Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Bis[1,1′-(1,3-phenylenedimethylene)-di(1*H*-imidazol-3-ium)] *β*-octamolybdate

**Xiao-Dan Wang, Guang-Feng Hou, Ying-Hui Yu and Jin-Sheng Gao***

Engineering Research Center of Pesticides of Heilongjiang University, Heilongjiang University, Harbin 150050, People's Republic of China, and College of Chemistry and Materials Science, Heilongjiang University, Harbin 150080, People's Republic of China
Correspondence e-mail: hgf1000@163.com

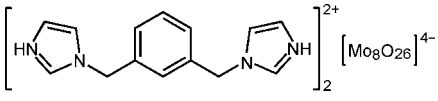
Received 24 November 2011; accepted 9 December 2011

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.006 Å; $R$ factor = 0.026; $wR$ factor = 0.057; data-to-parameter ratio = 16.2.

In the title compound, $(C_{14}H_{16}N_4)_2[Mo_8O_{26}]$, the *β*-octamolybdate anion is centrosymmetric. N—H···O hydrogen bonds link the diimidazolium cations and the polyoxidoanions into a chain structure along [100]. *π*–*π* interactions between the imidazole rings and between the imidazole and benzene rings [centroid–centroid distances = 3.611 (2) and 3.689 (3) Å, respectively] connect the chains.

## Related literature

For general background to polyoxidometalate-based organic-inorganic hybrid compounds, see: Xie *et al.* (2011); Xu *et al.* (1999). For the synthesis of the ligand, see: Yang *et al.* (2006).




## Experimental

### Crystal data

$(C_{14}H_{16}N_4)_2[Mo_8O_{26}]$
$M_r$ = 1664.14
Monoclinic, $P2_1/c$
$a$ = 12.163 (2) Å
$b$ = 12.785 (3) Å
$c$ = 14.937 (3) Å
$\beta$ = 96.82 (3)°
$V$ = 2306.3 (8) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 2.20 mm$^{-1}$
$T$ = 293 K
0.12 × 0.10 × 0.10 mm

### Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min}$ = 0.780, $T_{max}$ = 0.809
21595 measured reflections
5261 independent reflections
4579 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.057
$S$ = 1.01
5261 reflections
324 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.12 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.30 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H21···O12$^{i}$ | 0.90 (1) | 1.96 (2) | 2.844 (4) | 168 (4) |
| N4—H41···O5$^{ii}$ | 0.90 (1) | 2.51 (5) | 3.003 (5) | 115 (4) |
| N4—H41···O8$^{ii}$ | 0.90 (1) | 2.23 (4) | 2.909 (5) | 132 (5) |

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

The authors thank the Project of Innovation Service Platform of Heilongjiang Province (PG09J001) and Heilongjiang University for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2492).

## References

Brandenburg, K. (1999). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalClear*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Xie, Y.-M., Yu, R.-M., Wu, X.-Y., Wang, F., Zhang, J. & Lu, C.-Z. (2011). *Cryst. Growth Des.* **11**, 4739–4741.
Xu, Q., Wang, R.-Z., Yang, G.-Y., Xing, Y.-H., Li, D.-M., Bu, W.-M., Ye, L., Fan, Y.-G., Yang, G.-D., Xong, Y., Lin, Y.-H. & Jia, H.-Q. (1999). *Chem. Commun.* pp. 983–984.
Yang, J., Ma, J.-F., Liu, Y.-Y., Ma, J.-C., Jia, H.-Q. & Hu, N.-H. (2006). *Eur. J. Inorg. Chem.* pp. 1208–1215.

