure to L-type ligands, especially phosphines (Barnett & Treichel, 1967; Butler *et al.*, 1967). The steric bulk of the phosphine ligand strongly influences the stability of the resulting complexes, with bulkier groups exhibiting enhanced deinsertion rates (Barnett, 1969; Barnett & Pollmann, 1974).

We have previously described the solid-state structures of a number of four-legged piano-stool molybdenum acetyl complexes of the type Mo(PR_3)(C_5H_5)($CO)_2$($COCH_3$) derived from reaction of the molybdenum methyl precursor with various phosphines (Whited & Hofmeister, 2014). we have showed that the steric bulk of the phosphine substituents impacts the molecular structure of the insertion product in predictable ways, primarily evidenced through the Mo–P bond lengths and P–Mo–C bond angles (Whited *et al.*, 2012, Whited *et al.*, 2014), consistent with earlier findings on reactivity. We have also shown that use of tri(2-furyl)phosphine, which features heteroatoms as potential H-bond acceptors, leads to an unusual structure where the acetyl is oriented away from the Cp ring rather than toward it as in other cases (Whited *et al.*, 2013).

Here we report the structures of piano-stool molybdenum acetyl complexes with 1,3,5-triaza-7-phosphaadamantane (PTA) and 3,7-diacetyl-1,3,7-triaza-5-phosphabicyclo[3.3.1]nonane (DAPTA) ligands featuring potential H-bond accepting amines and carbonyl groups. The diacetylated ligand (DAPTA) shows little difference from PTA in local

structure, but the introduction of acetamide groups dramatically impacts supramolecular organization.

2. Structural commentary

The molecular structures of (1) and (2) are illustrated in Figs. 1 and 2. Both (1) and (2) exhibit a *trans* disposition of carbonyl ligands, as seen for other compounds of this type. It was envisioned that incorporation of hydrogen-bond acceptors (amines and amides) might allow access to an alternate orientation of the acetyl group, as observed for a related structure featuring a tri(2-furyl)phosphine ligand, but instead the oxygen of the acetyl group points toward the Cp ring, consistent with other structures of related complexes.

Selected geometric parameters for (1) and (2) are presented in Tables 1 and 2. The Mo1–P1 bond lengths for PTA derivative (1) [2.4321 (5) Å] and DAPTA derivative (2) [2.4259 (6) Å] are similar to one another and slightly shorter than related complexes we have reported. This finding is consistent with the lower steric profile of the polycyclic PTA and DAPTA ligands relative to, e.g., PPh_3, $PMePh_2$, and PMe_2Ph. The most significant molecular difference between (1) and (2) is seen in the C3–Mo1–P1 bond angle, which is larger for (1) [136.77 (4)°] than for (2) [133.97 (6)°].

3. Supramolecular features

Although the molecular structures of (1) and (2) are quite similar, the diacetylation of the phosphine ligand (transforming PTA to DAPTA) leads to significant differences in extended structure. As was the case for several previously reported complexes of this sort with different phosphine ligands, the extended structure of (1) is dominated by interactions of O3 from the molybdenum acetyl. Short, non-classical C—H···O interactions between O3 and H8 of a cyclopentadienyl (Cp) ring (2.49 Å) link (1) into centrosymmetrical dimers (Fig. 3). These dimers are further connected into layers parallel to (100) by non-classical C—H···O hydrogen bonds between O3 and H9 (2.37 Å) (Fig. 4). Although the PTA ligand features three nitrogen atoms as potential H-bond acceptors, these are not observed to play an important organizing structural role.

The acetamide groups of DAPTA, which are not present in PTA, play an important role in the extended structure of (2). Short, non-classical C–H···O interactions between O4 of an acetamide and H7 of a Cp ring (2.451 Å) link (2) into centrosymmetrical dimers (Fig. 5). A combination of non-classical hydrogen bonds involving acetamide groups link (2) in chains along [010]: O5···H11A (2.41 Å) and O4···H12B (2.60 Å) (Fig. 6). These chains are further joined into layers parallel to (101) through the molybdenum acetyl group via a non-classical C—H···O interaction between O3 and H13B (2.46 Å) (Fig. 7). The extensive network of interactions involving all three acetyl groups likely contribute to the low solubility of (2) in most organic solvents.

4. Database survey

The current version of the Cambridge Structural Database (Version 5.40, updated August 2019; Allen, 2002) has thirteen entries corresponding to molybdenum acyl complexes of the general form $Mo(C_5H_5)(CO)_2(PR_3)(COR)$. The *trans*-dicarbonyl structure, as observed for (1) and (2), 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).

The PTA and DAPTA ligands have not been extensively utilized on molybdenum. There are eight instances of the PTA ligand bonded to molybdenum or tungsten, five of which involve coordination of one or more of the ligand to a $M(CO)_n$ center to afford an octahedral product. Most relevant to this study is the tungsten complex $W(C_5H_5)(CO)_2(PTA)(H)$, which is analogous to (1) and (2) but features a hydride rather than an acyl ligand (Sears *et al.*, 2015).

5. Synthesis and crystallization

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H-atoms were treated in calculated positions and refined in the riding model approximation with distances of C—H = 0.93, 0.96, and 0.97 Å for the cyclopentadienyl, methyl, and methylene groups, respectively, and with U_{iso}(H) = k×U_{eq}(C), k = 1.2 for cyclopentadienyl 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 (one for (1); five for (2)) 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.

Table 1

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

Mo1—P1	2.4321 (5)	Mo1—C2	1.9746 (17)
Mo1—C1	1.9714 (17)	Mo1—C3	2.2398 (16)
C1—Mo1—P1	79.89 (5)	C2—Mo1—P1	78.80 (5)
C1—Mo1—C2	106.05 (7)	C2—Mo1—C3	78.51 (6)
C1—Mo1—C3	71.80 (6)	C3—Mo1—P1	136.77 (4)

Table 2

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

Mo1—P1	2.4258 (6)	Mo1—C2	1.971 (2)
Mo1—C1	1.964 (2)	Mo1—C3	2.243 (2)
C1—Mo1—P1	77.10 (7)	C2—Mo1—P1	81.75 (7)
C1—Mo1—C2	108.47 (10)	C2—Mo1—C3	76.10 (9)
C1—Mo1—C3	72.63 (9)	C3—Mo1—P1	133.99 (6)

Table 3

Experimental details

	(1)	(2)
Crystal data		
Chemical formula	$C_{15}H_{20}MoN_3O_3P$	$C_{18}H_{24}MoN_3O_5P$
M_r	417.25	489.31
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/n$
Temperature (K)	100	100
a, b, c (Å)	9.7919 (14), 14.5757 (13), 12.1140 (12)	12.8674 (4), 11.6366 (3), 14.4655 (5)
β (°)	107.694 (6)	113.224 (4)
V (Å^3)	1647.2 (3)	1990.45 (12)
Z	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$

