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Pentapotassium  $\mu$ -arsenato-bis(hydroxy-tetramolybdate) dihydrateHai-Hui Yu,<sup>a</sup> Li Kong,<sup>b\*</sup> Ji-Wen Cui<sup>c</sup> and Hai-Tao Wang<sup>d</sup>

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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{As-O}) = 0.005$  Å; R factor = 0.032; wR factor = 0.098; data-to-parameter ratio = 12.1.

The title arsenatomolybdate,  $\text{K}_5[\text{Mo}_8\text{O}_{24}(\text{OH})_2(\text{AsO}_4)] \cdot 2\text{H}_2\text{O}$ , which was obtained hydrothermally, features an  $[\text{AsMo}_8\text{O}_{28}(\text{OH})_2]^{5-}$  anion, which is formed by two  $\text{Mo}_4\text{O}_{14}(\text{OH})$  units that are linked by As in a sandwich-like fashion. The overall symmetry of the anion is  $m2m$ . The  $\{\text{Mo}_4\text{O}_{14}(\text{OH})\}$  core is composed of two pairs of confacial bioctahedral  $\{\text{Mo}_2\text{O}_9\}$  units with two  $\mu_4$ -O atoms which have been characterized as hydroxyl groups. The anions are further interconnected by potassium cations, forming a three-dimensional network structure with the uncoordinated water molecules occupying the channels. The structure is further stabilized by  $\text{O}-\text{H} \cdots \text{O}$  hydrogen bonding.

## Related literature

For isotypic  $\text{K}_{5-x}(\text{NH}_4)_x\text{P}[\text{Mo}_4\text{O}_{14}(\text{OH})_2] \cdot 2\text{H}_2\text{O}$  ( $x = 2.43$ ), see: Chen *et al.* (2009). For a general overview of polyoxometalates, see: Pope (1983). For the Mo/As/O system, see: Sun *et al.* (2007); He & Wang (1999). For bond-valence-sum calculations, see: Brown (1981); Hsu & Wang (1997).

## Experimental

## Crystal data

$\text{K}_5[\text{Mo}_8\text{O}_{24}(\text{OH})_2(\text{AsO}_4)] \cdot 2\text{H}_2\text{O}$   $a = 8.3024$  (12) Å  
 $M_r = 1553.95$   $b = 23.008$  (3) Å  
 Orthorhombic, *Cmcm*  $c = 15.279$  (2) Å

$V = 2918.6$  (7) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation

$\mu = 5.28$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.20 \times 0.18 \times 0.16$  mm

## Data collection

Rigaku R-Axis RAPID diffractometer  
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)  
 $T_{\text{min}} = 0.418$ ,  $T_{\text{max}} = 0.486$

12346 measured reflections  
 1606 independent reflections  
 1216 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.048$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$   
 $wR(F^2) = 0.098$   
 $S = 1.07$   
 1606 reflections

133 parameters  
 Only H-atom coordinates refined  
 $\Delta\rho_{\text{max}} = 1.99$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.72$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O1W}-\text{H1} \cdots \text{O12}^i$	0.82 (9)	2.06 (9)	2.831 (11)	157 (9)
$\text{O11}-\text{H4} \cdots \text{O1W}$	0.87 (11)	1.81 (11)	2.684 (13)	180 (1)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2007); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FI2098).

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**supplementary materials**

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## Pentapotassium $\mu$ -arsenato-bis(hydroxytetramolybdate) dihydrate

H.-H. Yu, L. Kong, J.-W. Cui and H.-T. Wang

### Comment

Polyoxometalates (POMs) are metal-oxygen anionic clusters, which act as multidentate inorganic ligands. They can bind most transition metals, rare earth metals and alkali metals, leading to a family of compounds with a variety of structures (Pope, 1983). Performing as an important part in this field, a number of compounds from the A/Mo/P/O system have been synthesized and structurally characterized, where A is an organic or inorganic cation. In contrast to the rich structural chemistry of molybdenum phosphates, the Mo/As/O system remains relatively undeveloped (Sun *et al.*, 2007; He & Wang, 1999). Here, we have hydrothermally synthesized the title compound,  $\text{K}_5\text{Mo}_8\text{O}_{24}(\text{OH})_2\text{AsO}_4 \cdot 2\text{H}_2\text{O}$ , which is isostructural with  $\text{K}_{5-x}(\text{NH}_4)_x\text{P}[\text{Mo}_4\text{O}_{14}(\text{OH})]_2 \cdot 2\text{H}_2\text{O}$  where  $x=2.43$  (Chen *et al.* (2009).

The structure of the title compound consists of  $[\text{AsMo}_8\text{O}_{28}(\text{OH})_2]^{5-}$  cluster anions,  $\text{K}^+$  cations, and water molecules (Fig. 1). The anion is formed by two crystallographically independent  $\{\text{Mo}_4\text{O}_{14}(\text{OH})\}$  cores, linked by an arsenic(V) atom in a sandwich-like fashion. The anion possesses  $m2m$  symmetry in that all four molybdenum atoms in the  $\{\text{Mo}_4\text{O}_{14}(\text{OH})\}$  core are coplanar, and one oxygen atom in each core is characterized as hydroxyl (O4 and O11) which is indicated by literature and bond valence sum calculation value (Hsu & Wang 1997, Brown *et al.*, 1981). The  $[\text{AsMo}_8\text{O}_{30}\text{H}_2]^{5-}$  cluster anions are bridged together by  $\text{K}^+$  cations into one-dimensional strings firstly. The anion-cation interactions are further extended into a three-dimensional architecture by other two  $\text{K}^+$  cations with each linking four cluster anions and one water molecule.