# supporting information

*Acta Cryst.* (2012). E**68**, m50 [doi:10.1107/S1600536811053050]

## Bis[1,1′-(1,3-phenylenedimethylene)di(1*H*-imidazol-3-ium)] *β*-octamolybdate

**Xiao-Dan Wang, Guang-Feng Hou, Ying-Hui Yu and Jin-Sheng Gao**

### S1. Comment

The synthesis and characterization of coordination networks based on the idea of self-assembly of specifically designed building blocks have been an area of rapid growth in recent years. In the last decades, more and more attention has been paid to the rational design and assembly of new polyoxometalate(POM)-based organic-inorganic hybrid compounds due to their structural diversities and abundant potential applications in catalysis, ion exchange, sorption and magnetism (Xie *et al.*, 2011). Octamolybdate family with a variety of structural isomers is a kind of important POMs building blocks (Xu *et al.*, 1999). The title compound was synthesized at a low pH value condition, as an unexpected product during the process of preparing POM-based Cu(II)–ligand complex. We report its structure here.

The asymmertric unit of the title compound contains one half of *β*-$[Mo_8O_{26}]^{4-}$ polyoxoanion and one (1,3-phenylenedimethylene)-di-1*H*-imidazolium cation (Fig. 1). The polyoxoanion is centrosymmetric. N—H···O hydrogen bonds link the cations and polyoxoanions into a chain structure along [1 0 0] (Fig. 2, Table 1). *π*–*π* interactions between the imidazole rings and between the imidazole and benzene rings [centroid–centroid distances = 3.611 (2) and 3.689 (3) Å] connect the chains.

### S2. Experimental

The 1,3-bis(imidazol-l-yl-methyl)benzene (bimb) ligand was synthesized following the literature method (Yang *et al.*, 2006). The title compound was synthesized by mixing bimb (0.101 g, 0.5 mmol), $Cu(NO_3)_2.4H_2O$ (0.102 g, 0.05 mmol), sodium molybdate (0.505 g, 2.5 mmol), $H_2O$ (8 ml) and ethanol (2 ml) and stirring at room temperature for 10 min. The pH value of the mixture was adjusted to 2.0 with 1M HCl, and then the mixture was sealed in a Teflon-lined autoclave and heated at 125°C for 4 days. After slow cooling to room temperature, black block crystals were obtained in 22% yield based on Mo atoms.

### S3. Refinement

The electron density residual peak (1.12) and hole (-1.30) are all around of Mo4 atom with distances of 0.71 and 0.81 Å, respectively. H atoms bound to C atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.93 (aromatic) and 0.97 (methylene) Å and with $U_{iso}$(H) = 1.2$U_{eq}$(C). H atoms bound to N atoms were located from a difference Fourier map and refined isotropically.

