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# Crystal structure of heptaguanidinium nonahydrogen bis[α-hexamolybdoplatinate(IV)] heptahydrate

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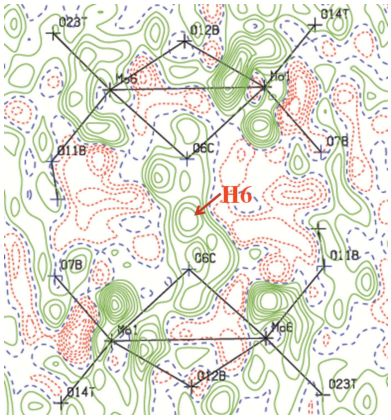
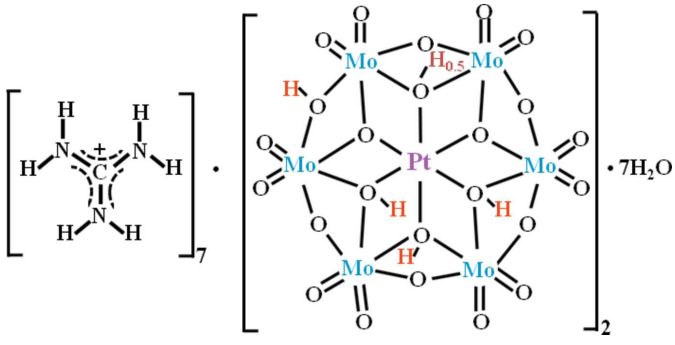
The title compound, $(CH_6N_3)_7H_9[PtMo_6O_{24}]_2\cdot 7H_2O$, containing the well-known Anderson-type heteropolyoxomolybdate, was obtained by recrystallization of its powdered guanidinium salt. The protonated O atoms in the polyanion were confirmed by electron-density maps, interpolyanion hydrogen bonds and bond-valance sums (BVS). The $\{[H_{4.5}PtMo_6O_{24}]_2\}^{7-}$ polyanion is the same as that already characterized in $K_7[H_{4.5}PtMo_6O_{24}]_2\cdot 11H_2O$ [space group $P\overline{1}$; Lee & Joo (2010). *Acta Cryst.* E**66**, i8–i9]. The heteropolyanions form inversion-generated dimers, $\{[H_{4.5}PtMo_6O_{24}]_2\}^{7-}$, held together by each of the four $\mu_3$-O—H$\cdots\mu_1$-O, two $\mu_2$-O—H$\cdots\mu_2$-O hydrogen bonds and one centrosymmetric $\mu_3$-O—H—$\mu_3$-O hydrogen bond. The H atom of the centrosymmetric hydrogen bond is located on an inversion centre. One guanidinium ion and one water molecule are equally disordered about a twofold rotation axis.

## 1. Chemical context

The α (planar structure)-β (bent structure)-α geometrical isomerization, according to stepwise protonation in the $[PtMo_6O_{24}]^{8-}$ polyoxometalate (POM) species, *viz*. $([H_{3.5}\alpha\text{-}PtMo_6O_{24}]^{4.5-}$ (Lee & Sasaki, 1994), $[H_4\beta\text{-}PtMo_6O_{24}]^{4-}$ (Lee & Sasaki, 1994; Joo *et al.*, 1994) and $[H_{4.5}\alpha\text{-}PtMo_6O_{24}]^{3.5-}$ (Lee & Sasaki, 1994; Lee *et al.*, 2010) is an unprecedented phenomenon in the Anderson-type heteropolyanion (Anderson, 1937), as well as in the chemistry of polyoxometalates.




As a result of the insolubility of the guanidinium salt, replaceable counter-cations in POMs can be exchanged by guanidinium ions. It is thus possible to obtain stable POMs by precipitation from aqueous solution with guanidinium salts. The guanidinium salts of platinum-containing POM species, *viz*. $(CH_6N_3)_8[PtW_6O_{24}]$ (Lee *et al.*, 2003), $(CH_6N_3)_5$-

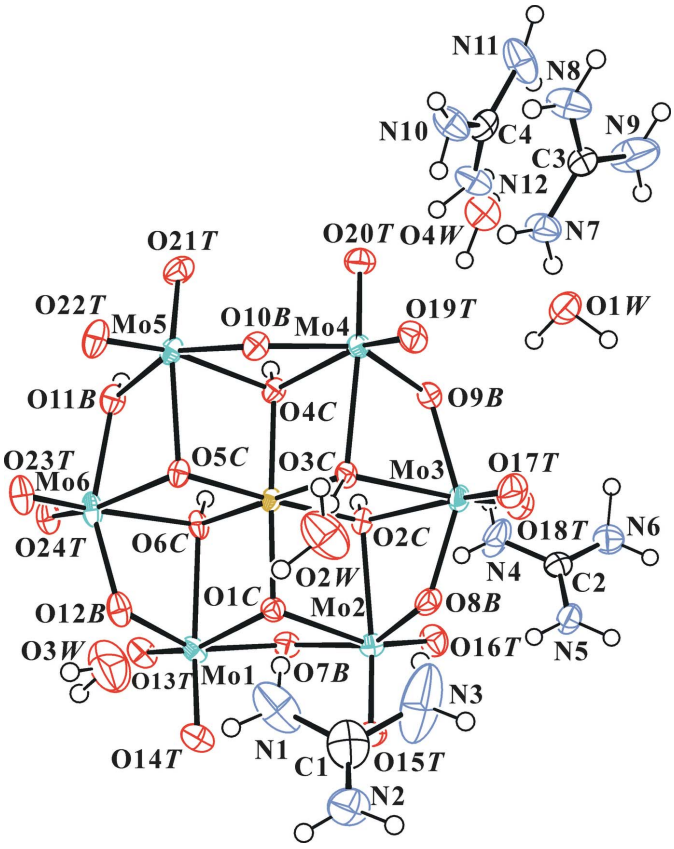



**Figure 1**
The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius. Disordered parts have been omitted for clarity.

$[H_2PtV_9O_{28}]$ (Joo *et al.*, 2011) and $(CH_6N_3)_8[\alpha$-$SiPt_2W_{10}O_{40}]\cdot 6H_2O$ (Lee *et al.*, 2003) have been reported by our group. The positions of the protonated O atoms in the $\{[H_{4.5}\alpha$-$PtMo_6O_{24}]_2\}^{7-}$ polyanion were reconfirmed in the present study.

Sometimes a short hydrogen bond, O···O distance < 2.60 Å, in which the H atom lies on a crystallographic center of symmetry, occurs in this class of structure. The H atom of the central hydrogen bond, O6*C*—H6—O6*C*$^{i}$ in the title compound lies on a crystallographic center of symmetry (space group *C*2/*c*: $\frac{3}{4}, \frac{1}{4}, \frac{1}{2}$).

## 2. Structural commentary

The structure of the title compound POM anion has been discussed in detail (Lee *et al.*, 2010). Fig. 1 shows the structure of the title compound, and selected geometrical parameters are given in Table 1. The complete polyanion has $C_1$ (1) symmetry. The O atoms of the heteropolyanion have been designated as O*T* (terminal Mo═O atom), O*B* (bridging $\mu_2$-O atom), and O*C* (centered $\mu_3$-O atom). The protonated O atoms in the polyanion were confirmed in electron density maps, interpolyanion hydrogen bonds (Table 2) and by bond-valence sums (BVS; Brown & Altermatt, 1985; Brese & O'Keeffe, 1991). Fig. 2 shows a symmetric electron-density map around the position of atom H6. The H atom of the centrosymmetric hydrogen bond in the compound lies on a crystallographic centre of symmetry (space group *C*2/*c*: $\frac{3}{4}, \frac{1}{4}, \frac{1}{2}$). The O6*C*—H6 and O6*C*···O6*C*$^{i}$ distances are 1.27 and 2.532 (6) Å, and the O6*C*—H6—O6*C*$^{i}$ angle is 180° (Table 2 and Fig. 3). Atom H3 does not contribute to dimer formation because it is located on the other side of the polyanion.

