



# Crystal structures and comparisons of potassium rare-earth molybdates $KRE(\text{MoO}_4)_2$ ( $RE = \text{Tb}, \text{Dy}, \text{Ho}, \text{Er}, \text{Yb}, \text{and Lu}$ )

Saehwa Chong,<sup>a\*</sup> Samuel Perry,<sup>b</sup> Brian J. Riley<sup>a</sup> and Zayne J. Nelson<sup>a</sup>

<sup>a</sup>Pacific Northwest National Laboratory, Richland, WA 99354, USA, and <sup>b</sup>Department of Civil and Environmental Engineering and Earth Sciences, University of Notre Dame, Notre Dame, IN 46556, USA. \*Correspondence e-mail: saehwa.chong@pnnl.gov

Received 20 July 2020

Accepted 20 November 2020

Edited by P. Roussel, ENSCL, France

**Keywords:** rare-earth molybdate; lanthanide molybdate; single-crystal XRD.

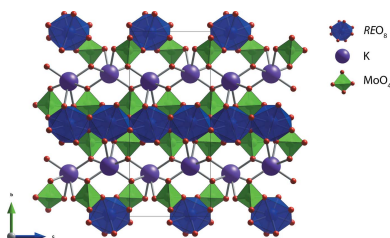
**CCDC references:** 2045648; 2045647; 2045646; 2045645; 2045644; 2045643

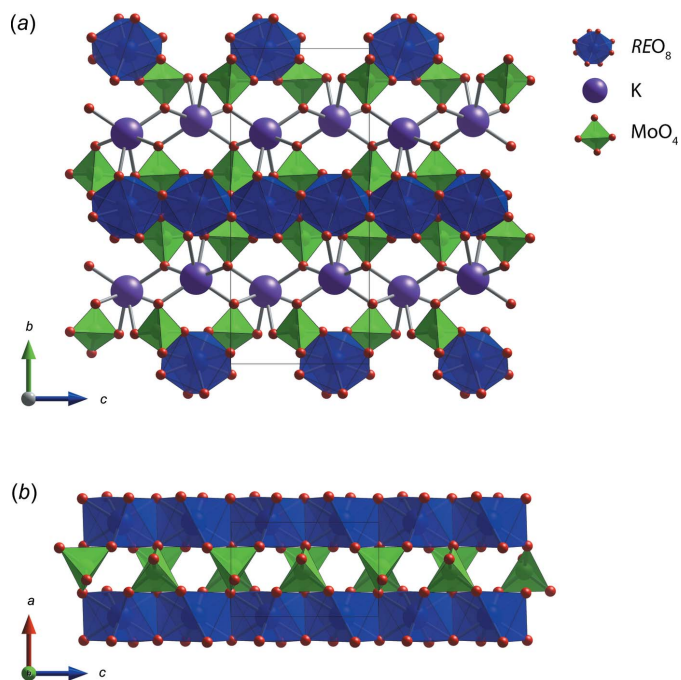
**Supporting information:** this article has supporting information at journals.iucr.org/e

Six potassium rare-earth molybdates  $KRE(\text{MoO}_4)_2$  ( $RE = \text{Tb}, \text{Dy}, \text{Ho}, \text{Er}, \text{Yb}, \text{and Lu}$ ) were synthesized by flux-assisted growth in  $\text{K}_2\text{Mo}_3\text{O}_{10}$ . The crystal structures were determined using single-crystal X-ray diffraction data. The synthesized molybdates crystallize with the orthorhombic  $Pbcn$  space group (No. 60). Trendlines for unit-cell parameters were calculated using data from the current study. The unit-cell parameters  $a$  and  $c$  increase linearly whereas  $b$  decreases with larger  $RE$  cations, based on crystal radii. The unit-cell volumes increase linearly and the densities decrease linearly with larger  $RE$  cations. The average distances between the  $RE$  cations and the nearest O atoms increase with larger cations whereas the average distances of  $\text{Mo}-\text{O}$  and  $\text{K}-\text{O}$  do not show specific trends.