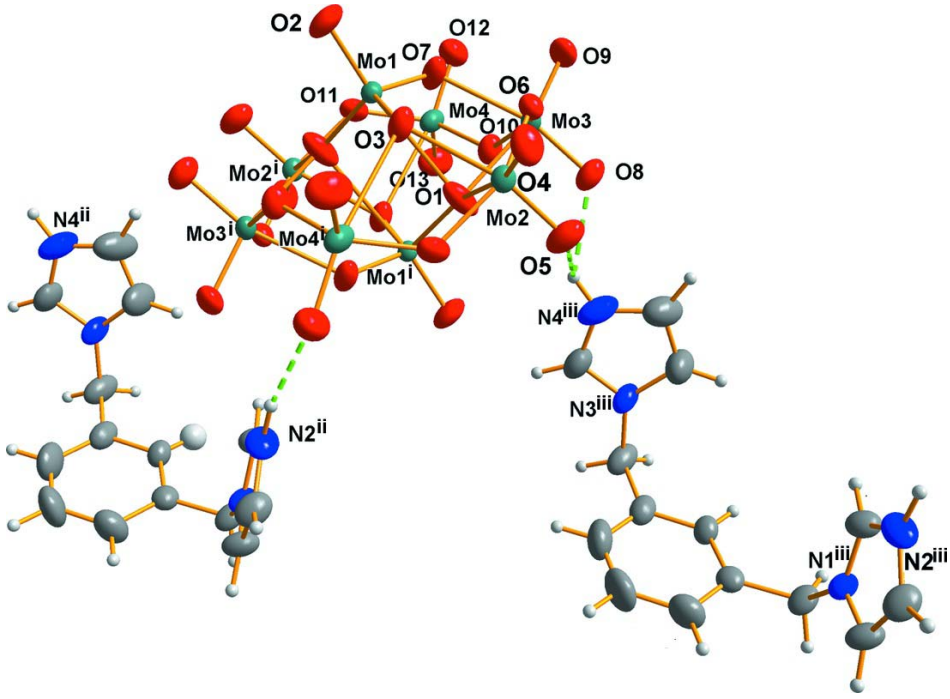



**Figure 1**

The molecular structure of the title compound, showing displacement ellipsoids at the 50% probability level. Dashed lines denote hydrogen bonds. [Symmetry codes: (i) 1-x, 2-y, 2-z; (ii) x, 1+y, z; (iii) -1+x, 1+y, z.]

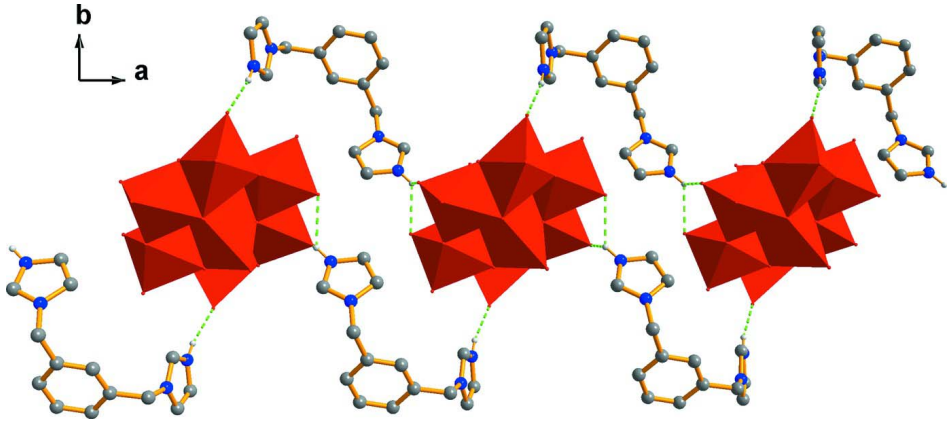



**Figure 2**

A view of the hydrogen-bonded chain structure along [1 0 0]. Dashed lines denote hydrogen bonds.

**Bis[1,1′-(1,3-phenylenedimethylene)di(1*H*-imidazol-3-ium)] *β*-octamolybdate**

*Crystal data*

$(C_{14}H_{16}N_4)_2[Mo_8O_{26}]$
$M_r$ = 1664.14
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 12.163 (2) Å
$b$ = 12.785 (3) Å
$c$ = 14.937 (3) Å
$\beta$ = 96.82 (3)°
$V$ = 2306.3 (8) Å$^3$
$Z$ = 2
$F(000)$ = 1600
$D_x$ = 2.396 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 17887 reflections

$\theta$ = 3.1–27.5°
$\mu$ = 2.20 mm$^{-1}$
$T$ = 293 K

Block, colorless
0.12 × 0.10 × 0.10 mm

*Data collection*

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scan
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min}$ = 0.780, $T_{max}$ = 0.809

21595 measured reflections
5261 independent reflections
4579 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 3.1°
$h$ = −15→15
$k$ = −16→15
$l$ = −19→19