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

| | | | |
|---|---|---|---|
| Pt1—O1*C* | 1.995 (3) | Mo5—O5*C* | 2.178 (3) |
| Pt1—O2*C* | 2.015 (3) | Mo6—O5*C* | 2.123 (3) |
| Pt1—O3*C* | 2.027 (3) | Mo6—O6*C* | 2.277 (3) |
| Pt1—O4*C* | 2.011 (3) | Mo1—O7*B* | 1.965 (3) |
| Pt1—O5*C* | 1.997 (3) | Mo1—O12*B* | 1.959 (3) |
| Pt1—O6*C* | 2.005 (3) | Mo2—O7*B* | 1.978 (3) |
| Mo1—O1*C* | 2.150 (3) | Mo2—O8*B* | 1.945 (3) |
| Mo1—O6*C* | 2.317 (3) | Mo3—O8*B* | 1.934 (3) |
| Mo2—O1*C* | 2.248 (3) | Mo3—O9*B* | 1.952 (3) |
| Mo2—O2*C* | 2.286 (3) | Mo4—O9*B* | 1.941 (3) |
| Mo3—O2*C* | 2.307 (3) | Mo4—O10*B* | 1.959 (3) |
| Mo3—O3*C* | 2.318 (3) | Mo5—O10*B* | 1.895 (3) |
| Mo4—O3*C* | 2.287 (3) | Mo5—O11*B* | 2.058 (3) |
| Mo4—O4*C* | 2.327 (3) | Mo6—O11*B* | 2.075 (4) |
| Mo5—O4*C* | 2.289 (3) | Mo6—O12*B* | 1.894 (4) |
| | | | |
| Mo1—O1*C*—Mo2 | 95.79 (12) | Mo1—O7*B*—Mo2 | 111.71 (15) |
| Mo2—O2*C*—Mo3 | 93.64 (11) | Mo3—O8*B*—Mo2 | 119.36 (16) |
| Mo4—O3*C*—Mo3 | 93.75 (12) | Mo4—O9*B*—Mo3 | 119.39 (17) |
| Mo5—O4*C*—Mo4 | 92.64 (11) | Mo5—O10*B*—Mo4 | 120.02 (16) |
| Mo6—O5*C*—Mo5 | 102.87 (13) | Mo5—O11*B*—Mo6 | 108.97 (15) |
| Mo6—O6*C*—Mo1 | 91.14 (12) | Mo6—O12*B*—Mo1 | 116.75 (17) |

**Table 2**
Hydrogen-bond geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2*C*—H2···O24*T*$^{i}$ | 0.96 (2) | 1.61 (2) | 2.578 (5) | 179 (6) |
| O3*C*—H3···O2*W* | 0.96 (2) | 1.69 (3) | 2.622 (6) | 164 (7) |
| O4*C*—H4···O13*T*$^{i}$ | 0.95 (2) | 1.63 (2) | 2.568 (5) | 173 (9) |
| O6*C*—H6···O6*C*$^{i}$ | 1.27 | 1.27 | 2.532 (6) | 180 |
| O11*B*—H11···O7*B*$^{i}$ | 0.95 (2) | 1.74 (2) | 2.679 (5) | 173 (10) |
| N1—H1*B*···O1*C* | 0.88 | 2.05 | 2.864 (6) | 154 |
| N1—H1*A*···O3*W* | 0.88 | 2.33 | 2.973 (9) | 130 |
| N2—H2*A*···O18*T*$^{ii}$ | 0.88 | 2.08 | 2.940 (7) | 165 |
| N2—H2*B*···O19*T*$^{iii}$ | 0.88 | 2.22 | 3.043 (6) | 155 |
| N3—H3*B*···O8*B* | 0.88 | 2.04 | 2.874 (7) | 157 |
| N3—H3*A*···O2*W*$^{iii}$ | 0.88 | 2.25 | 2.979 (9) | 140 |
| N4—H4*B*···O14*T*$^{iv}$ | 0.88 | 2.09 | 2.944 (6) | 164 |
| N4—H4*A*···O24*T*$^{i}$ | 0.88 | 2.48 | 3.006 (6) | 119 |
| N5—H5*A*···O16*T* | 0.88 | 2.06 | 2.890 (6) | 157 |
| N5—H5*B*···O21*T*$^{v}$ | 0.88 | 2.18 | 2.973 (5) | 149 |
| N6—H6*A*···O15*T*$^{iv}$ | 0.88 | 2.19 | 2.894 (6) | 136 |
| N6—H6*B*···O21*T*$^{v}$ | 0.88 | 2.59 | 3.281 (6) | 136 |
| N7—H7*B*···O19*T* | 0.88 | 2.40 | 2.936 (5) | 119 |
| N7—H7*A*···O1*W* | 0.88 | 2.11 | 2.927 (6) | 154 |
| N8—H8*B*···O13*T*$^{vi}$ | 0.88 | 2.39 | 3.006 (6) | 128 |
| N8—H8*A*···O23*T*$^{vii}$ | 0.88 | 2.04 | 2.918 (6) | 178 |
| N9—H9*A*···O22*T*$^{vii}$ | 0.88 | 2.21 | 2.938 (7) | 140 |
| O1*W*—H1*AW*···O9*B* | 0.94 (2) | 2.20 (5) | 2.916 (5) | 132 (5) |
| O1*W*—H1*BW*···O17*T*$^{viii}$ | 0.95 (2) | 1.85 (3) | 2.783 (5) | 166 (6) |
| O2*W*—H2*BW*···O4*W*$^{ii}$ | 0.95 (2) | 2.24 (7) | 2.902 (12) | 126 (6) |
| O3*W*—H3*BW*···O9*B*$^{ii}$ | 0.94 (2) | 2.35 (8) | 3.029 (7) | 128 (8) |

Symmetry codes: (i) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z+1$; (ii) $x, y-1, z$; (iii) $-x+1, -y, -z+1$; (iv) $x, y+1, z$; (v) $x, -y+1, z+\frac{1}{2}$; (vi) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (vii) $-x+1, y+1, -z+\frac{1}{2}$; (viii) $-x+1, -y+1, -z+1$.