### Experimental

The title compound was synthesized by hydrothermal reaction of  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  (2.5 mmol),  $\text{As}_2\text{O}_3$  (2.5 mmol),  $\text{MoO}_3 \cdot 2\text{H}_2\text{O}$  (3.0 mmol),  $\text{KOH}$  (5.0 mmol) and  $\text{H}_2\text{O}$  (18 ml) were stirred for 120 min. The pH of the mixture was adjusted to 6.5 with 1M nitric acid. The resultant mixture was sealed in a 25 ml Teflon-lined autoclave and heated at  $180\text{ }^\circ\text{C}$  for 8 d. The autoclave was then cooled to room temperature. The crystalline product was filtered, washed with distilled water and dried at ambient temperature to give black block solids.

### Refinement

The H atoms attached to oxygen molecular were located from difference Fourier maps, and the H atom attached o2w was disordered. The highest peak in the Fourier map is located  $1.30\text{ \AA}$  from O9.

## Figures

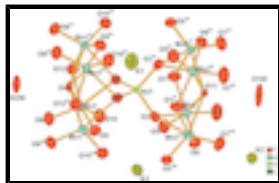


Fig. 1. ORTEP drawing of the title compound with thermal ellipsoids at 50% probability.

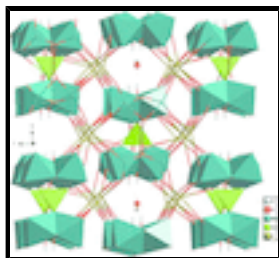


Fig. 2. A polyhedral representation of the three-dimensional network structure of the title compound. All of the water molecular is omitted for clarity. [symmetry codes: (i)  $x, y, z$ ; (ii)  $-x, -y, 1/2 + z$ ; (iii)  $x, -y, -z$ ; (iv)  $-x, y, 1/2 - z$ ; (v)  $-x, -y, -z$ ; (vi)  $x, y, 1/2 + z$ ; (vii)  $-x, y, z$ ; (viii)  $x, -y, 1/2 + z$ .]

## Pentapotassium $\mu$ -arsenato-bis(hydroxytetramolybdate) dihydrate

### Crystal data

$K_5[Mo_8O_{24}(OH)_2(AsO_4)] \cdot 2H_2O$

$M_r = 1553.95$

Orthorhombic,  $Cmcm$

Hall symbol:  $-C 2c 2$

$a = 8.3024$  (12) Å

$b = 23.008$  (3) Å

$c = 15.279$  (2) Å

$V = 2918.6$  (7) Å<sup>3</sup>

$Z = 4$

$F(000) = 2904$

$D_x = 3.541$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 3063 reflections

$\theta = 2.6$ – $26.0^\circ$

$\mu = 5.28$  mm<sup>-1</sup>

$T = 293$  K

Block, black

$0.20 \times 0.18 \times 0.16$  mm

### Data collection

Rigaku R-Axis RAPID  
diffractometer

Radiation source: fine-focus sealed tube  
graphite

Detector resolution: 10 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: multi-scan  
(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.418$ ,  $T_{\max} = 0.486$

12346 measured reflections

1606 independent reflections

1216 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.048$

$\theta_{\max} = 26.0^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -10 \rightarrow 10$

$k = -28 \rightarrow 28$

$l = -18 \rightarrow 18$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

Primary atom site location: structure-invariant direct  
methods

Secondary atom site location: difference Fourier map

$R[F^2 > 2\sigma(F^2)] = 0.032$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.098$	Only H-atom coordinates refined
$S = 1.07$	$w = 1/[\sigma^2(F_o^2) + (0.055P)^2]$
1606 reflections	where $P = (F_o^2 + 2F_c^2)/3$
133 parameters	$(\Delta/\sigma)_{\max} < 0.001$
0 restraints	$\Delta\rho_{\max} = 1.99 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.72 \text{ e } \text{\AA}^{-3}$

*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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O2W	0.5000	0.3660 (4)	0.7500	0.046 (3)	
H2W	0.4128	0.3599	0.7224	0.069*	0.50
Mo1	0.30043 (6)	0.49010 (2)	0.35477 (3)	0.01904 (18)	
Mo2	0.30698 (6)	0.26333 (2)	0.35911 (3)	0.02029 (18)	
As1	0.5000	0.37665 (4)	0.2500	0.0140 (3)	
K1	0.0000	0.38267 (14)	0.2500	0.0504 (10)	
K2	0.5000	0.12103 (8)	0.46558 (14)	0.0300 (5)	
K3	0.5000	0.36453 (9)	0.52457 (14)	0.0341 (5)	
O12	0.1808 (7)	0.5103 (3)	0.2500	0.0332 (16)	
O11	0.5000	0.2312 (3)	0.2500	0.0198 (18)	
O10	0.1715 (6)	0.4466 (2)	0.4095 (3)	0.0379 (12)	
O9	0.5000	0.4605 (3)	0.3962 (4)	0.0328 (15)	
O8	0.2890 (6)	0.55608 (19)	0.4041 (3)	0.0404 (13)	
O7	0.2196 (7)	0.1971 (2)	0.3681 (3)	0.0516 (15)	
O6	0.5000	0.2443 (3)	0.4234 (4)	0.0341 (16)	
O5	0.2029 (6)	0.30763 (19)	0.4280 (3)	0.0382 (12)	
O4	0.5000	0.5206 (3)	0.2500	0.0189 (17)	
O3	0.3350 (7)	0.4194 (2)	0.2500	0.0268 (14)	
O2	0.5000	0.3333 (2)	0.3397 (4)	0.0288 (15)	
O1	0.2301 (8)	0.2928 (3)	0.2500	0.0338 (15)	
O1W	0.5000	0.1146 (4)	0.2500	0.067 (5)	
H1	0.426 (11)	0.091 (4)	0.2500	0.04 (3)*	
H4	0.5000	0.193 (5)	0.2500	0.02 (3)*	
H3	0.5000	0.562 (5)	0.2500	0.02 (3)*	