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.057
$S$ = 1.01
5261 reflections
324 parameters
2 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0219P)^2 + 3.6973P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 1.12 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.30 e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.6205 (3) | 0.5883 (3) | 1.0719 (2) | 0.0442 (9) |
| H1 | 0.6032 | 0.6216 | 1.1237 | 0.053* |
| C2 | 0.6426 (3) | 0.6343 (3) | 0.9957 (2) | 0.0411 (8) |
| H2 | 0.6443 | 0.7059 | 0.9850 | 0.049* |
| C3 | 0.6529 (3) | 0.4656 (3) | 0.9766 (3) | 0.0414 (8) |
| H3 | 0.6623 | 0.4003 | 0.9512 | 0.050* |
| C4 | 0.6943 (3) | 0.5749 (3) | 0.8462 (2) | 0.0375 (8) |
| H4A | 0.6747 | 0.6456 | 0.8269 | 0.045* |
| H4B | 0.6542 | 0.5268 | 0.8039 | 0.045* |
| C5 | 0.8175 (3) | 0.5588 (3) | 0.8459 (2) | 0.0334 (7) |
| C6 | 0.8932 (4) | 0.6252 (3) | 0.8933 (3) | 0.0574 (11) |
| H6 | 0.8687 | 0.6820 | 0.9245 | 0.069* |
| C7 | 1.0057 (4) | 0.6069 (4) | 0.8944 (4) | 0.0722 (15) |
| H7 | 1.0564 | 0.6506 | 0.9275 | 0.087* |
| C8 | 1.0431 (3) | 0.5248 (4) | 0.8469 (4) | 0.0638 (13) |
| H8 | 1.1188 | 0.5140 | 0.8473 | 0.077* |
| C9 | 0.9687 (3) | 0.4581 (3) | 0.7984 (3) | 0.0416 (9) |
| C10 | 0.8560 (3) | 0.4767 (3) | 0.7982 (2) | 0.0332 (7) |
| H10 | 0.8054 | 0.4328 | 0.7652 | 0.040* |
| C11 | 1.0073 (3) | 0.3659 (3) | 0.7484 (3) | 0.0517 (11) |
| H11A | 0.9529 | 0.3499 | 0.6974 | 0.062* |

