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# Tetrakis($\mu_2$-diphenylphosphinato-$\kappa^2O,O'$)tetra-$\mu_3$-oxido-tetraoxidohexamolybdenum(V)

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**Keywords:** crystal structure; heterocubane; molybdenum; $Mo_4O_4$ core.

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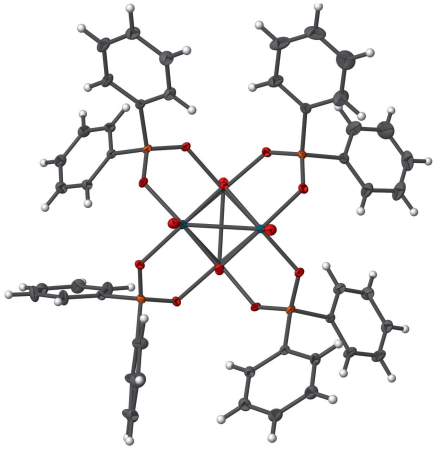
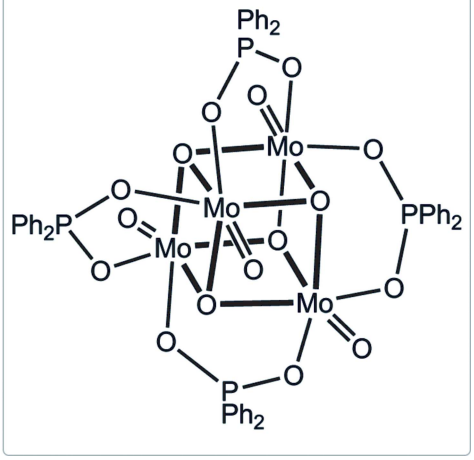
**Structural data**: full structural data are available from iucrdata.iucr.org

The molecule of the title compound, $[Mo_4(\mu_2\text{-}C_{12}H_{10}OP_2)_4(\mu_3\text{-}O)_4O_4]$, exhibits point group symmetry 2 with the twofold rotation axis passing through two opposite P atoms. Each $Mo^V$ atom is bridged by three O atoms resulting in an $Mo_4O_4$ heterocubane core. In the crystal, weak C—H···O interactions may help to consolidate packing of the molecules.

**3D view**



**Chemical scheme**




## Structure description

The title compound had formed accidentally due to the presence of oxygen in a solution originally intended to crystallize the PNP pincer compound $[Mo(PNP^{Me}—Ph)(CO)_2F_2]$ [$(PNP^{Me}—Ph)$ = *N*,*N*′-bis(diphenylphosphino)-*N*,*N*′-methyl-2,6-diaminopyridine; de Aguiar *et al.*, 2014]. A chloroform disolvate of the title compound was reported by Schirmer *et al.* (1989). The molecule of the title compound $[MoO(\mu_3\text{-}O)(\mu_2\text{-}C_{12}H_{10}PO_2)]_4$, exhibits point group symmetry 2 with the twofold rotation axis passing through two opposite P atoms (Fig. 1). Each molybdenum atom is bridged by three oxygen atoms resulting in a $Mo_4O_4$ heterocubane core. The distorted octahedral coordination spheres of the two unique molybdenum atoms [bond lengths range from 1.6686 (19) to 2.3982 (19) Å] are completed by a double-bonded terminal oxygen atom and two oxygen atoms of two diphenylphosphinate anions, each bridging two opposite molybdenum atoms in the heterocube. The short Mo···Mo distance of 2.6395 (3) Å indicates $Mo^V\cdots Mo^V$ interactions and causes a distortion of the $Mo_4O_4$ heterocubane core with O—Mo—O angles ranging from 77.94 (7) to 89.87 (7)° and Mo—O—Mo angles from 84.60 (7) to 102.02 (7) °. For characteristic bond lengths and angles of $\{Mo_4O_4(\mu_3\text{-}$