## supplementary materials

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### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O2W	0.046 (7)	0.017 (5)	0.076 (8)	0.000	0.000	0.000
Mo1	0.0219 (3)	0.0168 (3)	0.0184 (3)	0.0015 (2)	0.0028 (2)	-0.00153 (19)
Mo2	0.0246 (4)	0.0173 (3)	0.0190 (3)	-0.0017 (2)	0.0040 (2)	0.00186 (19)
As1	0.0179 (6)	0.0104 (5)	0.0138 (6)	0.000	0.000	0.000
K1	0.0242 (18)	0.047 (2)	0.080 (3)	0.000	0.000	0.000
K2	0.0328 (12)	0.0281 (11)	0.0291 (11)	0.000	0.000	-0.0002 (8)
K3	0.0390 (13)	0.0324 (11)	0.0310 (11)	0.000	0.000	-0.0039 (9)
O12	0.027 (4)	0.039 (4)	0.034 (4)	0.006 (3)	0.000	0.000
O11	0.032 (5)	0.012 (4)	0.016 (4)	0.000	0.000	0.000
O10	0.036 (3)	0.035 (3)	0.042 (3)	-0.004 (2)	0.016 (2)	0.000 (2)
O9	0.029 (4)	0.038 (4)	0.031 (4)	0.000	0.000	0.005 (3)
O8	0.051 (4)	0.028 (2)	0.042 (3)	0.005 (2)	0.006 (3)	-0.006 (2)
O7	0.058 (4)	0.031 (3)	0.066 (4)	-0.013 (3)	0.021 (3)	0.006 (2)
O6	0.042 (4)	0.035 (4)	0.025 (3)	0.000	0.000	0.005 (3)
O5	0.046 (3)	0.034 (3)	0.034 (3)	0.002 (2)	0.021 (2)	0.000 (2)
O4	0.021 (5)	0.009 (4)	0.027 (4)	0.000	0.000	0.000
O3	0.030 (4)	0.025 (3)	0.025 (3)	-0.006 (3)	0.000	0.000
O2	0.032 (4)	0.027 (3)	0.027 (3)	0.000	0.000	-0.005 (3)
O1	0.029 (4)	0.047 (4)	0.026 (3)	0.003 (3)	0.000	0.000
O1W	0.039 (8)	0.012 (5)	0.150 (14)	0.000	0.000	0.000

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

Mo1—O10	1.687 (4)	K2—O8 <sup>viii</sup>	2.979 (5)
Mo1—O8	1.697 (4)	K2—O8 <sup>ix</sup>	2.979 (5)
Mo1—O9	1.900 (3)	K2—O7 <sup>ii</sup>	3.272 (6)
Mo1—O12	1.940 (3)	K2—O7	3.272 (6)
Mo1—O3	2.300 (4)	K2—O1W	3.297 (2)
Mo1—O4	2.409 (2)	K2—O1W	3.297 (2)
Mo1—Mo1 <sup>i</sup>	3.2015 (10)	K2—Mo2 <sup>ii</sup>	3.9915 (18)
Mo1—K1	3.859 (2)	K3—O8 <sup>x</sup>	2.756 (5)
Mo2—O7	1.692 (5)	K3—O8 <sup>xi</sup>	2.756 (5)
Mo2—O5	1.701 (4)	K3—O7 <sup>vi</sup>	2.833 (5)
Mo2—O1	1.910 (3)	K3—O7 <sup>vii</sup>	2.833 (5)
Mo2—O6	1.930 (3)	K3—O2	2.914 (6)
Mo2—O2	2.291 (4)	K3—O9	2.953 (6)
Mo2—O11	2.427 (2)	K3—O5	3.158 (5)
Mo2—Mo2 <sup>ii</sup>	3.2050 (12)	K3—O5 <sup>ii</sup>	3.158 (5)
Mo2—K3	3.792 (2)	K3—O6	3.168 (6)
Mo2—K2	3.9915 (18)	K3—Mo2 <sup>ii</sup>	3.7922 (19)
Mo2—K1	4.100 (2)	K3—K2 <sup>xii</sup>	4.1672 (6)
As1—O3 <sup>iii</sup>	1.687 (6)	O12—Mo1 <sup>i</sup>	1.940 (3)