## 1. Chemical context

Rare-earth ( $RE$ ) molybdates have been studied extensively because of their luminescent, magnetoelectric, and ferroelectric properties (Borchardt & Bierstedt, 1967; Axe *et al.*, 1971; Pratap *et al.*, 1987; Ponomarev *et al.*, 1994; Shi *et al.*, 1996; Kut'ko, 2005; Wang *et al.*, 2007; Ponomarev & Zhukov, 2012). The  $RE$  molybdates of  $ARE(\text{MoO}_4)_2$  ( $A = \text{Li}, \text{Na}, \text{K}, \text{Rb}, \text{Cs}, \text{Ag}$ ) generally crystallize with the tetragonal  $I4_1/a$  space group with the scheelite ( $\text{CaWO}_4$ ) structure or the orthorhombic  $Pbcn$  space group (Wanklyn & Wondre, 1978; Hanuza & Fomitsev, 1980; Leask *et al.*, 1981; Hanuza *et al.*, 1994; Stedman *et al.*, 1994; Shi *et al.*, 1996; Voron'ko *et al.*, 2004; Kut'ko, 2005; Wang *et al.*, 2007; Mat'aš *et al.*, 2010; Poperezhai *et al.*, 2017). The  $ARE(\text{MoO}_4)_2$  compounds having the  $I4_1/a$  space group have luminescent properties with high thermal and hydrolytic stability (Stedman *et al.*, 1994; Shi *et al.*, 1996; Voron'ko *et al.*, 2004; Wang *et al.*, 2007) whereas the compounds with the  $Pbcn$  space group are known for the structural phase transition by the Jahn–Teller effect (Kut'ko, 2005; Mat'aš *et al.*, 2010; Kamenskyi *et al.*, 2014; Poperezhai *et al.*, 2017). Other well-known  $RE$  molybdates  $RE_2(\text{MoO}_4)_3$  ( $RE = \text{La}, \text{Ce}, \text{Pr}, \text{Nd}, \text{Sm}, \text{Eu}, \text{Gd}, \text{Tb}, \text{Dy}$ ) crystallize with different space groups including  $P2_1/c$ ,  $C2/c$ , or  $P2m$  depending on the  $RE$  cations and the synthesis conditions (Brixner *et al.*, 1979; Jeitschko, 1973; Ponomarev & Zhukov, 2012; Pratap *et al.*, 1987); these phases exhibit magnetoelectric and ferroelectric properties (Borchardt & Bierstedt, 1967; Axe *et al.*, 1971; Ponomarev *et al.*, 1994; Ponomarev & Zhukov, 2012). The  $RE$  molybdate compounds are synthesized using flux-assisted or solid-state synthesis methods. Wanklyn & Wondre (1978) synthesized





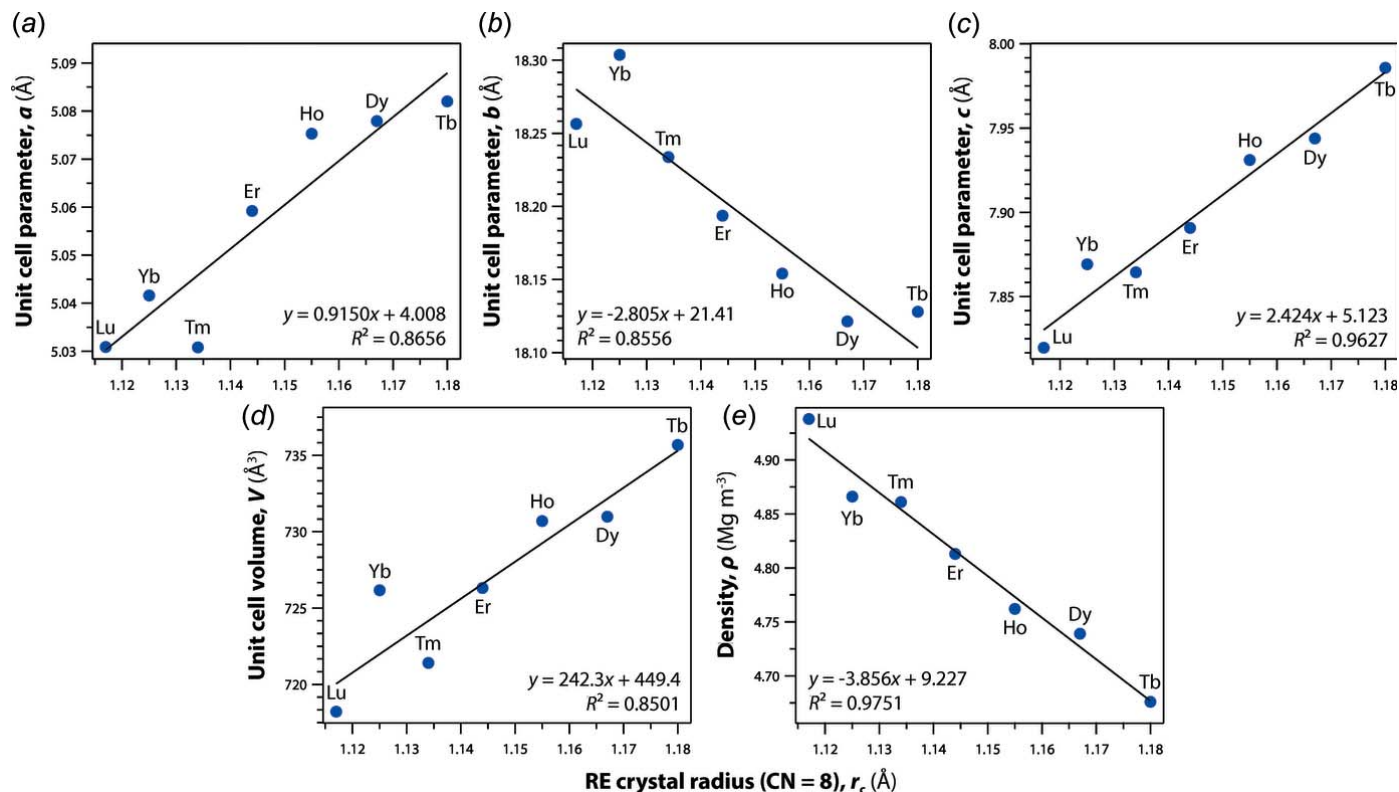
**Figure 1**  
(a) Crystal structure of  $KRE(MoO_4)_2$  and (b)  $[RE(MoO_4)_2]^-$  layer showing the chains composed of  $REO_8$  octahedra connected by  $MoO_4$  tetrahedra along the  $c$ -axis direction.