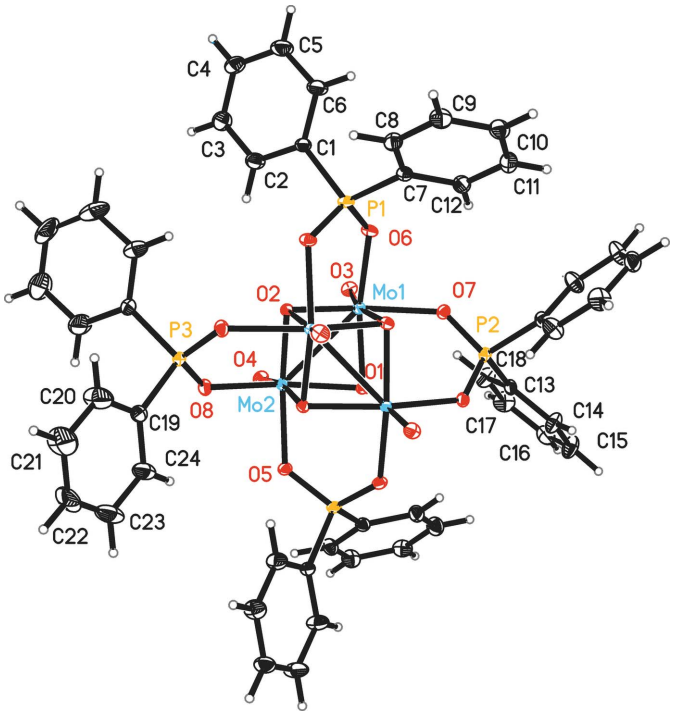



**Figure 1**
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level; H atoms are shown as spheres of arbitrary radius. Non-labelled atoms are generated by the symmetry code $-x + 2, y, -z + \frac{1}{2}$.

$O_4\}^{4+}$ heterocubane cores, see: Modec *et al.* (2003). In the crystal, weak C—H···O interactions, Table 1, may help to consolidate packing of the molecules, Fig. 2.

## Synthesis and crystallization

The original intention was to crystallize the compound [Mo(PNP$^{Me}$—Ph)(CO)$_2$F$_2$] (de Aguiar *et al.*, 2014). One equivalent of 1-fluoro-2,4,6-trimethylpyridinium tetrafluoridoborate was added to a solution of [Mo(PNP$^{Me}$—Ph)(CO)$_3$] in 10 ml $CH_2Cl_2$ and stirred for three days. After filtration, the solution was layered with pentane and left to stand for several days. Instead of the desired compound [Mo(PNP$^{Me}$—Ph)(CO)$_2$F$_2$], the title compound crystallized in form of dark-red blocks, apparently caused by the presence of larger amounts of oxygen in the reaction vessel.

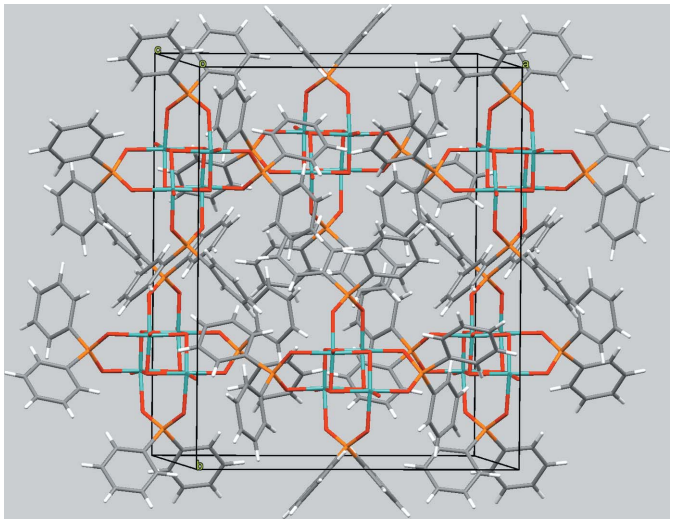


**Figure 2**
The packing of the molecules in the crystal structure of the title compound in a view approximately along [001].

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C4—H4···O3$^{i}$ | 0.93 | 2.52 | 3.188 (4) | 129 |
| C21—H21···O4$^{ii}$ | 0.93 | 2.57 | 3.420 (5) | 152 |

Symmetry codes: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, -z$; (ii) $x, -y, z + \frac{1}{2}$.