μ (mm^{-1})	0.91	0.77
Crystal size (mm)	0.2 × 0.19 × 0.15	0.17 × 0.1 × 0.02
Data collection		
Diffractometer	Bruker D8QUEST	XtaLAB Synergy, Single source at offset/far, Pilatus 200K
Absorption correction	Multi-scan *SADABS2016*/2 (Bruker,2016/2) was used for absorption correction. wR2(int) was 0.0571 before and 0.0546 after correction. The ratio of minimum to maximum transmission is 0.9505. The λ/2 correction factor is not present.	Multi-scan *CrysAlis PRO* 1.171.40.61a (Rigaku Oxford Diffraction, 2019) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
T_{min}, T_{max}	0.709, 0.746	0.868, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	32227, 3785, 3379	38060, 4726, 4075
R_{int}	0.040	0.044
$(\sin \theta/\lambda)_{max}$ ($Å^{-1}$)	0.651	0.658
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.020, 0.043, 1.07	0.028, 0.061, 1.09
No. of reflections	3785	4726
No. of parameters	210	256
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e $Å^{-3}$)	0.45, −0.35	0.57, −0.37

Computer programs: *SAINT* V8.38A (Bruker, 2018), *CrysAlis PRO* 1.171.40.61a (Rigaku OD, 2019), SHELXT 2014/5 (Sheldrick, 2014), *SHELXL* 2018/3 (Sheldrick, 2015), Olex2 1.3-alpha (Dolomanov *et al.*, 2009).

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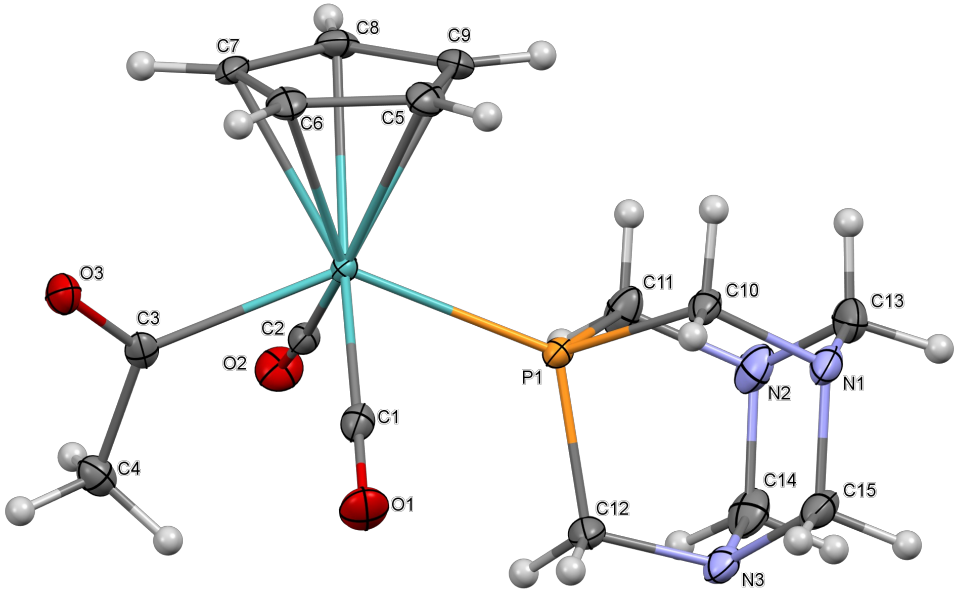

Figure 1

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

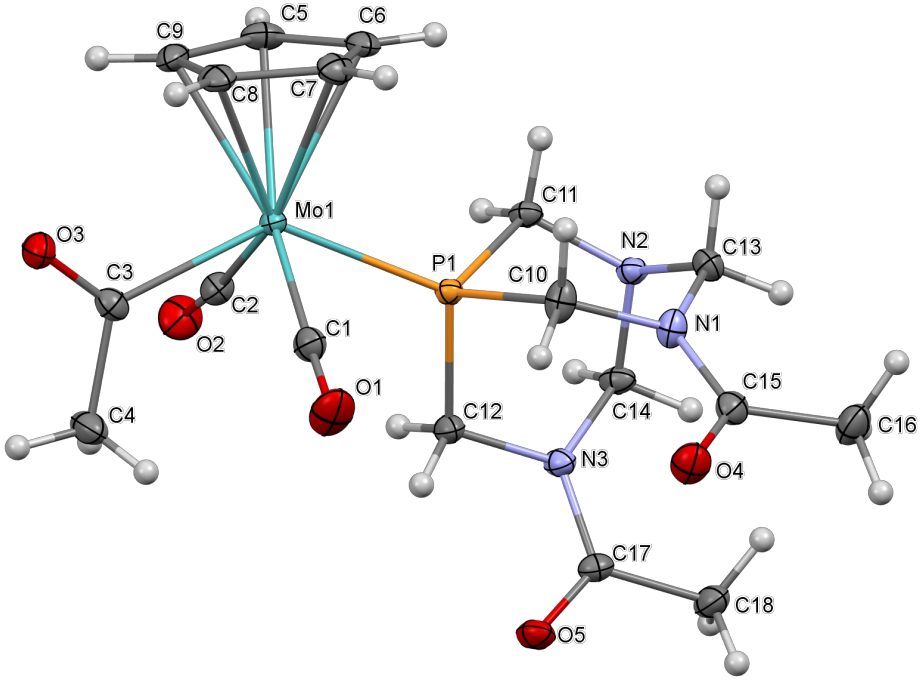

Figure 2

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

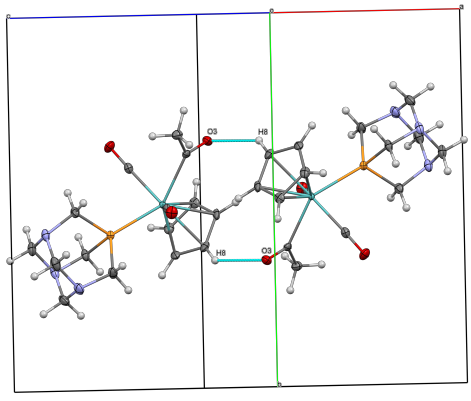

Figure 3

Centrosymmetrical dimer of (1), viewed perpendicular to (101).

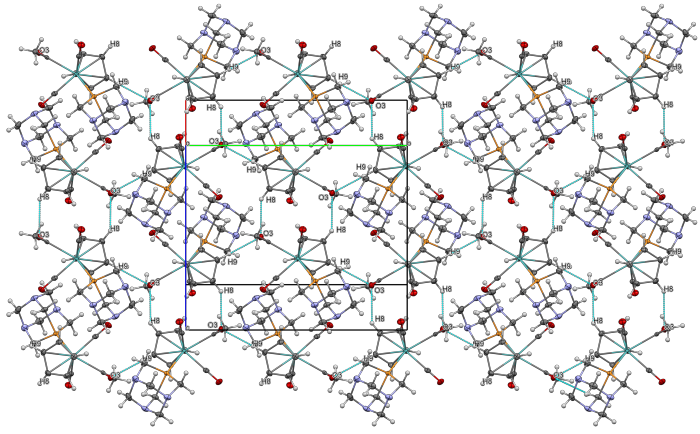

Figure 4

Layers of (1) formed by C—H···O interactions, viewed perpendicular to (100).