$KRE(MoO_4)_2$  ( $RE = La, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Lu$ ) compounds by the flux-assisted method using  $REO_x$ ,

$MoO_3$ , and  $K_2SO_4$  at  $1000^\circ C$  for 24 h. They reported that crystals containing  $RE = Tb \rightarrow Lu$  crystallized in the  $Pbcn$  space group whereas  $RE = La$  and  $Pr$  crystallized in the  $I4_1/a$  space group and others were not defined (Wanklyn & Wondre, 1978). Shi *et al.* (1996) synthesized the  $AgRE(MoO_4)_2$  ( $RE = Eu, Gd, Tb$ ) compounds with a tetragonal scheelite-type structure by heating the stoichiometric mixtures of  $REO_x$ ,  $Ag_2O$ , and  $MoO_3$  at  $800^\circ C$  for 50 h. Wang *et al.* (2007) synthesized the tetragonal  $AEu(MoO_4)_2$  ( $A = Li, Na, K$ ) compounds by heating a mixture of  $REO_x$ ,  $LiCO_3$ ,  $NaHCO_3$ ,  $K_2CO_3$ , and  $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$  at  $550\text{--}750^\circ C$  for 4 h. The  $RE_2(MoO_4)_3$  compounds were synthesized by heating a mixture of  $REO_x$  and  $MoO_3$  at  $900\text{--}1000^\circ C$  (Borchardt & Bierstedt, 1967; Guzmán-Afonso *et al.*, 2013).

## 2. Structural commentary

The title  $KRE(MoO_4)_2$  compounds crystallized in the orthorhombic  $Pbcn$  space group and have alternating layers of  $[RE(MoO_4)_2]^-$  and  $K^+$  ions (Fig. 1a). The  $[RE(MoO_4)_2]^-$  layer contains chains formed by edge-sharing  $REO_8$  octahedra connected by  $MoO_4$  tetrahedra along the  $c$ -axis direction (Fig. 1b). The trendlines of the structural parameters were calculated using data from the current study. The unit-cell parameters of  $a$  and  $c$  increase while those of  $b$  decrease linearly with increasing size of the  $RE$  cations (Fig. 2). Although these trends are shown clearly, there are large deviations from the trendlines for some molybdates including



**Figure 2**  
Summary of (a) unit-cell parameter  $a$ , (b) unit-cell parameter  $b$ , (c) unit-cell parameter  $c$ , (d) unit-cell volume ( $V$ ), and (e) density ( $\rho$ ) as a function of the average crystal radii of the  $RE$  in the crystal structures (coordination number = 8) from Shannon (1976).

**Table 1**

 Bond-valence (v.u.) calculations for the title  $KRE(\text{MoO}_4)_2$  compounds.

Detailed tables are included in the supporting information.