As1—O3	1.687 (6)	O11—Mo2 <sup>ii</sup>	2.427 (2)
As1—O2	1.695 (6)	O11—Mo2 <sup>i</sup>	2.427 (2)
As1—O2 <sup>i</sup>	1.695 (6)	O11—Mo2 <sup>iii</sup>	2.427 (2)
K1—O1 <sup>iv</sup>	2.815 (7)	O10—K2 <sup>vii</sup>	2.844 (5)
K1—O1	2.815 (7)	O9—Mo1 <sup>ii</sup>	1.900 (3)
K1—O3	2.907 (6)	O8—K3 <sup>x</sup>	2.756 (5)
K1—O3 <sup>iv</sup>	2.907 (6)	O8—K2 <sup>xiii</sup>	2.979 (5)
K1—O10	3.183 (5)	O7—K3 <sup>vii</sup>	2.833 (5)
K1—O10 <sup>v</sup>	3.183 (5)	O6—Mo2 <sup>ii</sup>	1.930 (3)
K1—O10 <sup>iv</sup>	3.183 (5)	O5—K2 <sup>vii</sup>	2.859 (4)
K1—O10 <sup>i</sup>	3.183 (5)	O4—Mo1 <sup>i</sup>	2.409 (2)
K1—O12 <sup>iv</sup>	3.298 (7)	O4—Mo1 <sup>ii</sup>	2.409 (2)
K1—O12	3.298 (7)	O4—Mo1 <sup>iii</sup>	2.409 (2)
K1—Mo1 <sup>i</sup>	3.859 (2)	O3—Mo1 <sup>i</sup>	2.300 (4)
K2—O10 <sup>vi</sup>	2.844 (5)	O2—Mo2 <sup>ii</sup>	2.291 (4)
K2—O10 <sup>vii</sup>	2.844 (5)	O1—Mo2 <sup>i</sup>	1.910 (3)
K2—O5 <sup>vi</sup>	2.859 (4)	O1W—O1W	0.00 (2)
K2—O5 <sup>vii</sup>	2.859 (4)	O1W—K2 <sup>i</sup>	3.297 (2)
K2—O6	2.909 (6)		
O10—Mo1—O8	106.0 (2)	O10 <sup>vi</sup> —K2—O7 <sup>ii</sup>	104.00 (13)
O10—Mo1—O9	100.1 (2)	O10 <sup>vii</sup> —K2—O7 <sup>ii</sup>	162.64 (14)
O8—Mo1—O9	102.8 (3)	O5 <sup>vi</sup> —K2—O7 <sup>ii</sup>	62.12 (14)
O10—Mo1—O12	103.1 (2)	O5 <sup>vii</sup> —K2—O7 <sup>ii</sup>	111.77 (14)
O8—Mo1—O12	97.1 (2)	O6—K2—O7 <sup>ii</sup>	51.49 (10)
O9—Mo1—O12	143.8 (2)	O8 <sup>viii</sup> —K2—O7 <sup>ii</sup>	134.27 (14)
O10—Mo1—O3	90.3 (2)	O8 <sup>ix</sup> —K2—O7 <sup>ii</sup>	63.37 (14)
O8—Mo1—O3	161.5 (2)	O10 <sup>vi</sup> —K2—O7	162.64 (14)
O9—Mo1—O3	82.5 (2)	O10 <sup>vii</sup> —K2—O7	104.00 (13)
O12—Mo1—O3	70.04 (19)	O5 <sup>vi</sup> —K2—O7	111.77 (14)
O10—Mo1—O4	159.8 (2)	O5 <sup>vii</sup> —K2—O7	62.12 (13)
O8—Mo1—O4	94.2 (2)	O6—K2—O7	51.49 (10)
O9—Mo1—O4	74.11 (18)	O8 <sup>viii</sup> —K2—O7	63.37 (13)
O12—Mo1—O4	74.57 (16)	O8 <sup>ix</sup> —K2—O7	134.27 (14)
O3—Mo1—O4	70.0 (2)	O7 <sup>ii</sup> —K2—O7	90.72 (18)
O10—Mo1—Mo1 <sup>i</sup>	119.71 (17)	O10 <sup>vi</sup> —K2—O1W	130.22 (18)
O8—Mo1—Mo1 <sup>i</sup>	116.36 (16)	O10 <sup>vii</sup> —K2—O1W	130.22 (18)
O9—Mo1—Mo1 <sup>i</sup>	109.46 (18)	O5 <sup>vi</sup> —K2—O1W	126.43 (16)
O12—Mo1—Mo1 <sup>i</sup>	34.40 (15)	O5 <sup>vii</sup> —K2—O1W	126.43 (16)
O3—Mo1—Mo1 <sup>i</sup>	45.89 (10)	O6—K2—O1W	79.8 (2)
O4—Mo1—Mo1 <sup>i</sup>	48.35 (5)	O8 <sup>viii</sup> —K2—O1W	70.27 (14)
O10—Mo1—K1	54.24 (16)	O8 <sup>ix</sup> —K2—O1W	70.27 (14)