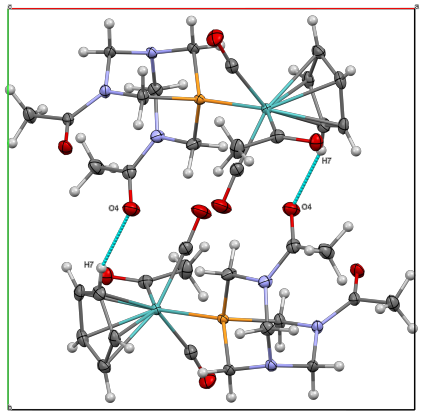

Figure 5

Centrosymmetrical dimer of (2), viewed parallel to (001).

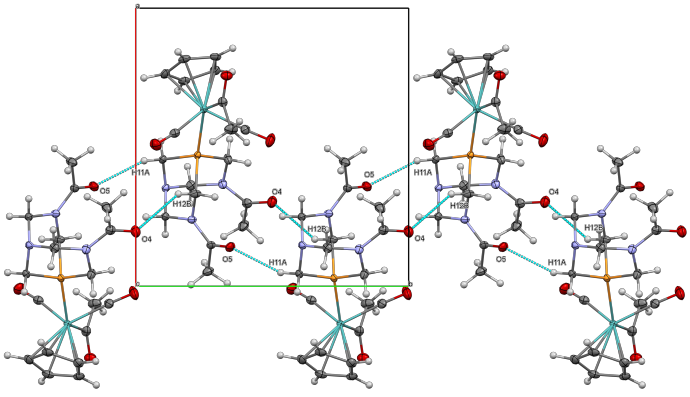

Figure 6

Chains of (2) along [010], viewed perpendicular to (010).

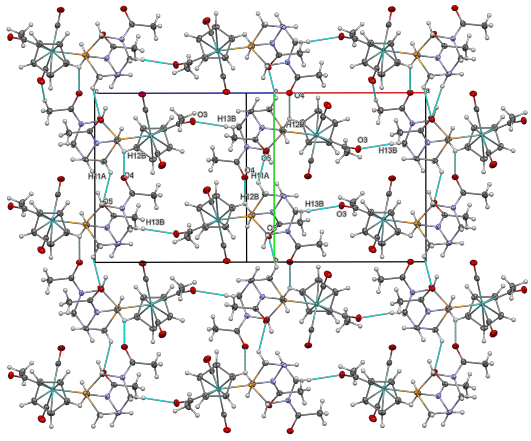

Figure 7

Layers of (2) formed by C—H···O interactions, viewed perpendicular to (101)

supporting information

Crystal Structures of *trans*-Acetyldicarbonyl(η^5-cyclopentadienyl)(1,3,5-tri–aza-7-phosphaadamantane)molybdenum(II) and *trans*-Acetyldicarbonyl(η^5-cyclopentadienyl)(3,7-diacetyl-1,3,7-triaza-5-phosphabicyclo–[3.3.1]nonane)molybdenum(II)

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Computing details

Data collection: *SAINT* V8.38A (Bruker, 2018) for (1); *CrysAlis PRO* 1.171.40.61a (Rigaku OD, 2019) for (2). Cell refinement: *SAINT* V8.38A (Bruker, 2018) for (1); *CrysAlis PRO* 1.171.40.61a (Rigaku OD, 2019) for (2). Data reduction: *SAINT* V8.38A (Bruker, 2018) for (1); *CrysAlis PRO* 1.171.40.61a (Rigaku OD, 2019) for (2). For both compounds, program(s) used to solve structure: SHELXT 2014/5 (Sheldrick, 2014); program(s) used to refine structure: *SHELXL* 2018/3 (Sheldrick, 2015); molecular graphics: Olex2 1.3-alpha (Dolomanov *et al.*, 2009); software used to prepare material for publication: Olex2 1.3-alpha (Dolomanov *et al.*, 2009).

(1)

Crystal data

$C_{15}H_{20}MoN_3O_3P$
M_r = 417.25
Monoclinic, $P2_1/c$
a = 9.7919 (14) Å
b = 14.5757 (13) Å
c = 12.1140 (12) Å
β = 107.694 (6)°
V = 1647.2 (3) Å^3
Z = 4
$F(000)$ = 848
D_x = 1.683 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9370 reflections
θ = 3.3–27.5°
μ = 0.91 mm^{-1}
T = 100 K
Block, orange
0.2 × 0.19 × 0.15 mm

Data collection

Bruker D8QUEST diffractometer
ω and φ scans
Absorption correction: multi-scan *SADABS2016*/2 (Bruker,2016/2) was used for absorption correction. wR2(int) was 0.0571 before and 0.0546 after correction. The ratio of minimum to maximum transmission is 0.9505. The λ/2 correction factor is not present.
T_{min} = 0.709, T_{max} = 0.746
32227 measured reflections
3785 independent reflections
3379 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 27.6°, θ_{min} = 3.3°
h = −12→12
k = −18→18
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.043
S = 1.07
3785 reflections
210 parameters
0 restraints
Primary atom site location: dual

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0124P)^2 + 1.3596P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}
Extinction correction: *SHELXL2018*/3 (Sheldrick 2018), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0040 (2)

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 (Å^2) for (1)

	x	*y*	*z*	U_{iso}*/U_{eq}
Mo1	0.59777 (2)	0.49328 (2)	0.28768 (2)	0.00886 (5)
P1	0.76523 (4)	0.41400 (3)	0.20808 (4)	0.01095 (9)
O1	0.65998 (14)	0.65083 (9)	0.13519 (11)	0.0225 (3)
O2	0.85977 (13)	0.47150 (9)	0.51067 (11)	0.0203 (3)
O3	0.47562 (13)	0.66109 (8)	0.37491 (10)	0.0174 (3)
N1	0.81148 (15)	0.31674 (11)	0.02647 (13)	0.0187 (3)
N2	0.95915 (17)	0.27288 (11)	0.22389 (14)	0.0216 (3)
N3	1.00813 (15)	0.41777 (10)	0.13718 (13)	0.0172 (3)
C1	0.64170 (17)	0.59232 (12)	0.19293 (14)	0.0145 (3)
C2	0.76722 (17)	0.48263 (11)	0.42615 (14)	0.0131 (3)
C3	0.59049 (18)	0.62704 (11)	0.37674 (13)	0.0126 (3)
C4	0.72533 (19)	0.67895 (11)	0.44048 (15)	0.0172 (3)
H4A	0.768353	0.651212	0.516872	0.026*
H4B	0.701269	0.743146	0.449920	0.026*
H4C	0.793647	0.676106	0.395903	0.026*
C5	0.39034 (18)	0.42909 (12)	0.16033 (15)	0.0165 (3)
H5	0.372255	0.432040	0.078793	0.020*
C6	0.35024 (17)	0.49564 (12)	0.22997 (14)	0.0155 (3)
H6	0.300765	0.551374	0.203612	0.019*
C7	0.39757 (17)	0.46382 (12)	0.34674 (15)	0.0155 (3)
H7	0.385727	0.495068	0.412043	0.019*
C8	0.46496 (18)	0.37807 (12)	0.34879 (15)	0.0164 (3)
H8	0.504922	0.340967	0.415265	0.020*
C9	0.46240 (18)	0.35724 (11)	0.23437 (15)	0.0164 (3)
H9	0.502247	0.304029	0.210865	0.020*
C10	0.69704 (17)	0.36231 (12)	0.06169 (14)	0.0155 (3)
H10A	0.621580	0.316952	0.061143	0.019*
H10B	0.653123	0.410872	0.004845	0.019*
C11	0.86374 (19)	0.31312 (12)	0.28477 (16)	0.0196 (4)
H11A	0.921558	0.331544	0.363817	0.023*
H11B	0.794116	0.266087	0.292213	0.023*
C12	0.91942 (17)	0.47689 (12)	0.18647 (15)	0.0163 (3)
H12A	0.883561	0.529431	0.133844	0.020*
H12B	0.979281	0.501540	0.261788	0.020*
C13	0.8764 (2)	0.24240 (13)	0.10693 (17)	0.0232 (4)
H13A	0.940468	0.206656	0.073719	0.028*
H13B	0.799353	0.200736	0.113403	0.028*