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

## Acknowledgements

Financial support by the Austrian Science Fund (FWF) (Project No. P24202-N17) is gratefully acknowledged. The

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $[Mo_4(C_{12}H_{10}O_2P)_4O_8]$ |
| $M_r$ | 1380.44 |
| Crystal system, space group | Orthorhombic, *Pbcn* |
| Temperature (K) | 100 |
| *a*, *b*, *c* (Å) | 16.1061 (19), 19.891 (2), 15.254 (3) |
| *V* (Å$^3$) | 4887.0 (11) |
| *Z* | 4 |
| Radiation type | Mo *Kα* |
| $\mu$ (mm$^{-1}$) | 1.21 |
| Crystal size (mm) | 0.61 × 0.49 × 0.30 |
| | |
| Data collection | |
| Diffractometer | Bruker Kappa APEXII CCD diffractometer |
| Absorption correction | Multi-scan (*SADABS*; Bruker, 2014) |
| $T_{min}$, $T_{max}$ | 0.45, 0.66 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 255304, 8962, 7558 |
| $R_{int}$ | 0.061 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.760 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.039, 0.090, 1.27 |
| No. of reflections | 8962 |
| No. of parameters | 326 |
| H-atom treatment | H-atom parameters constrained $w = 1/[\sigma^2(F_o^2) + 23.6442P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.92, −1.05 |

Computer programs: *APEX2* (Bruker, 2014), *SAINT* (Bruker, 2014), *SUPERFLIP* (Palatinus & Chapuis, 2007), *SHELXL2014* (Sheldrick, 2015), *XP* in *SHELXTL* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2006), *publCIF* (Westrip, 2010).

X-ray centre of TU Wien is acknowledged for providing access to the single-crystal diffractometer.

## References

Aguiar, S. R. M. M. de, Stöger, B., Pittenauer, E., Puchberger, M., Allmaier, G., Veiros, L. F. & Kirchner, K. (2014). *J. Organomet. Chem.* **760**, 74–83.
Bruker (2014). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Modec, B., Brenčič, J., Burkholder, E. M. & Zubieta, J. (2003). *Dalton Trans.* pp. 4618–4625.
Palatinus, L. & Chapuis, G. (2007). *J. Appl. Cryst.* **40**, 786–790.
Schirmer, W., Flörke, U. & Haupt, H.-J. (1989). *Z. Anorg. Allg. Chem.* **574**, 239–255.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

# full crystallographic data

*IUCrData* (2016). **1**, x160036 [doi:10.1107/S2414314616000365]

## Tetrakis($\mu_2$-diphenylphosphinato-$\kappa^2O,O'$)tetra-$\mu_3$-oxido-tetraoxidohexamolybdenum(V)

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**Tetrakis($\mu_2$-diphenylphosphinato-$\kappa^2O,O'$)tetra-$\mu_3$-oxido-tetraoxidohexamolybdenum(V)**

*Crystal data*

$[Mo_4(C_{12}H_{10}O_2P)_4O_8]$
$M_r$ = 1380.44
Orthorhombic, *Pbcn*
*a* = 16.1061 (19) Å
*b* = 19.891 (2) Å
*c* = 15.254 (3) Å
*V* = 4887.0 (11) Å$^3$
*Z* = 4
*F*(000) = 2736
$D_x$ = 1.876 Mg m$^{-3}$
Mo *Kα* radiation, $\lambda$ = 0.71073 Å
Cell parameters from 9220 reflections
$\theta$ = 2.4–30.0°
$\mu$ = 1.21 mm$^{-1}$
*T* = 100 K
Block, dark red
0.61 × 0.49 × 0.30 mm

*Data collection*

Bruker Kappa APEXII CCD diffractometer
Graphite monochromator
$\omega$– and $\varphi$–scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
$T_{min}$ = 0.45, $T_{max}$ = 0.66
255304 measured reflections
8962 independent reflections
7558 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.061
$\theta_{max}$ = 32.7°, $\theta_{min}$ = 1.6°
*h* = −24→24
*k* = −30→30
*l* = −23→23

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.090
*S* = 1.27
8962 reflections
326 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + 23.6442P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.92 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.05 e Å$^{-3}$