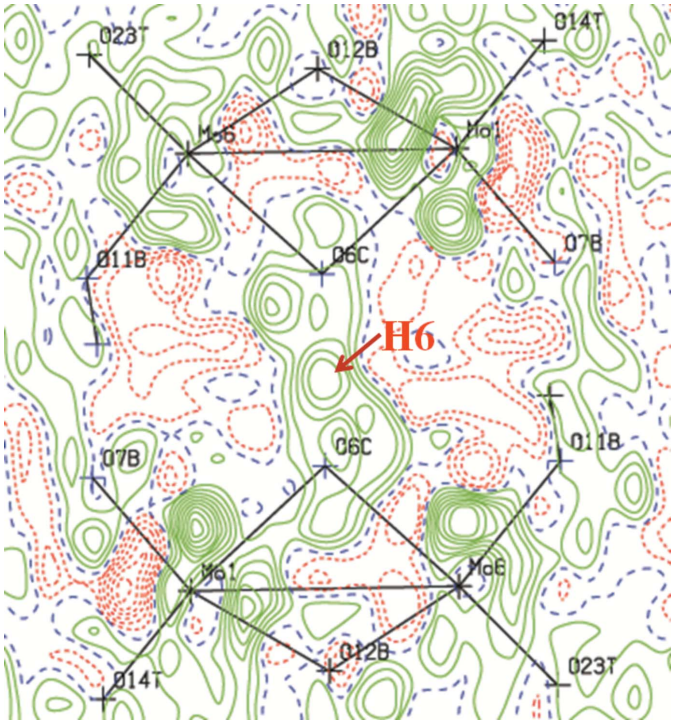



**Figure 2**
Difference-Fourier map around atom H6 (calculated with atom H6 absent from the model).

Confirmation of the protonated O atoms was strongly supported by the BVS analysis. The calculated BVS for atoms O2*C*, O3*C*, O4*C*, O6*C* and O11*B* are 1.40, 1.36, 1.38, 1.41 and 1.30 valence units (v.u.), respectively, if the valence of the O—H bond is not included. Since the BVS value around the $\mu_2$-O atom should be 2.0 v.u., the missing valences of O2*C*, O3*C*, O4*C*, O6*C* and O11*B* are 0.60, 0.64, 0.62, 0.59 and 0.70 v.u., respectively, which corresponds to the valence of the O—H bonds. The BVS value range for the unprotonated O*C* and O*B* atoms is 1.68–1.90 v.u. As a result, the protonated O atoms were O2*C*, O3*C*, O4*C*, O11*B* and O6*C*. The protonated features of both the $\{[H_{4.5}PtMo_6O_{24}]_2\}^{7-}$ polyanion in the title compound and in $K_7[H_{4.5}PtMo_6O_{24}]_2.11H_2O$ (space group $P\overline{1}$) are exactly the same. The bond lengths and bond angles involving protonated and unprotonated O atoms in the $\{[H_{4.5}PtMo_6O_{24}]_2\}^{7-}$ polyanion are compared in Table 1. The Pt—O*C* bond lengths were not affected by protonation of the O*C* atoms.

The C4 guanidinium ion and O4*W* water molecule are equally disordered about a twofold rotation axis.

## 3. Supramolecular features

The heteropolyanions form inversion-generated dimers, $\{[H_{4.5}PtMo_6O_{24}]_2\}^{7-}$ held together by each of the four $\mu_3$-O—H$\cdots\mu_1$-O (terminal O atom), two $\mu_2$-O—H$\cdots\mu_2$-O and one centrosymmetric $\mu_3$-O—H—$\mu_3$-O hydrogen bonds (Table 2). Furthermore, the polyanions are linked in three dimensions *via* N—H$\cdots$O hydrogen bonds. All water molecules form hydrogen bonds with O atoms of the polyanions except for the O2*W* water molecule (Table 2). Hydrogen-bonding interactions involving the disordered molecules have been omitted.

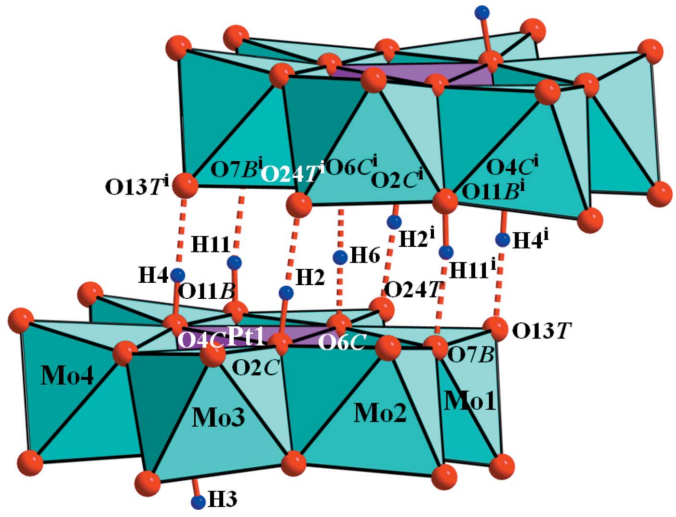



**Figure 3**
Polyhedral view of the heteropolyanion in the title compound with O—H$\cdots$O contacts of the interanion hydrogen bonds shown as red dashed lines. [Symmetry code: (i) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z+1$.]

## 4. Database survey

A number of Anderson-structure platinum(IV)-containing heteropolyoxomolybdates have been reported: $[H_{4.5}PtMo_6O_{24}]^{3.5-}$ and $[H_4PtMo_6O_{24}]^{4-}$, $[H_{3.5}PtMo_6O_{24}]^{4.5-}$ (Lee & Sasaki, 1994); $[H_4\beta\text{-}PtM0_60_{24}]^{4-}$ (Joo *et al.*, 1994); $[H_2PtMo_6O_{24}]^{6-}$ (Lee & Joo, 2000, 2004); $[H_{4.5}PtMo_6O_{24}]^{3.5-}$ (Lee *et al.*, 2010); $[H_6PtMo_6O_{24}]^{2-}$ (Lee & Joo, 2010); $[H_{23}(PtMo_6O_{24})_4]^{9-}$, $[H_{16}(PtMo_6O_{24})_3]^{8-}$ and $[H_{14}(PtMo_6O_{24})_3]^{14-}$ (Day *et al.*, 2009).

## 5. Synthesis and crystallization

A pale-yellow powder of the title compound was obtained by addition of a small excess of the stoichiometric quantity of guanidinium chloride, $CH_6N_3Cl$, to a solution of the sodium salt of hexamolybdoplatinate hydrate. Single crystals were obtained by recrystallization from a hot aqueous solution of the crude sample in an insulating chamber.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All the H atoms in the polyanion and all water H atoms were positioned using difference Fourier maps. All H atoms of the polyanion were refined with a distance restraint of O—H = 0.95 (2) Å using the DFIX command (Sheldrick, 2008). All H atoms of the guanidinium ions were positioned geometrically and refined using a riding model, with $U_{iso}$(H) = 1.5$U_{eq}$(N). The C4 guanidinium ion and O4*W* water molecule are equally disordered about a twofold rotation axis. Refinement of the site occupation factors (s.o.f)

**Table 3**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $(CH_6N_3)_7H_9[PtMo_6O_{24}]_2{\cdot}7H_2O$ |
| $M_r$ | 2865.26 |
| Crystal system, space group | Monoclinic, *C*2/*c* |
| Temperature (K) | 173 |
| *a*, *b*, *c* (Å) | 31.413 (10), 10.073 (3), 23.677 (7) |
| $\beta$ (°) | 119.451 (14) |
| *V* (Å$^3$) | 6524 (3) |
| *Z* | 4 |
| Radiation type | Mo *K*$\alpha$ |
| $\mu$ (mm$^{-1}$) | 6.62 |
| Crystal size (mm) | 0.30 × 0.12 × 0.05 |
| Data collection | |
| Diffractometer | Bruker *SMART* APEXII CCD |
| Absorption correction | Multi-scan (*SADABS*; Sheldrick, 2008) |
| $T_{min}$, $T_{max}$ | 0.241, 0.729 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 56606, 7107, 6050 |
| $R_{int}$ | 0.033 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.639 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S* | 0.028, 0.073, 1.03 |
| No. of reflections | 7107 |
| No. of parameters | 505 |
| No. of restraints | 22 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 2.50, −1.30 |

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2009) and *DIAMOND* (Brandenburg, 1998).

converged at values close to half occupancy. In the final refinement, the s.o.f.s were constrained to 0.5 and reasonable displacement parameters were obtained. The C—N and N—H bond lengths were restrained to 1.30 (2) and 0.90 (2) Å, respectively, and the H*A*—N—H*B* angles were restrained by restraining the H*A*···H*B* distance to 1.55 (2) Å in the disordered C4 guanidinium ion using the DFIX command. The H atoms of all water molecules (O*W*) were refined with a distance restraint of O—H = 0.95 (2) Å using the DFIX, and were included in the refinement with $U_{iso}$(H) = 1.5$U_{eq}$(O). The highest peak in the difference map is 0.98 Å from atom Pt1 and the largest hole is 0.36 Å from N3.