## supplementary materials

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O8—Mo1—K1	136.12 (19)	O7 <sup>ii</sup> —K2—O1W	64.48 (14)
O9—Mo1—K1	118.16 (19)	O7—K2—O1W	64.48 (14)
O12—Mo1—K1	58.68 (19)	O10 <sup>vi</sup> —K2—O1W	130.22 (18)
O3—Mo1—K1	48.63 (15)	O10 <sup>vii</sup> —K2—O1W	130.22 (18)
O4—Mo1—K1	110.84 (10)	O5 <sup>vi</sup> —K2—O1W	126.43 (16)
Mo1 <sup>i</sup> —Mo1—K1	65.495 (16)	O5 <sup>vii</sup> —K2—O1W	126.43 (16)
O7—Mo2—O5	105.7 (2)	O6—K2—O1W	79.8 (2)
O7—Mo2—O1	104.3 (3)	O8 <sup>viii</sup> —K2—O1W	70.27 (14)
O5—Mo2—O1	99.1 (2)	O8 <sup>ix</sup> —K2—O1W	70.27 (14)
O7—Mo2—O6	96.4 (3)	O7 <sup>ii</sup> —K2—O1W	64.48 (14)
O5—Mo2—O6	104.0 (2)	O7—K2—O1W	64.48 (14)
O1—Mo2—O6	143.4 (2)	O1W—K2—O1W	0.0 (4)
O7—Mo2—O2	160.5 (2)	O10 <sup>vi</sup> —K2—Mo2 <sup>ii</sup>	121.40 (10)
O5—Mo2—O2	90.80 (19)	O10 <sup>vii</sup> —K2—Mo2 <sup>ii</sup>	157.37 (11)
O1—Mo2—O2	82.6 (2)	O5 <sup>vi</sup> —K2—Mo2 <sup>ii</sup>	61.60 (9)
O6—Mo2—O2	69.15 (19)	O5 <sup>vii</sup> —K2—Mo2 <sup>ii</sup>	89.85 (10)
O7—Mo2—O11	93.7 (2)	O6—K2—Mo2 <sup>ii</sup>	27.12 (6)
O5—Mo2—O11	160.5 (2)	O8 <sup>viii</sup> —K2—Mo2 <sup>ii</sup>	127.33 (10)
O1—Mo2—O11	74.28 (19)	O8 <sup>ix</sup> —K2—Mo2 <sup>ii</sup>	87.68 (9)
O6—Mo2—O11	74.49 (16)	O7 <sup>ii</sup> —K2—Mo2 <sup>ii</sup>	24.47 (9)
O2—Mo2—O11	70.33 (19)	O7—K2—Mo2 <sup>ii</sup>	70.20 (9)
O7—Mo2—Mo2 <sup>ii</sup>	115.4 (2)	O1W—K2—Mo2 <sup>ii</sup>	68.26 (16)
O5—Mo2—Mo2 <sup>ii</sup>	120.53 (17)	O1W—K2—Mo2 <sup>ii</sup>	68.26 (16)
O1—Mo2—Mo2 <sup>ii</sup>	109.53 (19)	O8 <sup>x</sup> —K3—O8 <sup>xi</sup>	78.9 (2)
O6—Mo2—Mo2 <sup>ii</sup>	33.86 (14)	O8 <sup>x</sup> —K3—O7 <sup>vi</sup>	72.18 (16)
O2—Mo2—Mo2 <sup>ii</sup>	45.62 (10)	O8 <sup>xi</sup> —K3—O7 <sup>vi</sup>	120.80 (17)
O11—Mo2—Mo2 <sup>ii</sup>	48.69 (5)	O8 <sup>x</sup> —K3—O7 <sup>vii</sup>	120.80 (17)
O7—Mo2—K3	132.82 (18)	O8 <sup>xi</sup> —K3—O7 <sup>vii</sup>	72.18 (16)
O5—Mo2—K3	55.55 (17)	O7 <sup>vi</sup> —K3—O7 <sup>vii</sup>	80.1 (2)
O1—Mo2—K3	120.34 (19)	O8 <sup>x</sup> —K3—O2	123.14 (14)
O6—Mo2—K3	56.54 (18)	O8 <sup>xi</sup> —K3—O2	123.14 (14)
O2—Mo2—K3	50.13 (14)	O7 <sup>vi</sup> —K3—O2	115.96 (14)
O11—Mo2—K3	111.44 (10)	O7 <sup>vii</sup> —K3—O2	115.96 (14)
Mo2 <sup>ii</sup> —Mo2—K3	65.002 (16)	O8 <sup>x</sup> —K3—O9	76.54 (14)
O7—Mo2—K2	53.2 (2)	O8 <sup>xi</sup> —K3—O9	76.54 (14)
O5—Mo2—K2	116.31 (15)	O7 <sup>vi</sup> —K3—O9	139.37 (12)
O1—Mo2—K2	141.38 (18)	O7 <sup>vii</sup> —K3—O9	139.37 (12)
O6—Mo2—K2	43.40 (17)	O2—K3—O9	62.64 (17)
O2—Mo2—K2	110.39 (11)	O8 <sup>x</sup> —K3—O5	162.95 (14)
O11—Mo2—K2	76.43 (14)	O8 <sup>xi</sup> —K3—O5	87.88 (13)
Mo2 <sup>ii</sup> —Mo2—K2	66.329 (14)	O7 <sup>vi</sup> —K3—O5	124.43 (15)