| | | | | |
|---|---|---|---|---|
| H11B | 1.0764 | 0.3833 | 0.7254 | 0.062* |
| C12 | 0.9427 (4) | 0.2172 (4) | 0.8436 (4) | 0.0673 (14) |
| H12 | 0.8670 | 0.2300 | 0.8342 | 0.081* |
| C13 | 0.9922 (5) | 0.1420 (4) | 0.8937 (4) | 0.0722 (15) |
| H13 | 0.9581 | 0.0920 | 0.9264 | 0.087* |
| C14 | 1.1198 (3) | 0.2305 (3) | 0.8354 (3) | 0.0503 (10) |
| H14 | 1.1882 | 0.2521 | 0.8202 | 0.060* |
| Mo1 | 0.547848 (19) | 0.90288 (2) | 1.084672 (17) | 0.02207 (6) |
| Mo2 | 0.30667 (2) | 1.04699 (2) | 1.134611 (18) | 0.02643 (7) |
| Mo3 | 0.27124 (2) | 0.86907 (2) | 0.973110 (19) | 0.02576 (7) |
| Mo4 | 0.45286 (2) | 0.80953 (3) | 0.81444 (2) | 0.03626 (8) |
| N1 | 0.6622 (2) | 0.5573 (2) | 0.93667 (18) | 0.0312 (6) |
| N2 | 0.6281 (3) | 0.4835 (3) | 1.0589 (2) | 0.0463 (8) |
| N3 | 1.0240 (2) | 0.2736 (3) | 0.8073 (2) | 0.0425 (7) |
| N4 | 1.1028 (4) | 0.1517 (3) | 0.8884 (3) | 0.0622 (10) |
| O1 | 0.6277 (2) | 0.9749 (2) | 1.01029 (18) | 0.0472 (7) |
| O2 | 0.6373 (2) | 0.8237 (2) | 1.14858 (18) | 0.0477 (7) |
| O3 | 0.48333 (18) | 0.9874 (2) | 1.15775 (16) | 0.0372 (6) |
| O4 | 0.2810 (2) | 1.0400 (2) | 1.24323 (16) | 0.0439 (6) |
| O5 | 0.1925 (2) | 1.1056 (2) | 1.08042 (19) | 0.0500 (7) |
| O6 | 0.27884 (17) | 0.90605 (17) | 1.09648 (15) | 0.0298 (5) |
| O7 | 0.45232 (19) | 0.82404 (19) | 1.01803 (19) | 0.0429 (6) |
| O8 | 0.1530 (2) | 0.9344 (2) | 0.93423 (17) | 0.0417 (6) |
| O9 | 0.2328 (2) | 0.7420 (2) | 0.97568 (19) | 0.0439 (6) |
| O10 | 0.33456 (19) | 0.8728 (2) | 0.85883 (16) | 0.0361 (5) |
| O11 | 0.6000 (2) | 0.82712 (18) | 0.86168 (17) | 0.0371 (5) |
| O12 | 0.4326 (2) | 0.6786 (2) | 0.8263 (2) | 0.0548 (7) |
| O13 | 0.4385 (3) | 0.8367 (2) | 0.70317 (19) | 0.0555 (7) |
| H21 | 0.619 (4) | 0.433 (3) | 1.099 (2) | 0.066 (14)* |
| H41 | 1.155 (3) | 0.107 (4) | 0.911 (4) | 0.10 (2)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.052 (2) | 0.054 (2) | 0.0275 (18) | 0.0126 (18) | 0.0089 (15) | 0.0002 (17) |
| C2 | 0.058 (2) | 0.0311 (18) | 0.035 (2) | 0.0116 (16) | 0.0088 (16) | −0.0002 (15) |
| C3 | 0.051 (2) | 0.0339 (19) | 0.039 (2) | 0.0056 (16) | 0.0056 (16) | 0.0060 (15) |
| C4 | 0.0397 (19) | 0.046 (2) | 0.0268 (17) | 0.0124 (15) | 0.0046 (14) | 0.0046 (15) |
| C5 | 0.0370 (18) | 0.0339 (18) | 0.0295 (17) | 0.0007 (14) | 0.0049 (13) | 0.0056 (14) |
| C6 | 0.062 (3) | 0.047 (2) | 0.064 (3) | −0.013 (2) | 0.012 (2) | −0.013 (2) |
| C7 | 0.048 (3) | 0.074 (3) | 0.093 (4) | −0.028 (2) | 0.001 (2) | −0.012 (3) |
| C8 | 0.031 (2) | 0.082 (4) | 0.079 (3) | −0.004 (2) | 0.010 (2) | 0.010 (3) |
| C9 | 0.0353 (19) | 0.052 (2) | 0.039 (2) | 0.0081 (16) | 0.0115 (15) | 0.0141 (17) |
| C10 | 0.0288 (16) | 0.043 (2) | 0.0277 (17) | 0.0024 (14) | 0.0033 (12) | 0.0024 (14) |
| C11 | 0.049 (2) | 0.063 (3) | 0.047 (2) | 0.022 (2) | 0.0226 (18) | 0.016 (2) |
| C12 | 0.044 (2) | 0.076 (3) | 0.087 (4) | 0.012 (2) | 0.025 (2) | 0.026 (3) |
| C13 | 0.085 (4) | 0.059 (3) | 0.081 (4) | 0.021 (3) | 0.044 (3) | 0.024 (3) |