## Acknowledgements

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# supporting information

*Acta Cryst.* (2015). E**71**, 268-271 [doi:10.1107/S2056989015002601]

# Crystal structure of heptaguanidinium nonahydrogen bis[$\alpha$-hexamolybdoplatinate(IV)] heptahydrate

**Hea-Chung Joo, Ki-Min Park and Uk Lee**

## Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2009) and *DIAMOND* (Brandenburg,1998); software used to prepare material for publication: *SHELXL97*.

## Heptaguanidinium nonahydrogen bis[$\alpha$-hexamolybdoplatinate(IV)] heptahydrate

*Crystal data*

$(CH_6N_3)_7H_9[PtMo_6O_{24}]_2\cdot 7H_2O$
$M_r$ = 2865.26
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
*a* = 31.413 (10) Å
*b* = 10.073 (3) Å
*c* = 23.677 (7) Å
$\beta$ = 119.451 (14)°
*V* = 6524 (3) Å$^3$
*Z* = 4
*F*(000) = 5416
$D_x$ = 2.917 Mg m$^{-3}$
Mo *K*$\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 9569 reflections
$\theta$ = 2.2–28.2°
$\mu$ = 6.62 mm$^{-1}$
*T* = 173 K
Block, yellow
0.30 × 0.12 × 0.05 mm

*Data collection*

Bruker SMART APEXII CCD diffractometer
Radiation source: Rotating Anode
Graphite multilayer monochromator
Detector resolution: 10.0 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008)
$T_{min}$ = 0.241, $T_{max}$ = 0.729
56606 measured reflections
7107 independent reflections
6050 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033
$\theta_{max}$ = 27.0°, $\theta_{min}$ = 1.5°
*h* = −40→36
*k* = −12→12
*l* = −30→30

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.073
*S* = 1.03
7107 reflections
505 parameters
22 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0337P)^2 + 47.9084P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 2.50$ e Å$^{-3}$
$\Delta\rho_{min} = -1.30$ e Å$^{-3}$

*Special details*

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

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on $F^2$, conventional R-factors R are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Pt1 | 0.652431 (6) | 0.176198 (17) | 0.467608 (8) | 0.01525 (6) | |
| Mo1 | 0.743430 (14) | −0.00319 (4) | 0.57409 (2) | 0.02093 (10) | |
| Mo2 | 0.682566 (15) | 0.17866 (4) | 0.624195 (19) | 0.02035 (10) | |
| Mo3 | 0.583929 (15) | 0.34871 (4) | 0.51759 (2) | 0.02171 (10) | |
| Mo4 | 0.552026 (14) | 0.34729 (4) | 0.359683 (19) | 0.01849 (9) | |
| Mo5 | 0.618484 (14) | 0.17217 (4) | 0.311229 (19) | 0.01901 (10) | |
| Mo6 | 0.712270 (14) | −0.01255 (4) | 0.42001 (2) | 0.02259 (10) | |
| O1C | 0.66821 (11) | 0.0483 (3) | 0.53959 (14) | 0.0194 (7) | |
| O2C | 0.66214 (11) | 0.3107 (3) | 0.53597 (15) | 0.0187 (7) | |
| H2 | 0.6862 (17) | 0.378 (5) | 0.544 (3) | 0.050 (18)* | |
| O3C | 0.58105 (11) | 0.1962 (3) | 0.44249 (15) | 0.0178 (7) | |
| H3 | 0.568 (2) | 0.109 (3) | 0.439 (3) | 0.07 (2)* | |
| O4C | 0.63463 (11) | 0.3099 (3) | 0.39634 (15) | 0.0184 (7) | |
| H4 | 0.656 (3) | 0.379 (6) | 0.398 (4) | 0.10 (3)* | |
| O5C | 0.64084 (11) | 0.0488 (3) | 0.39683 (14) | 0.0191 (7) | |
| O6C | 0.72274 (11) | 0.1511 (3) | 0.49205 (15) | 0.0190 (7) | |
| H6 | 0.7500 | 0.2500 | 0.5000 | 0.06 (3)* | |
| O7B | 0.74507 (12) | 0.1539 (3) | 0.62431 (16) | 0.0229 (7) | |
| O8B | 0.61194 (12) | 0.2010 (3) | 0.57683 (16) | 0.0243 (7) | |
| O9B | 0.57705 (11) | 0.4420 (3) | 0.44128 (15) | 0.0226 (7) | |
| O10B | 0.55913 (11) | 0.1971 (3) | 0.31241 (15) | 0.0212 (7) | |
| O11B | 0.69300 (12) | 0.1458 (3) | 0.35603 (16) | 0.0235 (7) | |
| H11 | 0.713 (3) | 0.222 (7) | 0.362 (5) | 0.15 (4)* | |
| O12B | 0.71759 (12) | −0.1040 (3) | 0.49309 (17) | 0.0262 (8) | |
| O13T | 0.80360 (12) | 0.0093 (3) | 0.59080 (17) | 0.0285 (8) | |
| O14T | 0.74286 (12) | −0.1258 (3) | 0.62346 (17) | 0.0305 (8) | |
| O15T | 0.68520 (13) | 0.0508 (4) | 0.67282 (16) | 0.0299 (8) | |
| O16T | 0.69977 (13) | 0.3193 (3) | 0.67096 (17) | 0.0312 (8) | |
| O17T | 0.52417 (13) | 0.3226 (4) | 0.49587 (19) | 0.0371 (9) | |
| O18T | 0.60285 (14) | 0.4814 (4) | 0.56903 (17) | 0.0370 (9) | |
| O19T | 0.49288 (12) | 0.3228 (3) | 0.34110 (17) | 0.0275 (8) | |
| O20T | 0.55233 (12) | 0.4783 (3) | 0.31420 (16) | 0.0283 (8) | |