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Mo1 | 0.94264 (2) | 0.30980 (2) | 0.15695 (2) | 0.00840 (5) |
| Mo2 | 1.05855 (2) | 0.21599 (2) | 0.15724 (2) | 0.00870 (5) |
| P1 | 0.76853 (4) | 0.26945 (3) | 0.25500 (4) | 0.01124 (11) |
| P2 | 1.0000 | 0.45135 (4) | 0.2500 | 0.01029 (15) |
| P3 | 1.0000 | 0.07459 (5) | 0.2500 | 0.01125 (15) |
| O1 | 1.06085 (11) | 0.31230 (9) | 0.18613 (12) | 0.0102 (3) |
| O2 | 0.93968 (11) | 0.21393 (9) | 0.18558 (12) | 0.0095 (3) |
| O3 | 0.93008 (12) | 0.31794 (10) | 0.04868 (12) | 0.0133 (3) |
| O4 | 1.07105 (12) | 0.20672 (10) | 0.04932 (12) | 0.0142 (3) |
| O5 | 1.18201 (11) | 0.21577 (10) | 0.19500 (13) | 0.0121 (3) |
| O6 | 0.81967 (11) | 0.31237 (10) | 0.19196 (12) | 0.0120 (3) |
| O7 | 0.94434 (12) | 0.41077 (9) | 0.18832 (12) | 0.0114 (3) |
| O8 | 1.05526 (12) | 0.11516 (9) | 0.18828 (13) | 0.0133 (3) |
| C1 | 0.69240 (16) | 0.22699 (13) | 0.19004 (18) | 0.0136 (4) |
| C2 | 0.72255 (18) | 0.17894 (16) | 0.1312 (2) | 0.0199 (5) |
| H2 | 0.7791 | 0.1696 | 0.1289 | 0.024* |
| C3 | 0.6680 (2) | 0.14529 (17) | 0.0762 (2) | 0.0226 (6) |
| H3 | 0.6880 | 0.1134 | 0.0369 | 0.027* |
| C4 | 0.58371 (19) | 0.15910 (17) | 0.0796 (2) | 0.0218 (6) |
| H4 | 0.5471 | 0.1361 | 0.0433 | 0.026* |
| C5 | 0.5542 (2) | 0.20701 (18) | 0.1370 (2) | 0.0260 (6) |
| H5 | 0.4976 | 0.2162 | 0.1389 | 0.031* |
| C6 | 0.60803 (18) | 0.24191 (17) | 0.1924 (2) | 0.0212 (5) |
| H6 | 0.5878 | 0.2747 | 0.2303 | 0.025* |
| C7 | 0.71892 (16) | 0.32266 (13) | 0.33376 (17) | 0.0128 (4) |
| C8 | 0.66756 (17) | 0.29415 (14) | 0.39712 (19) | 0.0166 (5) |
| H8 | 0.6589 | 0.2479 | 0.3977 | 0.020* |
| C9 | 0.62922 (19) | 0.33445 (16) | 0.4595 (2) | 0.0208 (5) |
| H9 | 0.5942 | 0.3153 | 0.5010 | 0.025* |
| C10 | 0.64329 (19) | 0.40329 (16) | 0.4597 (2) | 0.0207 (5) |
| H10 | 0.6179 | 0.4303 | 0.5017 | 0.025* |
| C11 | 0.6953 (2) | 0.43199 (15) | 0.3972 (2) | 0.0215 (5) |
| H11 | 0.7045 | 0.4782 | 0.3975 | 0.026* |
| C12 | 0.73355 (18) | 0.39208 (14) | 0.3343 (2) | 0.0175 (5) |
| H12 | 0.7686 | 0.4113 | 0.2928 | 0.021* |
| C13 | 1.06447 (16) | 0.50353 (13) | 0.18254 (17) | 0.0130 (4) |
| C14 | 1.1143 (2) | 0.55275 (15) | 0.2199 (2) | 0.0220 (6) |
| H14 | 1.1133 | 0.5597 | 0.2802 | 0.026* |
| C15 | 1.1655 (2) | 0.59151 (16) | 0.1671 (2) | 0.0254 (6) |
| H15 | 1.1998 | 0.6238 | 0.1922 | 0.031* |
| C16 | 1.1659 (2) | 0.58250 (16) | 0.0774 (2) | 0.0226 (6) |
| H16 | 1.1989 | 0.6098 | 0.0422 | 0.027* |
| C17 | 1.1175 (2) | 0.53317 (18) | 0.0397 (2) | 0.0245 (6) |
| H17 | 1.1187 | 0.5265 | −0.0206 | 0.029* |
| C18 | 1.0667 (2) | 0.49336 (16) | 0.09217 (19) | 0.0210 (5) |