| C14 | 0.038 (2) | 0.056 (3) | 0.057 (3) | 0.0151 (18) | 0.0060 (18) | 0.006 (2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| Mo1 | 0.02070 (12) | 0.02499 (13) | 0.02034 (12) | 0.00065 (9) | 0.00169 (9) | 0.00309 (10) |
| Mo2 | 0.02634 (13) | 0.03154 (14) | 0.02250 (13) | 0.00129 (10) | 0.00740 (10) | −0.00118 (11) |
| Mo3 | 0.02085 (12) | 0.02662 (13) | 0.03005 (15) | −0.00019 (10) | 0.00399 (10) | −0.00093 (11) |
| Mo4 | 0.03172 (15) | 0.04101 (17) | 0.03793 (17) | 0.01049 (12) | 0.01196 (12) | 0.01119 (13) |
| N1 | 0.0332 (14) | 0.0329 (15) | 0.0278 (14) | 0.0086 (11) | 0.0043 (11) | 0.0027 (11) |
| N2 | 0.0480 (19) | 0.052 (2) | 0.0386 (18) | −0.0027 (15) | 0.0048 (14) | 0.0181 (15) |
| N3 | 0.0321 (15) | 0.0544 (19) | 0.0423 (18) | 0.0143 (14) | 0.0101 (13) | 0.0102 (15) |
| N4 | 0.072 (3) | 0.057 (2) | 0.057 (2) | 0.030 (2) | 0.005 (2) | 0.0138 (19) |
| O1 | 0.0685 (18) | 0.0394 (14) | 0.0395 (15) | −0.0240 (13) | 0.0304 (13) | −0.0137 (11) |
| O2 | 0.0417 (14) | 0.0540 (17) | 0.0440 (16) | 0.0217 (12) | −0.0092 (11) | 0.0005 (13) |
| O3 | 0.0266 (12) | 0.0462 (14) | 0.0375 (14) | 0.0049 (10) | −0.0015 (9) | −0.0123 (11) |
| O4 | 0.0429 (14) | 0.0641 (18) | 0.0268 (13) | −0.0067 (12) | 0.0124 (10) | −0.0035 (12) |
| O5 | 0.0521 (16) | 0.0485 (16) | 0.0464 (16) | 0.0204 (13) | −0.0062 (12) | −0.0066 (13) |
| O6 | 0.0279 (11) | 0.0316 (12) | 0.0299 (12) | −0.0047 (9) | 0.0038 (9) | 0.0041 (9) |
| O7 | 0.0268 (12) | 0.0363 (14) | 0.0635 (18) | 0.0056 (10) | −0.0038 (11) | −0.0175 (12) |
| O8 | 0.0347 (13) | 0.0524 (16) | 0.0370 (14) | 0.0147 (11) | −0.0001 (10) | −0.0028 (12) |
| O9 | 0.0382 (13) | 0.0332 (13) | 0.0588 (17) | −0.0100 (10) | −0.0004 (12) | −0.0036 (12) |
| O10 | 0.0316 (12) | 0.0479 (15) | 0.0290 (12) | 0.0017 (10) | 0.0045 (9) | −0.0064 (11) |
| O11 | 0.0449 (14) | 0.0251 (11) | 0.0435 (15) | 0.0036 (10) | 0.0149 (11) | −0.0008 (10) |
| O12 | 0.0512 (16) | 0.0449 (16) | 0.070 (2) | −0.0018 (13) | 0.0158 (14) | 0.0089 (14) |
| O13 | 0.073 (2) | 0.0581 (18) | 0.0390 (16) | 0.0120 (15) | 0.0235 (14) | 0.0048 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.336 (5) | C13—H13 | 0.9300 |
| C1—N2 | 1.359 (5) | C14—N3 | 1.312 (5) |
| C1—H1 | 0.9300 | C14—N4 | 1.313 (6) |
| C2—N1 | 1.362 (4) | C14—H14 | 0.9300 |
| C2—H2 | 0.9300 | Mo1—O2 | 1.695 (2) |
| C3—N2 | 1.319 (5) | Mo1—O7 | 1.756 (2) |
| C3—N1 | 1.326 (4) | Mo1—O3 | 1.783 (2) |
| C3—H3 | 0.9300 | Mo1—O1 | 1.811 (2) |
| C4—N1 | 1.468 (4) | Mo2—O4 | 1.690 (2) |
| C4—C5 | 1.513 (5) | Mo2—O5 | 1.697 (3) |
| C4—H4A | 0.9700 | Mo2—O6 | 1.908 (2) |
| C4—H4B | 0.9700 | Mo2—O11$^{i}$ | 1.966 (2) |
| C5—C10 | 1.381 (5) | Mo2—O3 | 2.267 (2) |
| C5—C6 | 1.384 (5) | Mo2—O1$^{i}$ | 2.410 (3) |
| C6—C7 | 1.387 (7) | Mo3—O9 | 1.692 (2) |
| C6—H6 | 0.9300 | Mo3—O8 | 1.704 (2) |
| C7—C8 | 1.374 (7) | Mo3—O6 | 1.894 (2) |
| C7—H7 | 0.9300 | Mo3—O10 | 1.955 (2) |
| C8—C9 | 1.383 (6) | Mo3—O7 | 2.298 (2) |
| C8—H8 | 0.9300 | Mo3—O1$^{i}$ | 2.341 (2) |
| C9—C10 | 1.391 (5) | Mo4—O13 | 1.687 (3) |
| C9—C11 | 1.501 (6) | Mo4—O12 | 1.704 (3) |
| C10—H10 | 0.9300 | Mo4—O10 | 1.842 (2) |
| C11—N3 | 1.472 (5) | Mo4—O11 | 1.858 (3) |