K3—Mo2—K2	93.57 (4)	O7 <sup>vii</sup> —K3—O5	63.91 (14)
O7—Mo2—K1	111.7 (2)	O2—K3—O5	56.30 (9)
O5—Mo2—K1	62.27 (16)	O9—K3—O5	89.98 (12)
O1—Mo2—K1	36.82 (17)	O8 <sup>x</sup> —K3—O5 <sup>ii</sup>	87.88 (13)
O6—Mo2—K1	151.01 (18)	O8 <sup>xi</sup> —K3—O5 <sup>ii</sup>	162.95 (14)
O2—Mo2—K1	84.92 (11)	O7 <sup>vi</sup> —K3—O5 <sup>ii</sup>	63.91 (14)
O11—Mo2—K1	109.56 (11)	O7 <sup>vii</sup> —K3—O5 <sup>ii</sup>	124.43 (15)
Mo2 <sup>ii</sup> —Mo2—K1	128.43 (3)	O2—K3—O5 <sup>ii</sup>	56.30 (9)
K3—Mo2—K1	97.04 (4)	O9—K3—O5 <sup>ii</sup>	89.98 (12)
K2—Mo2—K1	164.67 (4)	O5—K3—O5 <sup>ii</sup>	102.71 (16)
O3 <sup>iii</sup> —As1—O3	108.6 (4)	O8 <sup>x</sup> —K3—O6	140.50 (10)
O3 <sup>iii</sup> —As1—O2	110.06 (13)	O8 <sup>xi</sup> —K3—O6	140.50 (10)
O3—As1—O2	110.06 (13)	O7 <sup>vi</sup> —K3—O6	81.10 (14)
O3 <sup>iii</sup> —As1—O2 <sup>i</sup>	110.06 (13)	O7 <sup>vii</sup> —K3—O6	81.10 (14)
O3—As1—O2 <sup>i</sup>	110.06 (13)	O2—K3—O6	46.54 (15)
O2—As1—O2 <sup>i</sup>	108.0 (4)	O9—K3—O6	109.18 (17)
O1 <sup>iv</sup> —K1—O1	85.5 (3)	O5—K3—O6	53.87 (9)
O1 <sup>iv</sup> —K1—O3	149.7 (2)	O5 <sup>ii</sup> —K3—O6	53.87 (9)
O1—K1—O3	64.18 (17)	O8 <sup>x</sup> —K3—Mo2	159.94 (12)
O1 <sup>iv</sup> —K1—O3 <sup>iv</sup>	64.18 (17)	O8 <sup>xi</sup> —K3—Mo2	113.71 (10)
O1—K1—O3 <sup>iv</sup>	149.7 (2)	O7 <sup>vi</sup> —K3—Mo2	110.51 (12)
O3—K1—O3 <sup>iv</sup>	146.2 (3)	O7 <sup>vii</sup> —K3—Mo2	78.84 (11)
O1 <sup>iv</sup> —K1—O10	130.02 (9)	O2—K3—Mo2	37.12 (8)
O1—K1—O10	92.05 (13)	O9—K3—Mo2	90.93 (12)
O3—K1—O10	55.77 (9)	O5—K3—Mo2	26.37 (8)
O3 <sup>iv</sup> —K1—O10	107.08 (13)	O5 <sup>ii</sup> —K3—Mo2	76.36 (9)
O1 <sup>iv</sup> —K1—O10 <sup>v</sup>	92.05 (13)	O6—K3—Mo2	30.55 (6)
O1—K1—O10 <sup>v</sup>	130.02 (9)	O8 <sup>x</sup> —K3—Mo2 <sup>ii</sup>	113.71 (10)
O3—K1—O10 <sup>v</sup>	107.08 (13)	O8 <sup>xi</sup> —K3—Mo2 <sup>ii</sup>	159.94 (12)
O3 <sup>iv</sup> —K1—O10 <sup>v</sup>	55.77 (9)	O7 <sup>vi</sup> —K3—Mo2 <sup>ii</sup>	78.84 (11)
O10—K1—O10 <sup>v</sup>	53.16 (17)	O7 <sup>vii</sup> —K3—Mo2 <sup>ii</sup>	110.51 (12)
O1 <sup>iv</sup> —K1—O10 <sup>iv</sup>	92.05 (13)	O2—K3—Mo2 <sup>ii</sup>	37.12 (8)
O1—K1—O10 <sup>iv</sup>	130.02 (9)	O9—K3—Mo2 <sup>ii</sup>	90.93 (12)
O3—K1—O10 <sup>iv</sup>	107.08 (13)	O5—K3—Mo2 <sup>ii</sup>	76.36 (9)
O3 <sup>iv</sup> —K1—O10 <sup>iv</sup>	55.77 (9)	O5 <sup>ii</sup> —K3—Mo2 <sup>ii</sup>	26.37 (8)
O10—K1—O10 <sup>iv</sup>	125.0 (2)	O6—K3—Mo2 <sup>ii</sup>	30.55 (6)
O10 <sup>v</sup> —K1—O10 <sup>iv</sup>	99.94 (17)	Mo2—K3—Mo2 <sup>ii</sup>	50.00 (3)
O1 <sup>iv</sup> —K1—O10 <sup>i</sup>	130.02 (9)	O8 <sup>x</sup> —K3—K2 <sup>xii</sup>	45.54 (10)
O1—K1—O10 <sup>i</sup>	92.05 (13)	O8 <sup>xi</sup> —K3—K2 <sup>xii</sup>	124.48 (12)
O3—K1—O10 <sup>i</sup>	55.77 (9)	O7 <sup>vi</sup> —K3—K2 <sup>xii</sup>	51.54 (12)
O3 <sup>iv</sup> —K1—O10 <sup>i</sup>	107.08 (13)	O7 <sup>vii</sup> —K3—K2 <sup>xii</sup>	131.30 (14)