| | | | | |
|---|---|---|---|---|
| H18 | 1.0342 | 0.4599 | 0.0669 | 0.025* |
| C19 | 1.06746 (18) | 0.02056 (14) | 0.31128 (19) | 0.0167 (5) |
| C20 | 1.0336 (2) | −0.0317 (2) | 0.3592 (3) | 0.0397 (10) |
| H20 | 0.9762 | −0.0371 | 0.3614 | 0.048* |
| C21 | 1.0845 (3) | −0.0761 (2) | 0.4042 (4) | 0.0500 (13) |
| H21 | 1.0613 | −0.1109 | 0.4366 | 0.060* |
| C22 | 1.1683 (3) | −0.0686 (2) | 0.4008 (3) | 0.0392 (10) |
| H22 | 1.2023 | −0.0988 | 0.4303 | 0.047* |
| C23 | 1.2032 (2) | −0.0169 (2) | 0.3543 (3) | 0.0347 (9) |
| H23 | 1.2605 | −0.0117 | 0.3534 | 0.042* |
| C24 | 1.1529 (2) | 0.02794 (18) | 0.3083 (2) | 0.0240 (6) |
| H24 | 1.1766 | 0.0626 | 0.2759 | 0.029* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Mo1 | 0.00826 (8) | 0.00886 (8) | 0.00810 (8) | −0.00049 (6) | −0.00012 (6) | 0.00068 (6) |
| Mo2 | 0.00835 (8) | 0.00926 (9) | 0.00849 (8) | −0.00033 (6) | 0.00031 (6) | −0.00117 (6) |
| P1 | 0.0070 (2) | 0.0143 (3) | 0.0124 (3) | −0.0001 (2) | 0.0006 (2) | 0.0027 (2) |
| P2 | 0.0124 (4) | 0.0087 (3) | 0.0099 (4) | 0.000 | 0.0000 (3) | 0.000 |
| P3 | 0.0108 (4) | 0.0090 (3) | 0.0140 (4) | 0.000 | −0.0004 (3) | 0.000 |
| O1 | 0.0085 (7) | 0.0114 (7) | 0.0107 (7) | −0.0012 (6) | 0.0007 (6) | −0.0018 (6) |
| O2 | 0.0082 (7) | 0.0090 (7) | 0.0113 (7) | −0.0008 (6) | 0.0015 (6) | 0.0006 (6) |
| O3 | 0.0136 (8) | 0.0160 (8) | 0.0102 (8) | −0.0018 (7) | 0.0002 (6) | 0.0013 (6) |
| O4 | 0.0156 (8) | 0.0163 (8) | 0.0106 (8) | −0.0012 (7) | 0.0007 (6) | −0.0023 (7) |
| O5 | 0.0086 (7) | 0.0138 (8) | 0.0140 (8) | 0.0000 (6) | 0.0006 (6) | −0.0045 (6) |
| O6 | 0.0082 (7) | 0.0150 (8) | 0.0128 (8) | 0.0016 (6) | 0.0006 (6) | 0.0036 (6) |
| O7 | 0.0134 (8) | 0.0093 (7) | 0.0116 (7) | −0.0009 (6) | −0.0026 (6) | 0.0012 (6) |
| O8 | 0.0144 (8) | 0.0104 (8) | 0.0151 (8) | 0.0001 (6) | 0.0030 (7) | −0.0005 (6) |
| C1 | 0.0104 (10) | 0.0141 (10) | 0.0161 (11) | −0.0001 (8) | −0.0012 (8) | 0.0021 (9) |
| C2 | 0.0151 (11) | 0.0237 (13) | 0.0209 (13) | 0.0016 (10) | 0.0005 (10) | −0.0026 (11) |
| C3 | 0.0228 (14) | 0.0254 (14) | 0.0196 (13) | 0.0006 (11) | −0.0033 (11) | −0.0058 (11) |
| C4 | 0.0189 (13) | 0.0261 (14) | 0.0204 (13) | −0.0030 (11) | −0.0072 (10) | −0.0008 (11) |
| C5 | 0.0151 (12) | 0.0329 (16) | 0.0300 (16) | 0.0029 (12) | −0.0070 (11) | −0.0062 (13) |
| C6 | 0.0126 (11) | 0.0276 (14) | 0.0233 (13) | 0.0030 (10) | −0.0019 (10) | −0.0052 (11) |
| C7 | 0.0112 (10) | 0.0141 (10) | 0.0130 (10) | 0.0000 (8) | 0.0007 (8) | 0.0006 (8) |
| C8 | 0.0159 (11) | 0.0167 (11) | 0.0174 (11) | −0.0026 (9) | 0.0039 (9) | 0.0014 (9) |
| C9 | 0.0192 (12) | 0.0248 (14) | 0.0182 (12) | −0.0009 (11) | 0.0053 (10) | 0.0011 (11) |
| C10 | 0.0186 (12) | 0.0250 (14) | 0.0184 (12) | 0.0014 (11) | 0.0017 (10) | −0.0048 (11) |
| C11 | 0.0226 (13) | 0.0171 (12) | 0.0247 (14) | −0.0005 (10) | 0.0015 (11) | −0.0031 (11) |
| C12 | 0.0160 (11) | 0.0153 (11) | 0.0212 (12) | −0.0007 (9) | 0.0028 (10) | 0.0019 (10) |