| | | | |
|---|---|---|---|
| C11—H11A | 0.9700 | N2—H21 | 0.901 (10) |
| C11—H11B | 0.9700 | N4—H41 | 0.896 (10) |
| C12—C13 | 1.320 (7) | O1—Mo3$^{i}$ | 2.341 (2) |
| C12—N3 | 1.385 (5) | O1—Mo2$^{i}$ | 2.410 (2) |
| C12—H12 | 0.9300 | O11—Mo2$^{i}$ | 1.966 (2) |
| C13—N4 | 1.364 (7) | | |

| | | | |
|---|---|---|---|
| C2—C1—N2 | 106.7 (3) | O4—Mo2—O6 | 100.96 (12) |
| C2—C1—H1 | 126.6 | O5—Mo2—O6 | 99.70 (12) |
| N2—C1—H1 | 126.6 | O4—Mo2—O11$^{i}$ | 100.83 (12) |
| C1—C2—N1 | 107.6 (3) | O5—Mo2—O11$^{i}$ | 95.11 (13) |
| C1—C2—H2 | 126.2 | O6—Mo2—O11$^{i}$ | 149.63 (10) |
| N1—C2—H2 | 126.2 | O4—Mo2—O3 | 96.62 (11) |
| N2—C3—N1 | 107.9 (3) | O5—Mo2—O3 | 158.19 (12) |
| N2—C3—H3 | 126.1 | O6—Mo2—O3 | 81.93 (9) |
| N1—C3—H3 | 126.1 | O11$^{i}$—Mo2—O3 | 74.76 (10) |
| N1—C4—C5 | 110.7 (3) | O4—Mo2—O1$^{i}$ | 166.93 (12) |
| N1—C4—H4A | 109.5 | O5—Mo2—O1$^{i}$ | 87.85 (12) |
| C5—C4—H4A | 109.5 | O6—Mo2—O1$^{i}$ | 71.84 (9) |
| N1—C4—H4B | 109.5 | O11$^{i}$—Mo2—O1$^{i}$ | 82.45 (9) |
| C5—C4—H4B | 109.5 | O3—Mo2—O1$^{i}$ | 71.88 (10) |
| H4A—C4—H4B | 108.1 | O9—Mo3—O8 | 104.73 (13) |
| C10—C5—C6 | 119.0 (4) | O9—Mo3—O6 | 101.50 (12) |
| C10—C5—C4 | 120.1 (3) | O8—Mo3—O6 | 98.69 (11) |
| C6—C5—C4 | 121.0 (3) | O9—Mo3—O10 | 100.47 (12) |
| C5—C6—C7 | 119.9 (4) | O8—Mo3—O10 | 95.41 (11) |
| C5—C6—H6 | 120.1 | O6—Mo3—O10 | 149.84 (10) |
| C7—C6—H6 | 120.1 | O9—Mo3—O7 | 90.60 (11) |
| C8—C7—C6 | 120.6 (4) | O8—Mo3—O7 | 164.34 (12) |
| C8—C7—H7 | 119.7 | O6—Mo3—O7 | 81.03 (10) |
| C6—C7—H7 | 119.7 | O10—Mo3—O7 | 78.34 (10) |
| C7—C8—C9 | 120.3 (4) | O9—Mo3—O1$^{i}$ | 163.48 (12) |
| C7—C8—H8 | 119.8 | O8—Mo3—O1$^{i}$ | 91.69 (12) |
| C9—C8—H8 | 119.8 | O6—Mo3—O1$^{i}$ | 73.73 (9) |
| C8—C9—C10 | 118.6 (4) | O10—Mo3—O1$^{i}$ | 79.36 (10) |
| C8—C9—C11 | 121.4 (4) | O7—Mo3—O1$^{i}$ | 73.12 (10) |
| C10—C9—C11 | 120.0 (4) | O13—Mo4—O12 | 107.75 (16) |
| C5—C10—C9 | 121.5 (3) | O13—Mo4—O10 | 105.68 (13) |
| C5—C10—H10 | 119.2 | O12—Mo4—O10 | 105.41 (13) |
| C9—C10—H10 | 119.2 | O13—Mo4—O11 | 109.38 (14) |
| N3—C11—C9 | 111.1 (3) | O12—Mo4—O11 | 102.95 (12) |
| N3—C11—H11A | 109.4 | O10—Mo4—O11 | 124.66 (10) |
| C9—C11—H11A | 109.4 | C3—N1—C2 | 108.4 (3) |
| N3—C11—H11B | 109.4 | C3—N1—C4 | 126.6 (3) |
| C9—C11—H11B | 109.4 | C2—N1—C4 | 124.9 (3) |
| H11A—C11—H11B | 108.0 | C3—N2—C1 | 109.3 (3) |
| C13—C12—N3 | 107.7 (4) | C3—N2—H21 | 124 (3) |
| C13—C12—H12 | 126.2 | C1—N2—H21 | 127 (3) |