	KTb(MoO <sub>4</sub> ) <sub>2</sub>	KDy(MoO <sub>4</sub> ) <sub>2</sub>	KHo(MoO <sub>4</sub> ) <sub>2</sub>	KEr(MoO <sub>4</sub> ) <sub>2</sub>	KYb(MoO <sub>4</sub> ) <sub>2</sub>	KLu(MoO <sub>4</sub> ) <sub>2</sub>
RE	3.11	2.97	3.25	3.00	2.97	3.13
Mo	5.66	5.66	5.62	5.65	5.66	5.70
K	1.20	1.21	1.19	1.20	1.10	1.13
O <sub>1</sub>	1.99	1.97	2.00	1.98	1.98	2.00
O <sub>2</sub>	1.82	1.80	1.82	1.80	1.79	1.82
O <sub>3</sub>	1.90	1.92	1.97	1.92	1.91	1.91
O <sub>4</sub>	2.06	2.07	2.07	2.05	2.03	2.09

Tb and Tm molybdates for unit-cell parameter  $a$ , and Tb and Yb for unit-cell parameter  $b$ . The unit-cell volume of the Yb compound also shows a large deviation. Compared to the structural parameters from the previous studies (PDF 00-050-1762 and PDF 00-052-1688) on the Yb compound, the cell length  $b$  is longer by  $\sim 0.02 \text{ \AA}$ , and the unit-cell volume is larger by  $\sim 2 \text{ \AA}^3$ . The structural parameters in these previous studies are from powder samples whereas the data in this study are from single-crystal studies. These inconsistencies could be due to possible mixed valences of RE or non-stoichiometry of RE sites. However, the bond-valence calculations for all the  $KRE(\text{MoO}_4)_2$  compounds show that the bond-valence sums of RE cations are close to 3 (Table 1). The average distances between the RE cations and neighboring O atoms increase with larger RE cations whereas there are no trends for  $\langle \text{Mo}-\text{O} \rangle$  or  $\langle \text{K}-\text{O} \rangle$  (Fig. 3). Further investigation should be done in the future to understand these deviations from the law. The unit-cell volumes increase linearly whereas the densities of the phases decrease linearly as the radius of the RE cations increases (Fig. 2).

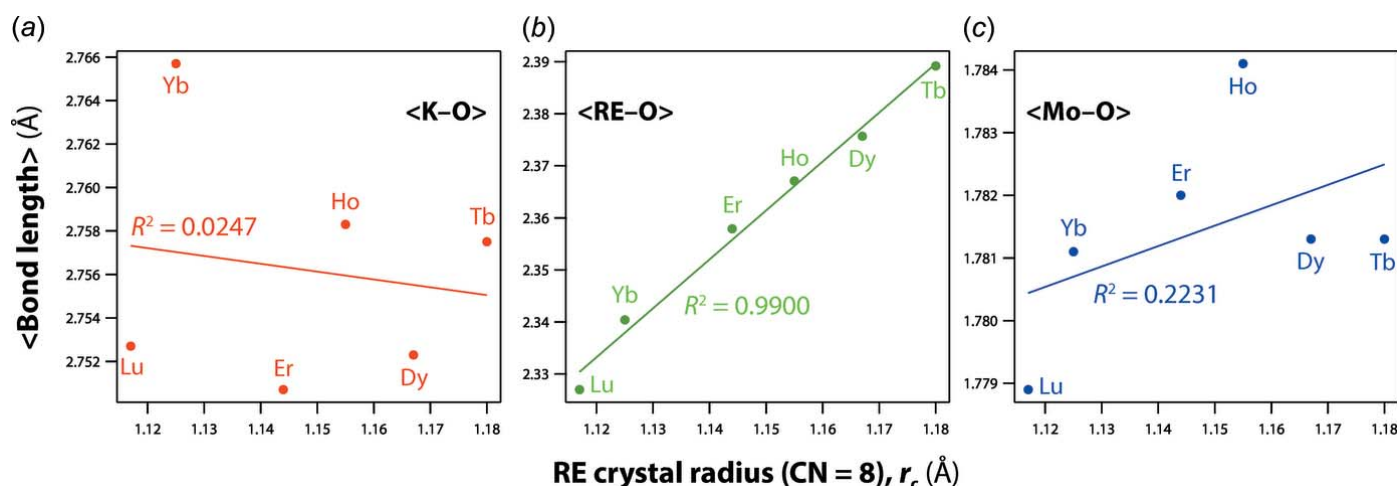
### 3. Synthesis and crystallization

The single crystals of  $KRE(\text{MoO}_4)_2$  were synthesized using Tb<sub>4</sub>O<sub>7</sub> (Alfa Aesar, 99.9%), Dy<sub>2</sub>O<sub>3</sub> (Alfa Aesar, 99.9%),