C14	1.06640 (18)	0.34022 (13)	0.21383 (16)	0.0220 (4)
H14A	1.116912	0.364261	0.292037	0.026*
H14B	1.138175	0.308452	0.184965	0.026*
C15	0.92521 (19)	0.38166 (13)	0.02367 (15)	0.0202 (4)
H15A	0.991757	0.350678	−0.011587	0.024*
H15B	0.881223	0.433817	−0.027049	0.024*

Atomic displacement parameters (Å²) for (1)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.00893 (7)	0.00963 (7)	0.00871 (7)	−0.00047 (5)	0.00374 (5)	0.00005 (5)
P1	0.01048 (19)	0.0127 (2)	0.01055 (19)	0.00010 (15)	0.00453 (15)	−0.00002 (15)
O1	0.0294 (7)	0.0204 (7)	0.0202 (7)	−0.0010 (5)	0.0112 (6)	0.0064 (5)
O2	0.0154 (6)	0.0258 (7)	0.0171 (6)	0.0012 (5)	0.0009 (5)	0.0017 (5)
O3	0.0188 (6)	0.0139 (6)	0.0211 (6)	0.0019 (5)	0.0087 (5)	−0.0011 (5)
N1	0.0138 (7)	0.0263 (8)	0.0182 (7)	−0.0026 (6)	0.0081 (6)	−0.0083 (6)
N2	0.0202 (7)	0.0230 (8)	0.0251 (8)	0.0084 (6)	0.0119 (7)	0.0022 (6)
N3	0.0120 (7)	0.0238 (8)	0.0176 (7)	−0.0007 (6)	0.0069 (6)	−0.0027 (6)
C1	0.0131 (8)	0.0179 (8)	0.0128 (8)	0.0013 (6)	0.0044 (6)	−0.0018 (6)
C2	0.0141 (8)	0.0124 (8)	0.0153 (8)	−0.0004 (6)	0.0082 (7)	−0.0005 (6)
C3	0.0193 (8)	0.0100 (7)	0.0094 (7)	0.0006 (6)	0.0057 (6)	0.0025 (6)
C4	0.0207 (9)	0.0139 (8)	0.0161 (8)	−0.0017 (7)	0.0042 (7)	0.0000 (6)
C5	0.0139 (8)	0.0212 (9)	0.0136 (8)	−0.0057 (7)	0.0027 (7)	−0.0039 (7)
C6	0.0083 (7)	0.0182 (8)	0.0188 (8)	−0.0017 (6)	0.0023 (6)	−0.0021 (7)
C7	0.0124 (8)	0.0205 (8)	0.0169 (8)	−0.0058 (6)	0.0094 (7)	−0.0049 (7)
C8	0.0164 (8)	0.0168 (8)	0.0170 (8)	−0.0062 (6)	0.0066 (7)	0.0021 (6)
C9	0.0167 (8)	0.0128 (8)	0.0226 (9)	−0.0057 (6)	0.0101 (7)	−0.0042 (7)
C10	0.0105 (7)	0.0222 (9)	0.0146 (8)	−0.0018 (6)	0.0051 (6)	−0.0047 (7)
C11	0.0217 (9)	0.0209 (9)	0.0190 (9)	0.0089 (7)	0.0105 (7)	0.0058 (7)
C12	0.0127 (8)	0.0181 (8)	0.0197 (8)	−0.0029 (6)	0.0071 (7)	−0.0043 (6)
C13	0.0218 (9)	0.0191 (9)	0.0328 (10)	0.0005 (7)	0.0143 (8)	−0.0081 (8)
C14	0.0126 (8)	0.0319 (10)	0.0212 (9)	0.0048 (7)	0.0047 (7)	−0.0025 (8)
C15	0.0154 (8)	0.0306 (10)	0.0173 (9)	−0.0029 (7)	0.0090 (7)	−0.0036 (7)

Geometric parameters (Å, º) for (1)

Mo1—P1	2.4321 (5)	C4—H4A	0.9800
Mo1—C1	1.9714 (17)	C4—H4B	0.9800
Mo1—C2	1.9746 (17)	C4—H4C	0.9800
Mo1—C3	2.2398 (16)	C5—H5	0.9500
Mo1—C5	2.3401 (16)	C5—C6	1.417 (2)
Mo1—C6	2.3097 (16)	C5—C9	1.419 (2)
Mo1—C7	2.3221 (16)	C6—H6	0.9500
Mo1—C8	2.3759 (16)	C6—C7	1.426 (2)
Mo1—C9	2.3634 (16)	C7—H7	0.9500
P1—C10	1.8543 (17)	C7—C8	1.410 (2)
P1—C11	1.8468 (17)	C8—H8	0.9500
P1—C12	1.8517 (17)	C8—C9	1.412 (2)
O1—C1	1.151 (2)	C9—H9	0.9500
O2—C2	1.154 (2)	C10—H10A	0.9900
O3—C3	1.223 (2)	C10—H10B	0.9900

N1—C10	1.473 (2)	C11—H11A	0.9900
N1—C13	1.467 (2)	C11—H11B	0.9900
N1—C15	1.470 (2)	C12—H12A	0.9900
N2—C11	1.476 (2)	C12—H12B	0.9900
N2—C13	1.471 (2)	C13—H13A	0.9900
N2—C14	1.469 (2)	C13—H13B	0.9900
N3—C12	1.473 (2)	C14—H14A	0.9900
N3—C14	1.464 (2)	C14—H14B	0.9900
N3—C15	1.466 (2)	C15—H15A	0.9900
C3—C4	1.514 (2)	C15—H15B	0.9900