| C13 | 0.0143 (10) | 0.0099 (9) | 0.0149 (10) | 0.0003 (8) | 0.0018 (8) | −0.0014 (8) |
| C14 | 0.0251 (14) | 0.0185 (12) | 0.0223 (13) | −0.0083 (11) | 0.0022 (11) | −0.0047 (10) |
| C15 | 0.0308 (16) | 0.0184 (13) | 0.0271 (15) | −0.0106 (12) | 0.0056 (12) | −0.0028 (11) |
| C16 | 0.0207 (13) | 0.0182 (12) | 0.0289 (15) | 0.0008 (10) | 0.0083 (11) | 0.0060 (11) |
| C17 | 0.0263 (15) | 0.0329 (16) | 0.0144 (12) | −0.0008 (12) | 0.0057 (11) | 0.0023 (11) |
| C18 | 0.0246 (13) | 0.0247 (14) | 0.0138 (11) | −0.0064 (11) | 0.0036 (10) | −0.0017 (10) |
| C19 | 0.0164 (11) | 0.0139 (11) | 0.0197 (12) | 0.0007 (9) | −0.0033 (9) | 0.0027 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C20 | 0.0243 (16) | 0.039 (2) | 0.055 (3) | −0.0017 (15) | −0.0042 (16) | 0.0294 (19) |
| C21 | 0.048 (3) | 0.040 (2) | 0.062 (3) | 0.0002 (19) | −0.014 (2) | 0.032 (2) |
| C22 | 0.044 (2) | 0.0339 (19) | 0.039 (2) | 0.0173 (17) | −0.0218 (18) | 0.0018 (16) |
| C23 | 0.0236 (15) | 0.050 (2) | 0.0302 (17) | 0.0138 (15) | −0.0116 (13) | −0.0103 (16) |
| C24 | 0.0177 (13) | 0.0319 (16) | 0.0226 (14) | 0.0053 (11) | −0.0051 (11) | −0.0037 (12) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Mo1—O3 | 1.6717 (19) | C5—C6 | 1.396 (4) |
| Mo1—O1 | 1.9559 (18) | C5—H5 | 0.9300 |
| Mo1—O2 | 1.9569 (18) | C6—H6 | 0.9300 |
| Mo1—O6 | 2.0519 (18) | C7—C8 | 1.393 (4) |
| Mo1—O7 | 2.0648 (18) | C7—C12 | 1.401 (4) |
| Mo1—O1$^{i}$ | 2.3950 (19) | C8—C9 | 1.389 (4) |
| Mo1—Mo2 | 2.6395 (3) | C8—H8 | 0.9300 |
| Mo2—O4 | 1.6686 (19) | C9—C10 | 1.388 (4) |
| Mo2—O2 | 1.9632 (18) | C9—H9 | 0.9300 |
| Mo2—O1 | 1.9661 (18) | C10—C11 | 1.392 (4) |
| Mo2—O8 | 2.0615 (19) | C10—H10 | 0.9300 |
| Mo2—O5 | 2.0701 (19) | C11—C12 | 1.390 (4) |
| Mo2—O2$^{i}$ | 2.3982 (19) | C11—H11 | 0.9300 |
| P1—O6 | 1.527 (2) | C12—H12 | 0.9300 |
| P1—O5$^{i}$ | 1.5351 (19) | C13—C14 | 1.389 (4) |
| P1—C1 | 1.789 (3) | C13—C18 | 1.394 (4) |
| P1—C7 | 1.789 (3) | C14—C15 | 1.388 (4) |
| P2—O7 | 1.5298 (19) | C14—H14 | 0.9300 |
| P2—O7$^{i}$ | 1.5298 (19) | C15—C16 | 1.379 (5) |
| P2—C13$^{i}$ | 1.793 (3) | C15—H15 | 0.9300 |
| P2—C13 | 1.793 (3) | C16—C17 | 1.379 (5) |
| P3—O8$^{i}$ | 1.526 (2) | C16—H16 | 0.9300 |
| P3—O8 | 1.526 (2) | C17—C18 | 1.391 (4) |
| P3—C19 | 1.791 (3) | C17—H17 | 0.9300 |
| P3—C19$^{i}$ | 1.791 (3) | C18—H18 | 0.9300 |
| O1—Mo1$^{i}$ | 2.3950 (19) | C19—C20 | 1.383 (5) |
| O2—Mo2$^{i}$ | 2.3982 (19) | C19—C24 | 1.385 (4) |
| O5—P1$^{i}$ | 1.5351 (19) | C20—C21 | 1.386 (5) |
| C1—C6 | 1.391 (4) | C20—H20 | 0.9300 |
| C1—C2 | 1.398 (4) | C21—C22 | 1.360 (7) |
| C2—C3 | 1.387 (4) | C21—H21 | 0.9300 |
| C2—H2 | 0.9300 | C22—C23 | 1.369 (7) |
| C3—C4 | 1.386 (4) | C22—H22 | 0.9300 |
| C3—H3 | 0.9300 | C23—C24 | 1.393 (5) |
| C4—C5 | 1.379 (5) | C23—H23 | 0.9300 |
| C4—H4 | 0.9300 | C24—H24 | 0.9300 |
| | | | |
| O3—Mo1—O1 | 109.88 (9) | C6—C1—P1 | 123.7 (2) |
| O3—Mo1—O2 | 108.17 (9) | C2—C1—P1 | 116.1 (2) |
| O1—Mo1—O2 | 89.87 (7) | C3—C2—C1 | 119.9 (3) |