| | | | | | |
|---|---|---|---|---|---|
| O21T | 0.62270 (13) | 0.3112 (4) | 0.27214 (17) | 0.0292 (8) | |
| O22T | 0.60336 (13) | 0.0433 (4) | 0.25787 (17) | 0.0315 (8) | |
| O23T | 0.69102 (12) | −0.1349 (3) | 0.36251 (18) | 0.0304 (8) | |
| O24T | 0.77302 (12) | 0.0093 (3) | 0.44119 (17) | 0.0290 (8) | |
| C1 | 0.5886 (2) | −0.1669 (7) | 0.5871 (3) | 0.0536 (18) | |
| N1 | 0.6167 (2) | −0.1725 (5) | 0.5534 (3) | 0.0611 (18) | |
| H1A | 0.6273 | −0.2493 | 0.5476 | 0.073* | |
| H1B | 0.6230 | −0.0991 | 0.5388 | 0.073* | |
| N2 | 0.57817 (17) | −0.2673 (5) | 0.6092 (2) | 0.0446 (13) | |
| H2A | 0.5879 | −0.3464 | 0.6046 | 0.053* | |
| H2B | 0.5611 | −0.2586 | 0.6293 | 0.053* | |
| N3 | 0.5678 (3) | −0.0394 (7) | 0.5888 (4) | 0.106 (3) | |
| H3A | 0.5480 | −0.0333 | 0.6052 | 0.127* | |
| H3B | 0.5748 | 0.0317 | 0.5735 | 0.127* | |
| C2 | 0.69992 (17) | 0.6537 (5) | 0.7035 (2) | 0.0252 (11) | |
| N4 | 0.7180 (2) | 0.6254 (5) | 0.6658 (3) | 0.0482 (14) | |
| H4A | 0.7210 | 0.5420 | 0.6571 | 0.058* | |
| H4B | 0.7273 | 0.6896 | 0.6490 | 0.058* | |
| N5 | 0.68617 (16) | 0.5573 (4) | 0.7284 (2) | 0.0310 (10) | |
| H5A | 0.6892 | 0.4741 | 0.7196 | 0.037* | |
| H5B | 0.6739 | 0.5760 | 0.7538 | 0.037* | |
| N6 | 0.69567 (17) | 0.7785 (5) | 0.7166 (2) | 0.0355 (11) | |
| H6A | 0.7050 | 0.8426 | 0.6999 | 0.043* | |
| H6B | 0.6835 | 0.7976 | 0.7420 | 0.043* | |
| C3 | 0.4036 (2) | 0.6616 (5) | 0.2726 (3) | 0.0290 (11) | |
| N7 | 0.43204 (15) | 0.5625 (4) | 0.3073 (2) | 0.0324 (10) | |
| H7A | 0.4546 | 0.5757 | 0.3479 | 0.039* | |
| H7B | 0.4284 | 0.4834 | 0.2898 | 0.039* | |
| N8 | 0.36976 (18) | 0.6419 (5) | 0.2123 (2) | 0.0444 (13) | |
| H8A | 0.3507 | 0.7078 | 0.1894 | 0.053* | |
| H8B | 0.3661 | 0.5628 | 0.1947 | 0.053* | |
| N9 | 0.4097 (3) | 0.7778 (5) | 0.2998 (3) | 0.075 (2) | |
| H9A | 0.3910 | 0.8448 | 0.2775 | 0.090* | |
| H9B | 0.4326 | 0.7894 | 0.3403 | 0.090* | |
| C4 | 0.5228 (3) | 0.8412 (9) | 0.2828 (4) | 0.028 (2) | 0.50 |
| N10 | 0.5000 | 0.7313 (6) | 0.2500 | 0.0368 (16) | |
| H10A | 0.4775 (14) | 0.686 (4) | 0.2159 (13) | 0.044* | |
| N11 | 0.5000 | 0.9567 (6) | 0.2500 | 0.0471 (19) | |
| H11A | 0.5223 (16) | 1.002 (4) | 0.2844 (14) | 0.057* | |
| N12 | 0.5653 (4) | 0.8331 (14) | 0.3368 (6) | 0.039 (3) | 0.50 |
| H12A | 0.579 (3) | 0.753 (5) | 0.343 (6) | 0.046* | 0.50 |
| H12B | 0.584 (3) | 0.905 (6) | 0.355 (6) | 0.046* | 0.50 |
| O1W | 0.51593 (15) | 0.6741 (4) | 0.42228 (19) | 0.0400 (10) | |
| H1AW | 0.5475 (12) | 0.638 (6) | 0.446 (3) | 0.060* | |
| H1BW | 0.507 (2) | 0.678 (6) | 0.456 (2) | 0.060* | |
| O2W | 0.52988 (18) | −0.0205 (5) | 0.4254 (3) | 0.0609 (13) | |
| H2AW | 0.508 (3) | −0.006 (8) | 0.3811 (14) | 0.091* | |
| H2BW | 0.553 (2) | −0.091 (6) | 0.435 (4) | 0.091* | |