C1—Mo1—P1	79.89 (5)	C6—C5—H5	126.2
C1—Mo1—C2	106.05 (7)	C6—C5—C9	107.66 (15)
C1—Mo1—C3	71.80 (6)	C9—C5—Mo1	73.34 (9)
C1—Mo1—C5	102.02 (6)	C9—C5—H5	126.2
C1—Mo1—C6	101.98 (6)	Mo1—C6—H6	119.7
C1—Mo1—C7	131.98 (6)	C5—C6—Mo1	73.43 (9)
C1—Mo1—C8	159.38 (6)	C5—C6—H6	126.2
C1—Mo1—C9	131.21 (6)	C5—C6—C7	107.62 (15)
C2—Mo1—P1	78.80 (5)	C7—C6—Mo1	72.55 (9)
C2—Mo1—C3	78.51 (6)	C7—C6—H6	126.2
C2—Mo1—C5	149.93 (6)	Mo1—C7—H7	119.7
C2—Mo1—C6	142.35 (6)	C6—C7—Mo1	71.60 (9)
C2—Mo1—C7	107.08 (6)	C6—C7—H7	125.8
C2—Mo1—C8	94.47 (6)	C8—C7—Mo1	74.63 (9)
C2—Mo1—C9	115.01 (6)	C8—C7—C6	108.37 (15)
C3—Mo1—P1	136.77 (4)	C8—C7—H7	125.8
C3—Mo1—C5	121.15 (6)	Mo1—C8—H8	122.9
C3—Mo1—C6	87.06 (6)	C7—C8—Mo1	70.46 (9)
C3—Mo1—C7	81.90 (6)	C7—C8—H8	126.1
C3—Mo1—C8	111.38 (6)	C7—C8—C9	107.74 (15)
C3—Mo1—C9	139.87 (6)	C9—C8—Mo1	72.19 (9)
C5—Mo1—P1	95.93 (4)	C9—C8—H8	126.1
C5—Mo1—C8	58.34 (6)	Mo1—C9—H9	121.3
C5—Mo1—C9	35.12 (6)	C5—C9—Mo1	71.54 (9)
C6—Mo1—P1	131.22 (4)	C5—C9—H9	125.7
C6—Mo1—C5	35.48 (6)	C8—C9—Mo1	73.15 (9)
C6—Mo1—C7	35.85 (6)	C8—C9—C5	108.60 (15)
C6—Mo1—C8	58.76 (6)	C8—C9—H9	125.7
C6—Mo1—C9	58.66 (6)	P1—C10—H10A	109.2
C7—Mo1—P1	140.20 (4)	P1—C10—H10B	109.2
C7—Mo1—C5	58.95 (6)	N1—C10—P1	112.07 (11)
C7—Mo1—C8	34.91 (6)	N1—C10—H10A	109.2
C7—Mo1—C9	58.20 (6)	N1—C10—H10B	109.2
C8—Mo1—P1	106.65 (4)	H10A—C10—H10B	107.9
C9—Mo1—P1	83.27 (4)	P1—C11—H11A	109.2
C9—Mo1—C8	34.66 (6)	P1—C11—H11B	109.2
C10—P1—Mo1	118.82 (5)	N2—C11—P1	112.21 (12)
C11—P1—Mo1	119.40 (6)	N2—C11—H11A	109.2
C11—P1—C10	97.98 (8)	N2—C11—H11B	109.2
C11—P1—C12	98.14 (8)	H11A—C11—H11B	107.9

C12—P1—Mo1	119.85 (6)	P1—C12—H12A	109.2
C12—P1—C10	97.93 (8)	P1—C12—H12B	109.2
C13—N1—C10	110.79 (14)	N3—C12—P1	111.98 (11)
C13—N1—C15	108.21 (14)	N3—C12—H12A	109.2
C15—N1—C10	111.40 (14)	N3—C12—H12B	109.2
C13—N2—C11	110.75 (14)	H12A—C12—H12B	107.9
C14—N2—C11	110.66 (14)	N1—C13—N2	114.68 (15)
C14—N2—C13	108.68 (14)	N1—C13—H13A	108.6
C14—N3—C12	111.03 (14)	N1—C13—H13B	108.6
C14—N3—C15	108.41 (14)	N2—C13—H13A	108.6
C15—N3—C12	111.33 (13)	N2—C13—H13B	108.6
O1—C1—Mo1	176.52 (15)	H13A—C13—H13B	107.6
O2—C2—Mo1	174.40 (14)	N2—C14—H14A	108.6
O3—C3—Mo1	120.54 (12)	N2—C14—H14B	108.6
O3—C3—C4	117.45 (15)	N3—C14—N2	114.69 (14)
C4—C3—Mo1	122.01 (11)	N3—C14—H14A	108.6
C3—C4—H4A	109.5	N3—C14—H14B	108.6
C3—C4—H4B	109.5	H14A—C14—H14B	107.6
C3—C4—H4C	109.5	N1—C15—H15A	108.6
H4A—C4—H4B	109.5	N1—C15—H15B	108.6
H4A—C4—H4C	109.5	N3—C15—N1	114.65 (14)
H4B—C4—H4C	109.5	N3—C15—H15A	108.6
Mo1—C5—H5	121.2	N3—C15—H15B	108.6
C6—C5—Mo1	71.09 (9)	H15A—C15—H15B	107.6

Mo1—P1—C10—N1	−179.79 (10)	C10—N1—C15—N3	66.86 (19)
Mo1—P1—C11—N2	179.31 (10)	C11—P1—C10—N1	−49.79 (14)
Mo1—P1—C12—N3	−179.75 (9)	C11—P1—C12—N3	49.41 (13)
Mo1—C5—C6—C7	−64.99 (11)	C11—N2—C13—N1	67.75 (19)
Mo1—C5—C9—C8	64.24 (12)	C11—N2—C14—N3	−67.81 (19)
Mo1—C6—C7—C8	−66.13 (11)	C12—P1—C10—N1	49.65 (14)
Mo1—C7—C8—C9	−62.98 (11)	C12—P1—C11—N2	−49.56 (14)
Mo1—C8—C9—C5	−63.20 (11)	C12—N3—C14—N2	68.04 (18)
C5—C6—C7—Mo1	65.58 (11)	C12—N3—C15—N1	−67.16 (19)
C5—C6—C7—C8	−0.55 (18)	C13—N1—C10—P1	60.54 (16)
C6—C5—C9—Mo1	−63.24 (11)	C13—N1—C15—N3	−55.17 (19)
C6—C5—C9—C8	1.00 (18)	C13—N2—C11—P1	−60.33 (18)
C6—C7—C8—Mo1	64.14 (11)	C13—N2—C14—N3	54.00 (19)
C6—C7—C8—C9	1.17 (18)	C14—N2—C11—P1	60.25 (17)
C7—C8—C9—Mo1	61.86 (11)	C14—N2—C13—N1	−54.01 (19)
C7—C8—C9—C5	−1.34 (18)	C14—N3—C12—P1	−60.30 (16)
C9—C5—C6—Mo1	64.71 (11)	C14—N3—C15—N1	55.25 (19)
C9—C5—C6—C7	−0.28 (18)	C15—N1—C10—P1	−59.98 (17)
C10—P1—C11—N2	49.70 (14)	C15—N1—C13—N2	54.43 (19)
C10—P1—C12—N3	−49.88 (13)	C15—N3—C12—P1	60.59 (17)
C10—N1—C13—N2	−67.97 (19)	C15—N3—C14—N2	−54.55 (19)

Hydrogen-bond geometry (Å, º) for (1)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O3^{i}	0.95	2.49	3.269 (2)	139

C9—H9···O3^{ii}	0.95	2.37	3.284 (2)	162

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1, *y*−1/2, −*z*+1/2.