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| O3—Mo1—O6 | 97.93 (9) | C3—C2—H2 | 120.1 |
| O1—Mo1—O6 | 151.61 (7) | C1—C2—H2 | 120.1 |
| O2—Mo1—O6 | 86.71 (7) | C4—C3—C2 | 120.1 (3) |
| O3—Mo1—O7 | 97.84 (8) | C4—C3—H3 | 119.9 |
| O1—Mo1—O7 | 84.81 (7) | C2—C3—H3 | 119.9 |
| O2—Mo1—O7 | 153.70 (7) | C5—C4—C3 | 119.9 (3) |
| O6—Mo1—O7 | 85.89 (8) | C5—C4—H4 | 120.0 |
| O3—Mo1—O1$^{i}$ | 169.29 (8) | C3—C4—H4 | 120.0 |
| O1—Mo1—O1$^{i}$ | 78.16 (7) | C4—C5—C6 | 120.9 (3) |
| O2—Mo1—O1$^{i}$ | 78.27 (7) | C4—C5—H5 | 119.6 |
| O6—Mo1—O1$^{i}$ | 73.54 (7) | C6—C5—H5 | 119.6 |
| O7—Mo1—O1$^{i}$ | 75.43 (7) | C1—C6—C5 | 119.0 (3) |
| O3—Mo1—Mo2 | 98.95 (7) | C1—C6—H6 | 120.5 |
| O1—Mo1—Mo2 | 47.86 (5) | C5—C6—H6 | 120.5 |
| O2—Mo1—Mo2 | 47.78 (5) | C8—C7—C12 | 119.8 (3) |
| O6—Mo1—Mo2 | 134.41 (5) | C8—C7—P1 | 119.4 (2) |
| O7—Mo1—Mo2 | 132.67 (5) | C12—C7—P1 | 120.8 (2) |
| O1$^{i}$—Mo1—Mo2 | 91.70 (4) | C9—C8—C7 | 120.3 (3) |
| O4—Mo2—O2 | 109.42 (9) | C9—C8—H8 | 119.9 |
| O4—Mo2—O1 | 109.06 (9) | C7—C8—H8 | 119.9 |
| O2—Mo2—O1 | 89.39 (7) | C10—C9—C8 | 119.9 (3) |
| O4—Mo2—O8 | 97.02 (9) | C10—C9—H9 | 120.1 |
| O2—Mo2—O8 | 84.49 (7) | C8—C9—H9 | 120.1 |
| O1—Mo2—O8 | 153.77 (8) | C9—C10—C11 | 120.1 (3) |
| O4—Mo2—O5 | 99.12 (9) | C9—C10—H10 | 120.0 |
| O2—Mo2—O5 | 151.09 (7) | C11—C10—H10 | 120.0 |
| O1—Mo2—O5 | 85.51 (7) | C12—C11—C10 | 120.4 (3) |
| O8—Mo2—O5 | 87.63 (8) | C12—C11—H11 | 119.8 |
| O4—Mo2—O2$^{i}$ | 169.42 (8) | C10—C11—H11 | 119.8 |
| O2—Mo2—O2$^{i}$ | 77.94 (7) | C11—C12—C7 | 119.5 (3) |
| O1—Mo2—O2$^{i}$ | 78.02 (7) | C11—C12—H12 | 120.2 |
| O8—Mo2—O2$^{i}$ | 75.76 (7) | C7—C12—H12 | 120.2 |
| O5—Mo2—O2$^{i}$ | 73.16 (7) | C14—C13—C18 | 119.6 (3) |
| O4—Mo2—Mo1 | 99.31 (7) | C14—C13—P2 | 120.5 (2) |
| O2—Mo2—Mo1 | 47.57 (5) | C18—C13—P2 | 119.9 (2) |
| O1—Mo2—Mo1 | 47.54 (5) | C15—C14—C13 | 119.8 (3) |
| O8—Mo2—Mo1 | 132.06 (5) | C15—C14—H14 | 120.1 |
| O5—Mo2—Mo1 | 132.95 (5) | C13—C14—H14 | 120.1 |
| O2$^{i}$—Mo2—Mo1 | 91.27 (4) | C16—C15—C14 | 120.4 (3) |
| O6—P1—O5$^{i}$ | 114.95 (10) | C16—C15—H15 | 119.8 |
| O6—P1—C1 | 106.55 (12) | C14—C15—H15 | 119.8 |
| O5$^{i}$—P1—C1 | 107.61 (12) | C17—C16—C15 | 120.2 (3) |
| O6—P1—C7 | 109.45 (12) | C17—C16—H16 | 119.9 |
| O5$^{i}$—P1—C7 | 108.04 (12) | C15—C16—H16 | 119.9 |
| C1—P1—C7 | 110.19 (12) | C16—C17—C18 | 119.8 (3) |
| O7—P2—O7$^{i}$ | 116.31 (15) | C16—C17—H17 | 120.1 |
| O7—P2—C13$^{i}$ | 108.61 (11) | C18—C17—H17 | 120.1 |
| O7$^{i}$—P2—C13$^{i}$ | 106.97 (11) | C17—C18—C13 | 120.1 (3) |