## supplementary materials

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O10—K1—O10 <sup>i</sup>	99.94 (17)	O2—K3—K2 <sup>xii</sup>	93.13 (4)
O10 <sup>v</sup> —K1—O10 <sup>i</sup>	125.0 (2)	O9—K3—K2 <sup>xii</sup>	87.96 (4)
O10 <sup>iv</sup> —K1—O10 <sup>i</sup>	53.16 (17)	O5—K3—K2 <sup>xii</sup>	145.89 (10)
O1 <sup>iv</sup> —K1—O12 <sup>iv</sup>	110.18 (16)	O5 <sup>ii</sup> —K3—K2 <sup>xii</sup>	43.27 (8)
O1—K1—O12 <sup>iv</sup>	164.35 (19)	O6—K3—K2 <sup>xii</sup>	95.00 (4)
O3—K1—O12 <sup>iv</sup>	100.17 (18)	Mo2—K3—K2 <sup>xii</sup>	119.60 (5)
O3 <sup>iv</sup> —K1—O12 <sup>iv</sup>	46.00 (15)	Mo2 <sup>ii</sup> —K3—K2 <sup>xii</sup>	69.64 (3)
O10—K1—O12 <sup>iv</sup>	78.02 (13)	Mo1—O12—Mo1 <sup>i</sup>	111.2 (3)
O10 <sup>v</sup> —K1—O12 <sup>iv</sup>	52.04 (9)	Mo1—O12—K1	91.2 (2)
O10 <sup>iv</sup> —K1—O12 <sup>iv</sup>	52.04 (9)	Mo1 <sup>i</sup> —O12—K1	91.2 (2)
O10 <sup>i</sup> —K1—O12 <sup>iv</sup>	78.02 (13)	Mo2 <sup>ii</sup> —O11—Mo2 <sup>i</sup>	144.6 (3)
O1 <sup>iv</sup> —K1—O12	164.35 (19)	Mo2 <sup>ii</sup> —O11—Mo2 <sup>iii</sup>	86.75 (11)
O1—K1—O12	110.18 (16)	Mo2 <sup>i</sup> —O11—Mo2 <sup>iii</sup>	82.63 (10)
O3—K1—O12	46.00 (15)	Mo2 <sup>ii</sup> —O11—Mo2	82.63 (10)
O3 <sup>iv</sup> —K1—O12	100.17 (18)	Mo2 <sup>i</sup> —O11—Mo2	86.75 (11)
O10—K1—O12	52.04 (9)	Mo2 <sup>iii</sup> —O11—Mo2	144.6 (3)
O10 <sup>v</sup> —K1—O12	78.02 (13)	Mo1—O10—K2 <sup>vii</sup>	167.1 (3)
O10 <sup>iv</sup> —K1—O12	78.02 (13)	Mo1—O10—K1	100.3 (2)
O10 <sup>i</sup> —K1—O12	52.04 (9)	K2 <sup>vii</sup> —O10—K1	92.12 (14)
O12 <sup>iv</sup> —K1—O12	54.2 (2)	Mo1 <sup>ii</sup> —O9—Mo1	121.4 (3)
O1 <sup>iv</sup> —K1—Mo1	155.37 (3)	Mo1 <sup>ii</sup> —O9—K3	119.31 (16)
O1—K1—Mo1	91.82 (12)	Mo1—O9—K3	119.31 (16)
O3—K1—Mo1	36.42 (8)	Mo1—O8—K3 <sup>x</sup>	137.1 (3)
O3 <sup>iv</sup> —K1—Mo1	115.59 (13)	Mo1—O8—K2 <sup>xiii</sup>	129.3 (3)
O10—K1—Mo1	25.48 (8)	K3 <sup>x</sup> —O8—K2 <sup>xiii</sup>	93.14 (13)
O10 <sup>v</sup> —K1—Mo1	71.07 (10)	Mo2—O7—K3 <sup>vii</sup>	140.7 (3)
O10 <sup>iv</sup> —K1—Mo1	108.11 (12)	Mo2—O7—K2	102.3 (3)
O10 <sup>i</sup> —K1—Mo1	74.48 (9)	K3 <sup>vii</sup> —O7—K2	85.77 (13)
O12 <sup>iv</sup> —K1—Mo1	73.98 (11)	Mo2 <sup>ii</sup> —O6—Mo2	112.3 (3)
O12—K1—Mo1	30.17 (6)	Mo2 <sup>ii</sup> —O6—K2	109.48 (19)
O1 <sup>iv</sup> —K1—Mo1 <sup>i</sup>	155.37 (3)	Mo2—O6—K2	109.48 (19)
O1—K1—Mo1 <sup>i</sup>	91.82 (12)	Mo2 <sup>ii</sup> —O6—K3	92.91 (19)
O3—K1—Mo1 <sup>i</sup>	36.42 (8)	Mo2—O6—K3	92.91 (19)
O3 <sup>iv</sup> —K1—Mo1 <sup>i</sup>	115.59 (13)	K2—O6—K3	138.0 (2)
O10—K1—Mo1 <sup>i</sup>	74.48 (9)	Mo2—O5—K2 <sup>vii</sup>	174.3 (3)
O10 <sup>v</sup> —K1—Mo1 <sup>i</sup>	108.11 (12)	Mo2—O5—K3	98.1 (2)
O10 <sup>iv</sup> —K1—Mo1 <sup>i</sup>	71.07 (10)	K2 <sup>vii</sup> —O5—K3	87.51 (12)
O10 <sup>i</sup> —K1—Mo1 <sup>i</sup>	25.48 (8)	Mo1 <sup>i</sup> —O4—Mo1 <sup>ii</sup>	146.1 (3)
O12 <sup>iv</sup> —K1—Mo1 <sup>i</sup>	73.98 (11)	Mo1 <sup>i</sup> —O4—Mo1	83.31 (9)
O12—K1—Mo1 <sup>i</sup>	30.17 (6)	Mo1 <sup>ii</sup> —O4—Mo1	86.93 (10)
Mo1—K1—Mo1 <sup>i</sup>	49.01 (3)	Mo1 <sup>i</sup> —O4—Mo1 <sup>iii</sup>	86.93 (10)