(2)

Crystal data

$C_{18}H_{24}MoN_3O_5P$
M_r = 489.31
Monoclinic, $P2_1/n$
a = 12.8674 (4) Å
b = 11.6366 (3) Å
c = 14.4655 (5) Å
β = 113.224 (4)°
V = 1990.45 (12) Å^3
Z = 4
F(000) = 1000
D_x = 1.633 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 13952 reflections
θ = 2.5–31.0°
μ = 0.77 mm^{-1}
T = 100 K
Plate, clear colorless
0.17 × 0.1 × 0.02 mm

Data collection

XtaLAB Synergy, Single source at offset/far, Pilatus 200K diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Mo) X-ray Source
Mirror monochromator
Detector resolution: 5.8140 pixels mm^{-1}
ω scans
Absorption correction: multi-scan *CrysAlis PRO* 1.171.40.61a (Rigaku Oxford Diffraction, 2019) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
T_{min} = 0.868, T_{max} = 1.000
38060 measured reflections
4726 independent reflections
4075 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 27.9°, θ_{min} = 2.3°
h = −16→16
k = −15→15
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.061
S = 1.09
4726 reflections
256 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
$w = 1/[\sigma^2(F_o^2) + (0.0017P)^2 + 3.9699P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 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 (Å^2) for (2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Mo1	0.63575 (2)	0.25030 (2)	0.30169 (2)	0.01028 (5)
P1	0.47121 (4)	0.22642 (5)	0.34228 (4)	0.01101 (11)
O1	0.52463 (17)	0.49328 (16)	0.25544 (15)	0.0312 (4)
O2	0.50920 (15)	0.07353 (16)	0.13206 (13)	0.0270 (4)

O3	0.75732 (14)	0.33298 (15)	0.17091 (12)	0.0209 (4)
O4	0.30320 (15)	0.49974 (15)	0.41980 (13)	0.0242 (4)
O5	0.14095 (13)	0.34628 (14)	0.16401 (12)	0.0195 (3)
N1	0.36745 (16)	0.31852 (17)	0.46343 (14)	0.0165 (4)
N2	0.35305 (15)	0.11049 (16)	0.43014 (13)	0.0135 (4)
N3	0.23918 (15)	0.20618 (16)	0.27035 (14)	0.0147 (4)
C1	0.5628 (2)	0.4017 (2)	0.26999 (18)	0.0185 (5)
C2	0.55033 (19)	0.1431 (2)	0.19267 (17)	0.0173 (5)
C3	0.66223 (19)	0.3187 (2)	0.16760 (16)	0.0163 (4)
C4	0.5638 (2)	0.3496 (2)	0.06996 (18)	0.0239 (5)
H4A	0.519961	0.281951	0.041945	0.036*
H4B	0.592380	0.380455	0.023061	0.036*
H4C	0.516914	0.405872	0.083315	0.036*
C5	0.76382 (19)	0.1081 (2)	0.39905 (17)	0.0197 (5)
H5	0.748692	0.030017	0.387962	0.024*
C6	0.73813 (19)	0.1777 (2)	0.46709 (16)	0.0179 (5)
H6	0.702176	0.153239	0.508153	0.022*
C7	0.77602 (19)	0.2908 (2)	0.46259 (17)	0.0184 (5)
H7	0.770231	0.353153	0.500496	0.022*
C8	0.82445 (18)	0.2920 (2)	0.38987 (17)	0.0188 (5)
H8	0.855613	0.355473	0.371134	0.023*
C9	0.81675 (19)	0.1787 (2)	0.35077 (17)	0.0192 (5)
H9	0.842145	0.155039	0.301873	0.023*
C10	0.4569 (2)	0.3377 (2)	0.42664 (18)	0.0184 (5)
H10A	0.528327	0.344180	0.484207	0.022*
H10B	0.442864	0.410721	0.391445	0.022*
C11	0.45578 (18)	0.09925 (19)	0.41018 (16)	0.0135 (4)
H11A	0.450942	0.030898	0.370298	0.016*
H11B	0.521186	0.091764	0.473140	0.016*
C12	0.33213 (18)	0.2198 (2)	0.23700 (17)	0.0168 (5)
H12A	0.320423	0.289683	0.197633	0.020*
H12B	0.331263	0.155760	0.193769	0.020*
C13	0.3622 (2)	0.2021 (2)	0.50113 (17)	0.0170 (5)
H13A	0.429693	0.189409	0.561541	0.020*
H13B	0.297707	0.197899	0.519767	0.020*
C14	0.25059 (18)	0.10856 (19)	0.33896 (17)	0.0151 (4)
H14A	0.185886	0.108202	0.357482	0.018*
H14B	0.248845	0.037642	0.303134	0.018*
C15	0.29827 (19)	0.4079 (2)	0.46058 (17)	0.0176 (5)
C16	0.2176 (2)	0.3947 (2)	0.5127 (2)	0.0250 (5)
H16A	0.259315	0.397149	0.584258	0.037*
H16B	0.178938	0.322462	0.494274	0.037*
H16C	0.163490	0.456177	0.492731	0.037*
C17	0.14790 (18)	0.27602 (19)	0.22978 (16)	0.0152 (4)
C18	0.05350 (19)	0.2655 (2)	0.26638 (18)	0.0216 (5)
H18A	0.010423	0.335387	0.252252	0.032*
H18B	0.084905	0.251691	0.337578	0.032*
H18C	0.005112	0.202610	0.232574	0.032*