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| O7—P2—C13 | 106.97 (11) | C17—C18—H18 | 119.9 |
| O7$^{i}$—P2—C13 | 108.61 (11) | C13—C18—H18 | 119.9 |
| C13$^{i}$—P2—C13 | 109.25 (17) | C20—C19—C24 | 119.3 (3) |
| O8$^{i}$—P3—O8 | 116.17 (15) | C20—C19—P3 | 119.2 (2) |
| O8$^{i}$—P3—C19 | 110.42 (12) | C24—C19—P3 | 121.5 (2) |
| O8—P3—C19 | 106.58 (12) | C19—C20—C21 | 120.4 (4) |
| O8$^{i}$—P3—C19$^{i}$ | 106.58 (12) | C19—C20—H20 | 119.8 |
| O8—P3—C19$^{i}$ | 110.42 (12) | C21—C20—H20 | 119.8 |
| C19—P3—C19$^{i}$ | 106.27 (19) | C22—C21—C20 | 119.9 (4) |
| Mo1—O1—Mo2 | 84.60 (7) | C22—C21—H21 | 120.0 |
| Mo1—O1—Mo1$^{i}$ | 101.77 (7) | C20—C21—H21 | 120.0 |
| Mo2—O1—Mo1$^{i}$ | 101.73 (8) | C21—C22—C23 | 120.6 (3) |
| Mo1—O2—Mo2 | 84.65 (7) | C21—C22—H22 | 119.7 |
| Mo1—O2—Mo2$^{i}$ | 101.90 (7) | C23—C22—H22 | 119.7 |
| Mo2—O2—Mo2$^{i}$ | 102.02 (7) | C22—C23—C24 | 120.2 (4) |
| P1$^{i}$—O5—Mo2 | 129.43 (11) | C22—C23—H23 | 119.9 |
| P1—O6—Mo1 | 132.12 (11) | C24—C23—H23 | 119.9 |
| P2—O7—Mo1 | 131.57 (11) | C19—C24—C23 | 119.5 (3) |
| P3—O8—Mo2 | 132.15 (12) | C19—C24—H24 | 120.2 |
| C6—C1—C2 | 120.1 (3) | C23—C24—H24 | 120.2 |

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

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C4—H4···O3$^{ii}$ | 0.93 | 2.52 | 3.188 (4) | 129 |
| C21—H21···O4$^{iii}$ | 0.93 | 2.57 | 3.420 (5) | 152 |

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