| | | | | | |
|---|---|---|---|---|---|
| O3W | 0.6421 (2) | −0.3187 (6) | 0.4650 (3) | 0.0764 (17) | |
| H3AW | 0.6747 (13) | −0.346 (9) | 0.487 (4) | 0.115* | |
| H3BW | 0.632 (4) | −0.398 (6) | 0.441 (4) | 0.115* | |
| O4W | 0.5802 (4) | 0.8321 (11) | 0.3704 (5) | 0.036 (2) | 0.50 |
| H4AW | 0.599 (4) | 0.911 (8) | 0.381 (6) | 0.055* | 0.50 |
| H4BW | 0.602 (4) | 0.770 (10) | 0.402 (5) | 0.055* | 0.50 |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Pt1 | 0.01074 (9) | 0.01946 (10) | 0.01597 (9) | −0.00051 (6) | 0.00690 (7) | −0.00191 (7) |
| Mo1 | 0.01368 (19) | 0.0186 (2) | 0.0308 (2) | 0.00080 (15) | 0.01116 (17) | 0.00340 (17) |
| Mo2 | 0.0199 (2) | 0.0212 (2) | 0.0193 (2) | −0.00066 (16) | 0.00908 (17) | 0.00091 (16) |
| Mo3 | 0.0190 (2) | 0.0282 (2) | 0.0213 (2) | 0.00438 (17) | 0.01255 (17) | −0.00020 (17) |
| Mo4 | 0.01413 (19) | 0.0230 (2) | 0.01809 (19) | 0.00203 (15) | 0.00773 (16) | 0.00188 (16) |
| Mo5 | 0.0170 (2) | 0.0242 (2) | 0.01814 (19) | −0.00386 (16) | 0.01039 (17) | −0.00427 (16) |
| Mo6 | 0.01442 (19) | 0.0212 (2) | 0.0358 (2) | −0.00414 (16) | 0.01523 (18) | −0.01035 (18) |
| O1C | 0.0188 (16) | 0.0194 (17) | 0.0189 (15) | 0.0004 (13) | 0.0083 (13) | −0.0001 (13) |
| O2C | 0.0175 (16) | 0.0200 (17) | 0.0203 (16) | −0.0025 (13) | 0.0107 (14) | −0.0044 (13) |
| O3C | 0.0110 (15) | 0.0224 (17) | 0.0212 (16) | 0.0003 (13) | 0.0089 (13) | −0.0007 (13) |
| O4C | 0.0157 (16) | 0.0218 (17) | 0.0194 (16) | −0.0030 (13) | 0.0099 (13) | −0.0016 (13) |
| O5C | 0.0160 (15) | 0.0227 (17) | 0.0195 (15) | −0.0018 (13) | 0.0093 (13) | −0.0053 (13) |
| O6C | 0.0086 (14) | 0.0223 (17) | 0.0225 (16) | 0.0019 (12) | 0.0049 (13) | −0.0056 (13) |
| O7B | 0.0182 (16) | 0.0249 (18) | 0.0228 (17) | −0.0009 (14) | 0.0077 (14) | 0.0004 (14) |
| O8B | 0.0213 (17) | 0.0329 (19) | 0.0235 (17) | −0.0006 (15) | 0.0149 (15) | 0.0021 (15) |
| O9B | 0.0224 (17) | 0.0214 (17) | 0.0241 (16) | 0.0033 (14) | 0.0116 (14) | 0.0006 (14) |
| O10B | 0.0144 (15) | 0.0283 (18) | 0.0201 (16) | −0.0035 (13) | 0.0079 (13) | −0.0024 (14) |
| O11B | 0.0180 (16) | 0.0287 (18) | 0.0271 (17) | −0.0031 (14) | 0.0135 (15) | −0.0036 (15) |
| O12B | 0.0200 (17) | 0.0226 (18) | 0.041 (2) | −0.0029 (14) | 0.0185 (16) | −0.0060 (15) |
| O13T | 0.0204 (17) | 0.0244 (18) | 0.038 (2) | −0.0017 (14) | 0.0124 (16) | 0.0006 (15) |
| O14T | 0.0245 (18) | 0.0262 (19) | 0.038 (2) | 0.0018 (15) | 0.0131 (16) | 0.0075 (16) |
| O15T | 0.0318 (19) | 0.032 (2) | 0.0264 (18) | 0.0031 (16) | 0.0144 (16) | 0.0078 (16) |
| O16T | 0.030 (2) | 0.029 (2) | 0.0307 (19) | 0.0001 (15) | 0.0115 (17) | −0.0062 (15) |
| O17T | 0.0255 (19) | 0.057 (3) | 0.037 (2) | 0.0066 (17) | 0.0210 (18) | 0.0066 (18) |
| O18T | 0.048 (2) | 0.036 (2) | 0.0266 (19) | 0.0097 (18) | 0.0184 (18) | −0.0030 (17) |
| O19T | 0.0159 (16) | 0.033 (2) | 0.0313 (19) | 0.0022 (14) | 0.0099 (15) | 0.0011 (15) |
| O20T | 0.0261 (18) | 0.031 (2) | 0.0254 (17) | 0.0023 (15) | 0.0112 (15) | 0.0051 (15) |
| O21T | 0.0292 (19) | 0.038 (2) | 0.0257 (18) | −0.0018 (16) | 0.0173 (16) | 0.0027 (15) |
| O22T | 0.0311 (19) | 0.038 (2) | 0.0292 (18) | −0.0069 (17) | 0.0176 (16) | −0.0152 (16) |
| O23T | 0.0255 (18) | 0.0244 (18) | 0.044 (2) | −0.0039 (15) | 0.0197 (17) | −0.0126 (17) |
| O24T | 0.0257 (18) | 0.0280 (19) | 0.040 (2) | 0.0018 (15) | 0.0208 (17) | −0.0045 (16) |
| C1 | 0.035 (3) | 0.063 (5) | 0.052 (4) | −0.005 (3) | 0.013 (3) | −0.013 (3) |
| N1 | 0.035 (3) | 0.035 (3) | 0.100 (5) | −0.006 (2) | 0.024 (3) | 0.000 (3) |
| N2 | 0.035 (3) | 0.049 (3) | 0.057 (3) | −0.003 (2) | 0.029 (3) | 0.012 (3) |
| N3 | 0.134 (7) | 0.078 (5) | 0.185 (9) | −0.068 (5) | 0.140 (7) | −0.066 (5) |
| C2 | 0.019 (2) | 0.030 (3) | 0.021 (2) | −0.002 (2) | 0.006 (2) | 0.000 (2) |
| N4 | 0.075 (4) | 0.040 (3) | 0.063 (3) | −0.011 (3) | 0.059 (3) | −0.005 (3) |
| N5 | 0.038 (3) | 0.029 (2) | 0.035 (2) | −0.002 (2) | 0.026 (2) | −0.003 (2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N6 | 0.038 (3) | 0.032 (3) | 0.038 (3) | 0.000 (2) | 0.021 (2) | 0.003 (2) |
| C3 | 0.034 (3) | 0.030 (3) | 0.028 (3) | 0.004 (2) | 0.019 (2) | 0.005 (2) |
| N7 | 0.031 (2) | 0.032 (2) | 0.026 (2) | 0.003 (2) | 0.0069 (19) | 0.0025 (19) |
| N8 | 0.038 (3) | 0.044 (3) | 0.036 (3) | 0.005 (2) | 0.006 (2) | 0.010 (2) |
| N9 | 0.154 (7) | 0.029 (3) | 0.045 (3) | 0.016 (4) | 0.050 (4) | 0.004 (3) |
| C4 | 0.022 (5) | 0.039 (6) | 0.031 (6) | −0.007 (4) | 0.019 (5) | −0.002 (5) |
| N10 | 0.033 (4) | 0.022 (3) | 0.045 (4) | 0.000 | 0.012 (3) | 0.000 |
| N11 | 0.042 (4) | 0.019 (3) | 0.083 (6) | 0.000 | 0.033 (4) | 0.000 |
| N12 | 0.035 (8) | 0.030 (6) | 0.043 (7) | 0.007 (5) | 0.013 (6) | 0.011 (7) |
| O1W | 0.039 (2) | 0.055 (3) | 0.031 (2) | 0.0063 (19) | 0.0207 (19) | 0.0038 (18) |
| O2W | 0.051 (3) | 0.037 (3) | 0.091 (4) | 0.000 (2) | 0.032 (3) | 0.006 (3) |
| O3W | 0.069 (4) | 0.072 (4) | 0.097 (5) | 0.027 (3) | 0.048 (4) | 0.009 (3) |
| O4W | 0.025 (6) | 0.034 (6) | 0.050 (7) | −0.010 (4) | 0.018 (5) | 0.001 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Pt1—O1C | 1.995 (3) | O6C—O6C$^{i}$ | 2.532 (6) |
| Pt1—O2C | 2.015 (3) | O6C—H6 | 1.266 (3) |
| Pt1—O3C | 2.027 (3) | O11B—H11 | 0.95 (2) |
| Pt1—O4C | 2.011 (3) | C1—N2 | 1.254 (8) |
| Pt1—O5C | 1.997 (3) | C1—N1 | 1.451 (10) |
| Pt1—O6C | 2.005 (3) | C1—N3 | 1.451 (10) |
| Mo1—O1C | 2.150 (3) | N1—H1A | 0.8800 |
| Mo1—O6C | 2.317 (3) | N1—H1B | 0.8800 |
| Mo2—O1C | 2.248 (3) | N2—H2A | 0.8800 |
| Mo2—O2C | 2.286 (3) | N2—H2B | 0.8800 |
| Mo3—O2C | 2.307 (3) | N3—H3A | 0.8800 |
| Mo3—O3C | 2.318 (3) | N3—H3B | 0.8800 |
| Mo4—O3C | 2.287 (3) | C2—N4 | 1.304 (7) |
| Mo4—O4C | 2.327 (3) | C2—N5 | 1.314 (6) |
| Mo5—O4C | 2.289 (3) | C2—N6 | 1.317 (7) |
| Mo5—O5C | 2.178 (3) | N4—H4A | 0.8800 |
| Mo6—O5C | 2.123 (3) | N4—H4B | 0.8800 |
| Mo6—O6C | 2.277 (3) | N5—H5A | 0.8800 |
| Mo1—O7B | 1.965 (3) | N5—H5B | 0.8800 |
| Mo1—O12B | 1.959 (3) | N6—H6A | 0.8800 |
| Mo2—O7B | 1.978 (3) | N6—H6B | 0.8800 |
| Mo2—O8B | 1.945 (3) | C3—N9 | 1.303 (7) |
| Mo3—O8B | 1.934 (3) | C3—N8 | 1.310 (7) |
| Mo3—O9B | 1.952 (3) | C3—N7 | 1.322 (6) |
| Mo4—O9B | 1.941 (3) | N7—H7A | 0.8800 |
| Mo4—O10B | 1.959 (3) | N7—H7B | 0.8800 |
| Mo5—O10B | 1.895 (3) | N8—H8A | 0.8800 |
| Mo5—O11B | 2.058 (3) | N8—H8B | 0.8800 |
| Mo6—O11B | 2.075 (4) | N9—H9A | 0.8800 |
| Mo6—O12B | 1.894 (4) | N9—H9B | 0.8800 |
| Mo1—O14T | 1.706 (3) | C4—N12 | 1.320 (12) |
| Mo1—O13T | 1.735 (3) | C4—N10 | 1.340 (10) |