Atomic displacement parameters (Å²) for (2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.00936 (8)	0.01073 (9)	0.01029 (8)	0.00077 (7)	0.00339 (6)	0.00039 (7)
P1	0.0108 (2)	0.0103 (3)	0.0115 (2)	0.00093 (19)	0.0039 (2)	0.00144 (19)
O1	0.0374 (11)	0.0174 (9)	0.0469 (12)	0.0094 (8)	0.0253 (10)	0.0102 (8)
O2	0.0255 (9)	0.0273 (10)	0.0249 (9)	−0.0042 (8)	0.0063 (8)	−0.0112 (8)
O3	0.0181 (8)	0.0268 (9)	0.0193 (8)	−0.0005 (7)	0.0088 (7)	0.0017 (7)
O4	0.0283 (9)	0.0172 (9)	0.0308 (10)	0.0027 (7)	0.0158 (8)	0.0035 (7)
O5	0.0175 (8)	0.0192 (9)	0.0203 (8)	0.0031 (7)	0.0059 (7)	0.0087 (7)
N1	0.0185 (9)	0.0144 (10)	0.0204 (10)	−0.0016 (7)	0.0117 (8)	−0.0020 (7)
N2	0.0118 (9)	0.0135 (9)	0.0143 (9)	0.0015 (7)	0.0042 (7)	0.0042 (7)
N3	0.0113 (8)	0.0164 (9)	0.0155 (9)	0.0001 (7)	0.0041 (7)	0.0037 (7)
C1	0.0183 (11)	0.0194 (12)	0.0197 (11)	−0.0009 (9)	0.0096 (9)	0.0032 (9)
C2	0.0144 (10)	0.0199 (12)	0.0179 (11)	0.0017 (9)	0.0066 (9)	0.0013 (9)
C3	0.0192 (11)	0.0156 (11)	0.0136 (10)	−0.0007 (9)	0.0060 (9)	−0.0003 (8)
C4	0.0195 (12)	0.0338 (14)	0.0164 (11)	−0.0006 (10)	0.0048 (10)	0.0068 (10)
C5	0.0172 (11)	0.0161 (12)	0.0193 (11)	0.0047 (9)	0.0002 (9)	0.0023 (9)
C6	0.0122 (10)	0.0251 (12)	0.0126 (10)	0.0029 (9)	0.0008 (8)	0.0057 (9)
C7	0.0170 (11)	0.0209 (12)	0.0132 (10)	0.0007 (9)	0.0016 (9)	−0.0031 (9)
C8	0.0101 (10)	0.0258 (12)	0.0164 (11)	−0.0005 (9)	0.0008 (9)	0.0027 (9)
C9	0.0120 (10)	0.0280 (13)	0.0154 (11)	0.0058 (9)	0.0031 (9)	−0.0007 (9)
C10	0.0219 (12)	0.0134 (11)	0.0248 (12)	−0.0041 (9)	0.0144 (10)	−0.0041 (9)
C11	0.0122 (10)	0.0121 (10)	0.0146 (10)	0.0015 (8)	0.0037 (8)	0.0032 (8)
C12	0.0127 (10)	0.0217 (12)	0.0152 (10)	0.0020 (8)	0.0048 (8)	0.0041 (8)
C13	0.0183 (11)	0.0187 (11)	0.0154 (11)	0.0013 (9)	0.0081 (9)	0.0032 (9)
C14	0.0120 (10)	0.0131 (11)	0.0179 (11)	−0.0006 (8)	0.0034 (9)	0.0038 (8)
C15	0.0177 (11)	0.0194 (12)	0.0147 (10)	−0.0006 (9)	0.0054 (9)	−0.0025 (9)
C16	0.0245 (13)	0.0229 (13)	0.0336 (14)	0.0066 (10)	0.0180 (11)	0.0043 (10)
C17	0.0122 (10)	0.0151 (11)	0.0150 (10)	0.0003 (8)	0.0019 (8)	−0.0007 (8)
C18	0.0156 (10)	0.0251 (13)	0.0252 (12)	0.0051 (10)	0.0091 (9)	0.0079 (10)

Geometric parameters (Å, º) for (2)

Mo1—P1	2.4258 (6)	C4—H4C	0.9600
Mo1—C1	1.964 (2)	C5—H5	0.9300
Mo1—C2	1.971 (2)	C5—C6	1.411 (3)
Mo1—C3	2.243 (2)	C5—C9	1.415 (3)
Mo1—C5	2.368 (2)	C6—H6	0.9300
Mo1—C6	2.385 (2)	C6—C7	1.413 (3)
Mo1—C7	2.363 (2)	C7—H7	0.9300
Mo1—C8	2.306 (2)	C7—C8	1.419 (3)
Mo1—C9	2.306 (2)	C8—H8	0.9300
P1—C10	1.838 (2)	C8—C9	1.423 (3)
P1—C11	1.830 (2)	C9—H9	0.9300
P1—C12	1.838 (2)	C10—H10A	0.9700
O1—C1	1.157 (3)	C10—H10B	0.9700
O2—C2	1.157 (3)	C11—H11A	0.9700
O3—C3	1.217 (3)	C11—H11B	0.9700
O4—C15	1.234 (3)	C12—H12A	0.9700
O5—C17	1.231 (3)	C12—H12B	0.9700

N1—C10	1.463 (3)	C13—H13A	0.9700
N1—C13	1.471 (3)	C13—H13B	0.9700
N1—C15	1.359 (3)	C14—H14A	0.9700
N2—C11	1.466 (3)	C14—H14B	0.9700
N2—C13	1.453 (3)	C15—C16	1.512 (3)
N2—C14	1.451 (3)	C16—H16A	0.9600
N3—C12	1.464 (3)	C16—H16B	0.9600
N3—C14	1.477 (3)	C16—H16C	0.9600
N3—C17	1.357 (3)	C17—C18	1.509 (3)
C3—C4	1.522 (3)	C18—H18A	0.9600
C4—H4A	0.9600	C18—H18B	0.9600
C4—H4B	0.9600	C18—H18C	0.9600

C1—Mo1—P1	77.10 (7)	C5—C6—H6	125.6
C1—Mo1—C2	108.47 (10)	C5—C6—C7	108.8 (2)
C1—Mo1—C3	72.63 (9)	C7—C6—Mo1	71.84 (13)
C1—Mo1—C5	157.27 (9)	C7—C6—H6	125.6
C1—Mo1—C6	125.17 (9)	Mo1—C7—H7	121.9
C1—Mo1—C7	99.22 (9)	C6—C7—Mo1	73.52 (13)
C1—Mo1—C8	104.03 (9)	C6—C7—H7	126.2
C1—Mo1—C9	136.72 (9)	C6—C7—C8	107.7 (2)
C2—Mo1—P1	81.75 (7)	C8—C7—Mo1	70.12 (12)
C2—Mo1—C3	76.10 (9)	C8—C7—H7	126.2
C2—Mo1—C5	94.08 (9)	Mo1—C8—H8	119.1
C2—Mo1—C6	119.45 (9)	C7—C8—Mo1	74.54 (13)
C2—Mo1—C7	151.96 (9)	C7—C8—H8	126.2
C2—Mo1—C8	135.18 (9)	C7—C8—C9	107.6 (2)
C2—Mo1—C9	101.20 (9)	C9—C8—Mo1	72.05 (13)
C3—Mo1—P1	133.99 (6)	C9—C8—H8	126.2
C3—Mo1—C5	117.05 (8)	Mo1—C9—H9	119.2
C3—Mo1—C6	141.34 (8)	C5—C9—Mo1	74.77 (13)
C3—Mo1—C7	117.61 (8)	C5—C9—C8	108.3 (2)
C3—Mo1—C8	85.28 (8)	C5—C9—H9	125.9
C3—Mo1—C9	85.13 (8)	C8—C9—Mo1	72.00 (13)
C5—Mo1—P1	104.19 (6)	C8—C9—H9	125.9
C5—Mo1—C6	34.54 (8)	P1—C10—H10A	108.4
C6—Mo1—P1	84.62 (6)	P1—C10—H10B	108.4
C7—Mo1—P1	100.66 (6)	N1—C10—P1	115.67 (16)
C7—Mo1—C5	58.09 (8)	N1—C10—H10A	108.4
C7—Mo1—C6	34.64 (8)	N1—C10—H10B	108.4
C8—Mo1—P1	136.01 (6)	H10A—C10—H10B	107.4
C8—Mo1—C5	58.94 (9)	P1—C11—H11A	109.8
C8—Mo1—C6	58.33 (8)	P1—C11—H11B	109.8
C8—Mo1—C7	35.35 (8)	N2—C11—P1	109.32 (14)
C8—Mo1—C9	35.95 (9)	N2—C11—H11A	109.8
C9—Mo1—P1	139.10 (6)	N2—C11—H11B	109.8
C9—Mo1—C5	35.20 (9)	H11A—C11—H11B	108.3
C9—Mo1—C6	58.12 (8)	P1—C12—H12A	109.0
C9—Mo1—C7	58.84 (8)	P1—C12—H12B	109.0
C10—P1—Mo1	113.94 (8)	N3—C12—P1	112.74 (15)
C11—P1—Mo1	120.83 (7)	N3—C12—H12A	109.0