Ho<sub>2</sub>O<sub>3</sub> (Alfa Aesar, 99.9%), Er<sub>2</sub>O<sub>3</sub> (Alfa Aesar, 99.9%), Yb<sub>2</sub>O<sub>3</sub> (Alfa Aesar, 99.9%), Lu<sub>2</sub>O<sub>3</sub> (Alfa Aesar, 99.9%), K<sub>2</sub>CO<sub>3</sub> (Alfa Aesar, 99%), and MoO<sub>3</sub> (Alfa Aesar, 99.5%). All the chemicals were used as received. First, K<sub>2</sub>Mo<sub>3</sub>O<sub>10</sub> was synthesized using K<sub>2</sub>CO<sub>3</sub> and MoO<sub>3</sub> by heating at 520°C for 8 h as described in a previous study (Chong *et al.*, 2020). The stoichiometric mixture of REO<sub>x</sub> and K<sub>2</sub>Mo<sub>3</sub>O<sub>10</sub> was put into a Pt/10%Rh crucible with a lid and placed in a Thermolyne box furnace. The furnace was heated to 1150°C at 5°C min<sup>-1</sup>, dwelled for 10 h, cooled to 400°C at 5°C h<sup>-1</sup> in air, and then shut off. The single crystals were recovered from the solidified flux after washing in an ultrasonic bath with deionized water and using vacuum filtration. In addition to the listed six crystals, KTm(MoO<sub>4</sub>)<sub>2</sub> was synthesized similarly, but it was not reported in this study due to the unresolved residual electron densities during structural refinement. All the  $KRE(\text{MoO}_4)_2$  crystals were plates (Fig. 4) with different colors (Fig. 5).

### 4. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. A hemisphere of data was collected on two crystals (RE = Ho, Yb) using a Bruker APEXII Quazar diffractometer equipped with a microsource tube emitting monochromated Mo K $\alpha$  X-ray radiation and collected on a CCD detector. Data were collected for five


**Figure 3**

 Average distances of (a)  $\langle \text{K}-\text{O} \rangle$ , (b)  $\langle \text{RE}-\text{O} \rangle$ , and (c)  $\langle \text{Mo}-\text{O} \rangle$  of  $KRE(\text{MoO}_4)_2$  compounds.