| | | | |
|---|---|---|---|
| Mo2—O15T | 1.702 (3) | C4—N11 | 1.387 (10) |
| Mo2—O16T | 1.713 (3) | N10—H10A | 0.893 (17) |
| Mo3—O17T | 1.705 (4) | N11—H11A | 0.898 (18) |
| Mo3—O18T | 1.706 (4) | N12—H12A | 0.90 (2) |
| Mo4—O19T | 1.704 (3) | N12—H12B | 0.90 (2) |
| Mo4—O20T | 1.706 (3) | O1W—H1AW | 0.94 (2) |
| Mo5—O22T | 1.708 (3) | O1W—H1BW | 0.95 (2) |
| Mo5—O21T | 1.719 (3) | O2W—H2AW | 0.94 (2) |
| Mo6—O23T | 1.710 (3) | O2W—H2BW | 0.95 (2) |
| Mo6—O24T | 1.732 (3) | O3W—H3AW | 0.93 (2) |
| O2C—H2 | 0.96 (2) | O3W—H3BW | 0.94 (2) |
| O3C—H3 | 0.96 (2) | O4W—H4AW | 0.95 (2) |
| O4C—H4 | 0.95 (2) | O4W—H4BW | 0.95 (2) |

| | | | |
|---|---|---|---|
| Mo1—O1C—Mo2 | 95.79 (12) | O21T—Mo5—O4C | 86.44 (14) |
| Mo2—O2C—Mo3 | 93.64 (11) | O10B—Mo5—O4C | 72.56 (12) |
| Mo4—O3C—Mo3 | 93.75 (12) | O11B—Mo5—O4C | 85.46 (12) |
| Mo5—O4C—Mo4 | 92.64 (11) | O5C—Mo5—O4C | 72.30 (12) |
| Mo6—O5C—Mo5 | 102.87 (13) | O23T—Mo6—O24T | 105.40 (16) |
| Mo6—O6C—Mo1 | 91.14 (12) | O23T—Mo6—O12B | 101.61 (17) |
| Mo1—O7B—Mo2 | 111.71 (15) | O24T—Mo6—O12B | 101.99 (16) |
| Mo3—O8B—Mo2 | 119.36 (16) | O23T—Mo6—O11B | 96.50 (16) |
| Mo4—O9B—Mo3 | 119.39 (17) | O24T—Mo6—O11B | 89.87 (15) |
| Mo5—O10B—Mo4 | 120.02 (16) | O12B—Mo6—O11B | 154.72 (14) |
| Mo5—O11B—Mo6 | 108.97 (15) | O23T—Mo6—O5C | 93.14 (14) |
| Mo6—O12B—Mo1 | 116.75 (17) | O24T—Mo6—O5C | 155.73 (14) |
| O1C—Pt1—O5C | 99.68 (14) | O12B—Mo6—O5C | 89.14 (13) |
| O1C—Pt1—O6C | 84.05 (13) | O11B—Mo6—O5C | 72.28 (12) |
| O5C—Pt1—O6C | 83.21 (12) | O23T—Mo6—O6C | 166.99 (14) |
| O1C—Pt1—O4C | 177.35 (12) | O24T—Mo6—O6C | 87.60 (14) |
| O5C—Pt1—O4C | 82.27 (13) | O12B—Mo6—O6C | 75.67 (13) |
| O6C—Pt1—O4C | 97.99 (13) | O11B—Mo6—O6C | 82.71 (13) |
| O1C—Pt1—O2C | 82.64 (13) | O5C—Mo6—O6C | 74.21 (11) |
| O5C—Pt1—O2C | 177.41 (13) | Pt1—O1C—Mo1 | 104.21 (13) |
| O6C—Pt1—O2C | 98.20 (13) | Pt1—O1C—Mo2 | 103.95 (14) |
| O4C—Pt1—O2C | 95.37 (13) | Pt1—O2C—Mo2 | 101.92 (13) |
| O1C—Pt1—O3C | 95.12 (13) | Pt1—O2C—Mo3 | 103.51 (13) |
| O5C—Pt1—O3C | 95.66 (12) | Pt1—O2C—H2 | 116 (4) |
| O6C—Pt1—O3C | 178.46 (12) | Mo2—O2C—H2 | 113 (4) |
| O4C—Pt1—O3C | 82.87 (13) | Mo3—O2C—H2 | 125 (4) |
| O2C—Pt1—O3C | 82.96 (13) | Pt1—O3C—Mo4 | 103.59 (13) |
| O14T—Mo1—O13T | 105.87 (16) | Pt1—O3C—Mo3 | 102.74 (13) |
| O14T—Mo1—O12B | 99.01 (16) | Pt1—O3C—H3 | 108 (4) |
| O13T—Mo1—O12B | 98.14 (15) | Mo4—O3C—H3 | 124 (4) |
| O14T—Mo1—O7B | 100.03 (16) | Mo3—O3C—H3 | 122 (4) |
| O13T—Mo1—O7B | 95.86 (15) | Pt1—O4C—Mo5 | 100.53 (13) |
| O12B—Mo1—O7B | 152.25 (14) | Pt1—O4C—Mo4 | 102.74 (13) |
| O14T—Mo1—O1C | 93.17 (14) | Pt1—O4C—H4 | 124 (5) |

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| O13T—Mo1—O1C | 160.02 (14) | Mo5—O4C—H4 | 109 (5) |
| O12B—Mo1—O1C | 84.50 (12) | Mo4—O4C—H4 | 122 (5) |
| O7B—Mo1—O1C | 74.48 (12) | Pt1—O5C—Mo6 | 103.69 (13) |
| O14T—Mo1—O6C | 165.12 (14) | Pt1—O5C—Mo5 | 104.87 (14) |
| O13T—Mo1—O6C | 88.14 (14) | Pt1—O6C—Mo6 | 98.17 (12) |
| O12B—Mo1—O6C | 73.55 (13) | Pt1—O6C—Mo1 | 98.14 (13) |
| O7B—Mo1—O6C | 83.11 (12) | Pt1—O6C—O6C$^{i}$ | 120.8 (2) |
| O1C—Mo1—O6C | 73.54 (11) | Mo6—O6C—O6C$^{i}$ | 121.27 (19) |
| O15T—Mo2—O16T | 107.06 (18) | Mo1—O6C—O6C$^{i}$ | 120.85 (19) |
| O15T—Mo2—O8B | 98.04 (15) | Pt1—O6C—H6 | 120.8 (2) |
| O16T—Mo2—O8B | 100.65 (16) | Mo6—O6C—H6 | 121.27 (19) |
| O15T—Mo2—O7B | 100.70 (15) | Mo1—O6C—H6 | 120.85 (19) |
| O16T—Mo2—O7B | 96.07 (16) | O6C$^{i}$—O6C—H6 | 0.00 (18) |
| O8B—Mo2—O7B | 149.93 (14) | Mo5—O11B—H11 | 118 (7) |
| O15T—Mo2—O1C | 94.74 (15) | Mo6—O11B—H11 | 125 (7) |
| O16T—Mo2—O1C | 156.95 (15) | N2—C1—N1 | 123.5 (6) |
| O8B—Mo2—O1C | 83.19 (13) | N2—C1—N3 | 119.2 (7) |
| O7B—Mo2—O1C | 72.05 (12) | N1—C1—N3 | 117.0 (6) |
| O15T—Mo2—O2C | 163.07 (14) | C1—N1—H1A | 120.0 |
| O16T—Mo2—O2C | 88.12 (15) | C1—N1—H1B | 120.0 |
| O8B—Mo2—O2C | 71.19 (12) | H1A—N1—H1B | 120.0 |
| O7B—Mo2—O2C | 84.67 (12) | C1—N2—H2A | 120.0 |
| O1C—Mo2—O2C | 71.47 (12) | C1—N2—H2B | 120.0 |
| O17T—Mo3—O18T | 106.36 (19) | H2A—N2—H2B | 120.0 |
| O17T—Mo3—O8B | 98.10 (16) | C1—N3—H3A | 120.0 |
| O18T—Mo3—O8B | 102.14 (16) | C1—N3—H3B | 120.0 |
| O17T—Mo3—O9B | 100.99 (16) | H3A—N3—H3B | 120.0 |
| O18T—Mo3—O9B | 97.16 (16) | N4—C2—N5 | 119.7 (5) |
| O8B—Mo3—O9B | 147.71 (13) | N4—C2—N6 | 120.0 (5) |
| O17T—Mo3—O2C | 160.66 (16) | N5—C2—N6 | 120.3 (5) |
| O18T—Mo3—O2C | 91.76 (16) | C2—N4—H4A | 120.0 |
| O8B—Mo3—O2C | 70.90 (12) | C2—N4—H4B | 120.0 |
| O9B—Mo3—O2C | 82.96 (12) | H4A—N4—H4B | 120.0 |
| O17T—Mo3—O3C | 92.54 (15) | C2—N5—H5A | 120.0 |
| O18T—Mo3—O3C | 159.28 (16) | C2—N5—H5B | 120.0 |
| O8B—Mo3—O3C | 83.03 (13) | H5A—N5—H5B | 120.0 |
| O9B—Mo3—O3C | 70.38 (13) | C2—N6—H6A | 120.0 |
| O2C—Mo3—O3C | 70.78 (11) | C2—N6—H6B | 120.0 |
| O19T—Mo4—O20T | 107.04 (16) | H6A—N6—H6B | 120.0 |
| O19T—Mo4—O9B | 100.65 (15) | N9—C3—N8 | 121.1 (5) |
| O20T—Mo4—O9B | 97.05 (15) | N9—C3—N7 | 118.8 (5) |
| O19T—Mo4—O10B | 99.01 (15) | N8—C3—N7 | 120.1 (5) |
| O20T—Mo4—O10B | 101.47 (15) | C3—N7—H7A | 120.0 |
| O9B—Mo4—O10B | 147.60 (13) | C3—N7—H7B | 120.0 |
| O19T—Mo4—O3C | 92.83 (14) | H7A—N7—H7B | 120.0 |
| O20T—Mo4—O3C | 158.72 (14) | C3—N8—H8A | 120.0 |
| O9B—Mo4—O3C | 71.24 (13) | C3—N8—H8B | 120.0 |
| O10B—Mo4—O3C | 82.34 (13) | H8A—N8—H8B | 120.0 |