| | | | |
|---|---|---|---|
| N3—C12—H12 | 126.2 | C14—N3—C12 | 107.7 (4) |
| C12—C13—N4 | 106.7 (4) | C14—N3—C11 | 125.6 (3) |
| C12—C13—H13 | 126.7 | C12—N3—C11 | 126.7 (3) |
| N4—C13—H13 | 126.7 | C14—N4—C13 | 109.4 (4) |
| N3—C14—N4 | 108.6 (4) | C14—N4—H41 | 125 (4) |
| N3—C14—H14 | 125.7 | C13—N4—H41 | 125 (4) |
| N4—C14—H14 | 125.7 | Mo1—O1—Mo3$^{i}$ | 132.92 (13) |
| O2—Mo1—O7 | 108.29 (13) | Mo1—O1—Mo2$^{i}$ | 138.66 (13) |
| O2—Mo1—O3 | 108.47 (12) | Mo3$^{i}$—O1—Mo2$^{i}$ | 88.18 (8) |
| O7—Mo1—O3 | 112.67 (11) | Mo1—O3—Mo2 | 126.04 (12) |
| O2—Mo1—O1 | 107.09 (14) | Mo3—O6—Mo2 | 120.84 (11) |
| O7—Mo1—O1 | 108.11 (13) | Mo1—O7—Mo3 | 124.65 (12) |
| O3—Mo1—O1 | 111.99 (12) | Mo4—O10—Mo3 | 135.18 (14) |
| O4—Mo2—O5 | 104.33 (14) | Mo4—O11—Mo2$^{i}$ | 129.54 (13) |

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

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H21···O12$^{ii}$ | 0.90 (1) | 1.96 (2) | 2.844 (4) | 168 (4) |
| N4—H41···O5$^{iii}$ | 0.90 (1) | 2.51 (5) | 3.003 (5) | 115 (4) |
| N4—H41···O8$^{iii}$ | 0.90 (1) | 2.23 (4) | 2.909 (5) | 132 (5) |

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