C11—P1—C10	98.79 (11)	N3—C12—H12B	109.0
C11—P1—C12	97.73 (10)	H12A—C12—H12B	107.8
C12—P1—Mo1	117.51 (7)	N1—C13—H13A	108.6
C12—P1—C10	105.05 (11)	N1—C13—H13B	108.6
C10—N1—C13	115.65 (18)	N2—C13—N1	114.62 (18)
C15—N1—C10	118.14 (19)	N2—C13—H13A	108.6
C15—N1—C13	126.21 (19)	N2—C13—H13B	108.6
C13—N2—C11	112.11 (17)	H13A—C13—H13B	107.6
C14—N2—C11	112.71 (17)	N2—C14—N3	114.37 (18)
C14—N2—C13	116.44 (18)	N2—C14—H14A	108.7
C12—N3—C14	115.27 (17)	N2—C14—H14B	108.7
C17—N3—C12	118.24 (18)	N3—C14—H14A	108.7
C17—N3—C14	126.23 (19)	N3—C14—H14B	108.7
O1—C1—Mo1	176.4 (2)	H14A—C14—H14B	107.6
O2—C2—Mo1	173.5 (2)	O4—C15—N1	121.4 (2)
O3—C3—Mo1	120.51 (16)	O4—C15—C16	120.1 (2)
O3—C3—C4	117.4 (2)	N1—C15—C16	118.5 (2)
C4—C3—Mo1	122.10 (16)	C15—C16—H16A	109.5
C3—C4—H4A	109.5	C15—C16—H16B	109.5
C3—C4—H4B	109.5	C15—C16—H16C	109.5
C3—C4—H4C	109.5	H16A—C16—H16B	109.5
H4A—C4—H4B	109.5	H16A—C16—H16C	109.5
H4A—C4—H4C	109.5	H16B—C16—H16C	109.5
H4B—C4—H4C	109.5	O5—C17—N3	121.3 (2)
Mo1—C5—H5	122.1	O5—C17—C18	120.1 (2)
C6—C5—Mo1	73.39 (13)	N3—C17—C18	118.6 (2)
C6—C5—H5	126.2	C17—C18—H18A	109.5
C6—C5—C9	107.6 (2)	C17—C18—H18B	109.5
C9—C5—Mo1	70.03 (13)	C17—C18—H18C	109.5
C9—C5—H5	126.2	H18A—C18—H18B	109.5
Mo1—C6—H6	122.2	H18A—C18—H18C	109.5
C5—C6—Mo1	72.07 (13)	H18B—C18—H18C	109.5

Mo1—P1—C10—N1	−172.38 (14)	C11—P1—C10—N1	−42.89 (19)
Mo1—P1—C11—N2	176.42 (11)	C11—P1—C12—N3	49.62 (18)
Mo1—P1—C12—N3	−179.56 (13)	C11—N2—C13—N1	67.4 (2)
Mo1—C5—C6—C7	−62.85 (16)	C11—N2—C14—N3	−64.3 (2)
Mo1—C5—C9—C8	64.72 (15)	C12—P1—C10—N1	57.6 (2)
Mo1—C6—C7—C8	−62.18 (15)	C12—P1—C11—N2	−54.99 (16)
Mo1—C7—C8—C9	−64.94 (15)	C12—N3—C14—N2	57.2 (3)
Mo1—C8—C9—C5	−66.55 (15)	C12—N3—C17—O5	2.6 (3)
C5—C6—C7—Mo1	63.00 (15)	C12—N3—C17—C18	−177.9 (2)
C5—C6—C7—C8	0.8 (2)	C13—N1—C10—P1	46.5 (2)
C6—C5—C9—Mo1	−64.27 (15)	C13—N1—C15—O4	−173.8 (2)
C6—C5—C9—C8	0.5 (2)	C13—N1—C15—C16	8.8 (3)
C6—C7—C8—Mo1	64.41 (15)	C13—N2—C11—P1	−67.49 (19)
C6—C7—C8—C9	−0.5 (2)	C13—N2—C14—N3	67.3 (2)
C7—C8—C9—Mo1	66.60 (15)	C14—N2—C11—P1	66.2 (2)
C7—C8—C9—C5	0.0 (2)	C14—N2—C13—N1	−64.5 (2)
C9—C5—C6—Mo1	62.07 (15)	C14—N3—C12—P1	−53.5 (2)
C9—C5—C6—C7	−0.8 (2)	C14—N3—C17—O5	−171.1 (2)

C10—P1—C11—N2	51.64 (16)	C14—N3—C17—C18	8.4 (3)
C10—P1—C12—N3	−51.70 (19)	C15—N1—C10—P1	−133.89 (18)
C10—N1—C13—N2	−55.1 (3)	C15—N1—C13—N2	125.4 (2)
C10—N1—C15—O4	6.6 (3)	C17—N3—C12—P1	132.06 (18)
C10—N1—C15—C16	−170.7 (2)	C17—N3—C14—N2	−128.9 (2)

Hydrogen-bond geometry (Å, º) for (2)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O4^{i}	0.93	2.45	3.353 (3)	163
C11—H11*A*···O5^{ii}	0.97	2.41	3.211 (3)	140
C12—H12*B*···O4^{ii}	0.97	2.60	3.409 (3)	141
C13—H13*A*···O5^{iii}	0.97	2.57	3.474 (3)	156
C13—H13*B*···O3^{iv}	0.97	2.46	3.261 (3)	139

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1/2, *y*−1/2, −*z*+1/2; (iii) *x*+1/2, −*y*+1/2, *z*+1/2; (iv) *x*−1/2, −*y*+1/2, *z*+1/2.