O10 <sup>vi</sup> —K2—O10 <sup>vii</sup>	60.1 (2)	Mo1 <sup>ii</sup> —O4—Mo1 <sup>iii</sup>	83.31 (9)
O10 <sup>vi</sup> —K2—O5 <sup>vi</sup>	68.74 (15)	Mo1—O4—Mo1 <sup>iii</sup>	146.1 (3)
O10 <sup>vii</sup> —K2—O5 <sup>vi</sup>	103.14 (16)	As1—O3—Mo1 <sup>i</sup>	120.9 (2)
O10 <sup>vi</sup> —K2—O5 <sup>vii</sup>	103.14 (16)	As1—O3—Mo1	120.9 (2)
O10 <sup>vii</sup> —K2—O5 <sup>vii</sup>	68.74 (15)	Mo1 <sup>i</sup> —O3—Mo1	88.2 (2)
O5 <sup>vi</sup> —K2—O5 <sup>vii</sup>	72.2 (2)	As1—O3—K1	127.4 (3)
O10 <sup>vi</sup> —K2—O6	132.99 (14)	Mo1 <sup>i</sup> —O3—K1	94.95 (18)
O10 <sup>vii</sup> —K2—O6	132.99 (14)	Mo1—O3—K1	94.95 (18)
O5 <sup>vi</sup> —K2—O6	64.28 (13)	As1—O2—Mo2	121.18 (19)
O5 <sup>vii</sup> —K2—O6	64.28 (13)	As1—O2—Mo2 <sup>ii</sup>	121.18 (19)
O10 <sup>vi</sup> —K2—O8 <sup>viii</sup>	109.91 (14)	Mo2—O2—Mo2 <sup>ii</sup>	88.8 (2)
O10 <sup>vii</sup> —K2—O8 <sup>viii</sup>	62.22 (14)	As1—O2—K3	129.7 (3)
O5 <sup>vi</sup> —K2—O8 <sup>viii</sup>	160.37 (15)	Mo2—O2—K3	92.75 (17)
O5 <sup>vii</sup> —K2—O8 <sup>viii</sup>	89.59 (13)	Mo2 <sup>ii</sup> —O2—K3	92.75 (17)
O6—K2—O8 <sup>viii</sup>	114.80 (11)	Mo2—O1—Mo2 <sup>i</sup>	121.6 (3)
O10 <sup>vi</sup> —K2—O8 <sup>ix</sup>	62.22 (14)	Mo2—O1—K1	119.19 (17)
O10 <sup>vii</sup> —K2—O8 <sup>ix</sup>	109.91 (14)	Mo2 <sup>i</sup> —O1—K1	119.19 (17)
O5 <sup>vi</sup> —K2—O8 <sup>ix</sup>	89.59 (13)	O1W—O1W—K2 <sup>i</sup>	0(10)
O5 <sup>vii</sup> —K2—O8 <sup>ix</sup>	160.37 (15)	O1W—O1W—K2	0(10)
O6—K2—O8 <sup>ix</sup>	114.80 (11)	K2 <sup>i</sup> —O1W—K2	174.9 (4)
O8 <sup>viii</sup> —K2—O8 <sup>ix</sup>	107.32 (19)		

Symmetry codes: (i)  $x, y, -z+1/2$ ; (ii)  $-x+1, y, z$ ; (iii)  $-x+1, y, -z+1/2$ ; (iv)  $-x, y, -z+1/2$ ; (v)  $-x, y, z$ ; (vi)  $x+1/2, -y+1/2, -z+1$ ; (vii)  $-x+1/2, -y+1/2, -z+1$ ; (viii)  $-x+1/2, y-1/2, z$ ; (ix)  $x+1/2, y-1/2, z$ ; (x)  $-x+1, -y+1, -z+1$ ; (xi)  $x, -y+1, -z+1$ ; (xii)  $-x+3/2, -y+1/2, -z+1$ ; (xiii)  $x-1/2, y+1/2, z$ .

*Hydrogen-bond geometry* (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—H1 <sup>⋯</sup> O12 <sup>xiv</sup>	0.82 (9)	2.06 (9)	2.831 (11)	157 (9)
O11—H4 <sup>⋯</sup> O1W	0.87 (11)	1.81 (11)	2.684 (13)	180.(1)

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

Fig. 1

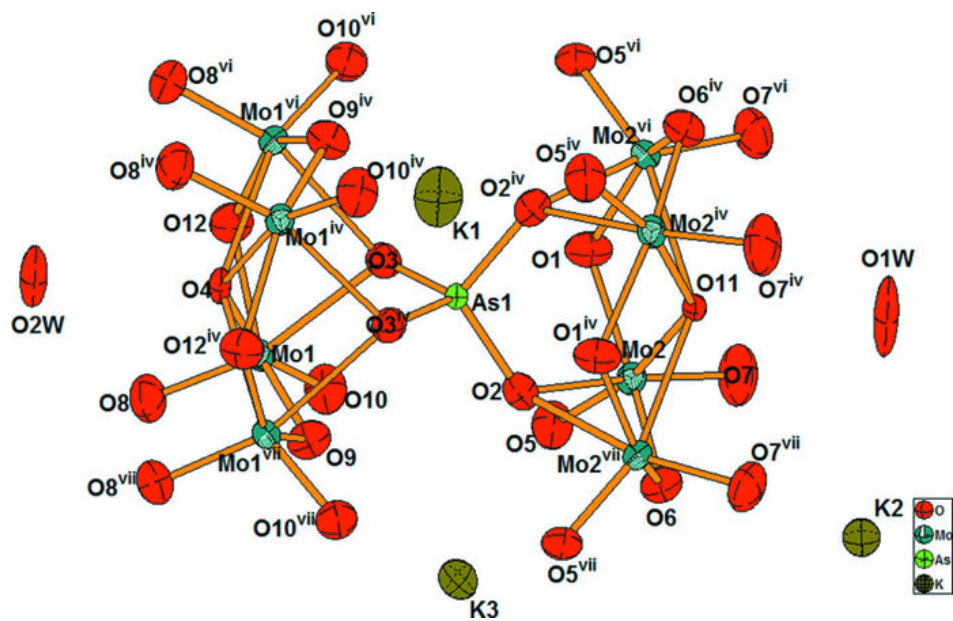


Fig. 2