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| O19T—Mo4—O4C | 161.33 (14) | C3—N9—H9A | 120.0 |
| O20T—Mo4—O4C | 90.53 (14) | C3—N9—H9B | 120.0 |
| O9B—Mo4—O4C | 82.92 (12) | H9A—N9—H9B | 120.0 |
| O10B—Mo4—O4C | 70.65 (12) | N12—C4—N10 | 120.6 (9) |
| O3C—Mo4—O4C | 70.80 (11) | N12—C4—N11 | 126.3 (9) |
| O22T—Mo5—O21T | 106.73 (18) | N10—C4—N11 | 112.7 (7) |
| O22T—Mo5—O10B | 101.18 (15) | C4—N10—H10A | 155 (2) |
| O21T—Mo5—O10B | 103.69 (15) | C4—N12—H12A | 113 (7) |
| O22T—Mo5—O11B | 96.63 (15) | C4—N12—H12B | 122 (8) |
| O21T—Mo5—O11B | 90.77 (15) | H12A—N12—H4AW | 106 (10) |
| O10B—Mo5—O11B | 152.59 (14) | H12B—N12—H4AW | 20 (10) |
| O22T—Mo5—O5C | 95.73 (15) | H1AW—O1W—H1BW | 100 (6) |
| O21T—Mo5—O5C | 152.95 (14) | H2AW—O2W—H2BW | 116 (8) |
| O10B—Mo5—O5C | 86.07 (13) | H3AW—O3W—H3BW | 92 (8) |
| O11B—Mo5—O5C | 71.47 (12) | H4AW—O4W—H4BW | 103 (10) |
| O22T—Mo5—O4C | 166.58 (15) | | |

Symmetry code: (i) $-x+3/2, -y+1/2, -z+1$.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2*C*—H2···O24*T*$^{i}$ | 0.96 (2) | 1.61 (2) | 2.578 (5) | 179 (6) |
| O3*C*—H3···O2*W* | 0.96 (2) | 1.69 (3) | 2.622 (6) | 164 (7) |
| O4*C*—H4···O13*T*$^{i}$ | 0.95 (2) | 1.63 (2) | 2.568 (5) | 173 (9) |
| O6*C*—H6···O6*C*$^{i}$ | 1.27 | 1.27 | 2.532 (6) | 180 |
| O11*B*—H11···O7*B*$^{i}$ | 0.95 (2) | 1.74 (2) | 2.679 (5) | 173 (10) |
| N1—H1*B*···O1*C* | 0.88 | 2.05 | 2.864 (6) | 154 |
| N1—H1*A*···O3*W* | 0.88 | 2.33 | 2.973 (9) | 130 |
| N2—H2*A*···O18*T*$^{ii}$ | 0.88 | 2.08 | 2.940 (7) | 165 |
| N2—H2*B*···O19*T*$^{iii}$ | 0.88 | 2.22 | 3.043 (6) | 155 |
| N3—H3*B*···O8*B* | 0.88 | 2.04 | 2.874 (7) | 157 |
| N3—H3*A*···O2*W*$^{iii}$ | 0.88 | 2.25 | 2.979 (9) | 140 |
| N4—H4*B*···O14*T*$^{iv}$ | 0.88 | 2.09 | 2.944 (6) | 164 |
| N4—H4*A*···O24*T*$^{i}$ | 0.88 | 2.48 | 3.006 (6) | 119 |
| N5—H5*A*···O16*T* | 0.88 | 2.06 | 2.890 (6) | 157 |
| N5—H5*B*···O21*T*$^{v}$ | 0.88 | 2.18 | 2.973 (5) | 149 |
| N6—H6*A*···O15*T*$^{iv}$ | 0.88 | 2.19 | 2.894 (6) | 136 |
| N6—H6*B*···O21*T*$^{v}$ | 0.88 | 2.59 | 3.281 (6) | 136 |
| N7—H7*B*···O19*T* | 0.88 | 2.40 | 2.936 (5) | 119 |
| N7—H7*A*···O1*W* | 0.88 | 2.11 | 2.927 (6) | 154 |
| N8—H8*B*···O13*T*$^{vi}$ | 0.88 | 2.39 | 3.006 (6) | 128 |
| N8—H8*A*···O23*T*$^{vii}$ | 0.88 | 2.04 | 2.918 (6) | 178 |
| N9—H9*A*···O22*T*$^{vii}$ | 0.88 | 2.21 | 2.938 (7) | 140 |
| O1*W*—H1*AW*···O9*B* | 0.94 (2) | 2.20 (5) | 2.916 (5) | 132 (5) |
| O1*W*—H1*BW*···O17*T*$^{viii}$ | 0.95 (2) | 1.85 (3) | 2.783 (5) | 166 (6) |

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|---|---|---|---|---|
| O2*W*—H2*BW*···O4*W*$^{vi}$ | 0.95 (2) | 2.24 (7) | 2.902 (12) | 126 (6) |
| O3*W*—H3*BW*···O9*B*$^{ii}$ | 0.94 (2) | 2.35 (8) | 3.029 (7) | 128 (8) |

Symmetry codes: (i) −*x*+3/2, −*y*+1/2, −*z*+1; (ii) *x*, *y*−1, *z*; (iii) −*x*+1, −*y*, −*z*+1; (iv) *x*, *y*+1, *z*; (v) *x*, −*y*+1, *z*+1/2; (vi) *x*−1/2, −*y*+1/2, *z*−1/2; (vii) −*x*+1, *y*+1, −*z*+1/2; (viii) −*x*+1, −*y*+1, −*z*+1.