**Table 2**  
Experimental details.

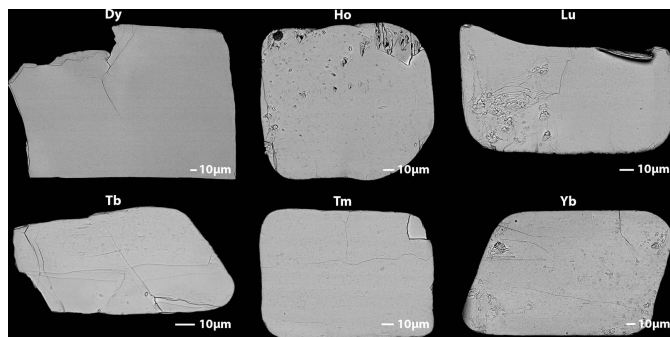
	KTb(MoO <sub>4</sub> ) <sub>2</sub>	KDy(MoO <sub>4</sub> ) <sub>2</sub>	KHo(MoO <sub>4</sub> ) <sub>2</sub>
<b>Crystal data</b>			
$M_r$	517.90	521.48	523.91
Crystal system, space group	Orthorhombic, <i>Pbcn</i>	Orthorhombic, <i>Pbcn</i>	Orthorhombic, <i>Pbcn</i>
Temperature (K)	100	100	273
$a, b, c$ (Å)	5.0826 (1), 18.1273 (7), 7.9875 (2)	5.0776 (2), 18.1214 (7), 7.9428 (3)	5.0770 (15), 18.161 (5), 7.934 (2)
$V$ (Å <sup>3</sup> )	735.92 (4)	730.84 (5)	731.5 (4)
$Z$	4	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	13.43	14.07	14.66
Crystal size (mm)	0.36 × 0.20 × 0.04	0.11 × 0.11 × 0.03	0.06 × 0.06 × 0.04
<b>Data collection</b>			
Diffractometer	Rigaku XtaLAB Synergy-S, HyPix	Rigaku XtaLAB Synergy-S, HyPix	Bruker APEXII CCD
Absorption correction	Gaussian ( <i>CrysAlis PRO</i> ; Rigaku OD, 2019)	Gaussian ( <i>CrysAlis PRO</i> ; Rigaku OD, 2019)	Multi-scan ( <i>SADABS</i> ; Krause et al., 2015)
$T_{\min}$ , $T_{\max}$	0.035, 0.703	0.247, 0.695	0.248, 0.343
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	13792, 1350, 1238	17317, 1388, 1196	7665, 936, 818
$R_{\text{int}}$	0.069	0.075	0.032
<b>Refinement</b>			
$R[F^2 > 2\sigma(F^2)]$ , $wR(F^2)$ , $S$	0.033, 0.102, 1.08	0.033, 0.088, 1.10	0.027, 0.063, 1.25
No. of reflections	1350	1388	936
No. of parameters	56	56	56
$\Delta\rho_{\text{max}}$ , $\Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	2.63, -2.78	2.38, -2.65	1.51, -1.37
<hr/>			
	KEr(MoO <sub>4</sub> ) <sub>2</sub>	KYb(MoO <sub>4</sub> ) <sub>2</sub>	KLu(MoO <sub>4</sub> ) <sub>2</sub>
<b>Crystal data</b>			
$M_r$	526.24	532.02	533.95
Crystal system, space group	Orthorhombic, <i>Pbcn</i>	Orthorhombic, <i>Pbcn</i>	Orthorhombic, <i>Pbcn</i>
Temperature (K)	100	273	100
$a, b, c$ (Å)	5.0602 (2), 18.1965 (8), 7.8920 (3)	5.0417 (5), 18.3039 (19), 7.8693 (8)	5.0292 (2), 18.2519 (10), 7.8174 (4)
$V$ (Å <sup>3</sup> )	726.68 (5)	726.20 (13)	717.58 (6)
$Z$	4	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	15.42	16.75	17.68
Crystal size (mm)	0.28 × 0.15 × 0.02	0.06 × 0.06 × 0.04	0.44 × 0.15 × 0.04
<b>Data collection</b>			
Diffractometer	Rigaku XtaLAB Synergy-S, HyPix	Bruker APEXII CCD	Rigaku XtaLAB Synergy-S, HyPix
Absorption correction	Gaussian ( <i>CrysAlis PRO</i> ; Rigaku OD, 2019)	Multi-scan ( <i>SADABS</i> ; Krause et al., 2015)	Multi-scan ( <i>CrysAlis PRO</i> ; Rigaku OD, 2019)
$T_{\min}$ , $T_{\max}$	0.091, 0.911	0.198, 0.301	0.240, 1.000
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	21246, 1386, 1255	8164, 978, 845	19857, 1105, 1004
$R_{\text{int}}$	0.072	0.038	0.087
<b>Refinement</b>			
$R[F^2 > 2\sigma(F^2)]$ , $wR(F^2)$ , $S$	0.027, 0.073, 1.06	0.022, 0.063, 1.11	0.027, 0.077, 1.17
No. of reflections	1386	978	1105
No. of parameters	56	56	57
$\Delta\rho_{\text{max}}$ , $\Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	2.04, -1.62	1.55, -1.14	2.05, -3.03

Computer programs: *CrysAlis PRO* (Rigaku OD, 2019), *APEX3* (Bruker, 2014), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b), *OLEX2* (Dolomanov et al., 2009), and *pubCIF* (Westrip, 2010).

crystals ( $RE = \text{Dy, Er, Lu, Tb, Tm}$ ) with a Rigaku XtaLab Synergy diffractometer using a single microfocus Mo  $K\alpha$  X-ray radiation source in a sealed tube, equipped with a Hybrid Pixel (HyPix) Array detector and using an Oxford liquid-nitrogen Cryostream. For the Bruker datasets, *APEX3* software (Bruker, 2014) was used for determining the unit cell and integrating the collected reflection data. Absorption corrections were applied with the *SADABS* software package

(Krause et al., 2015). For the Rigaku datasets, the *CrysAlis Pro* software package was used for unit-cell determination and data integration (Rigaku OD, 2019). The numerical absorption correction was applied utilizing *SCALE3 ABSPACK* (Clark & Reid, 1995). All structures were solved by the intrinsic phasing method using *SHELXT* and refined with *SHELXL* (Sheldrick, 2015a,b) within the *OLEX2* software package (Dolomanov et al., 2009).




**Figure 4**

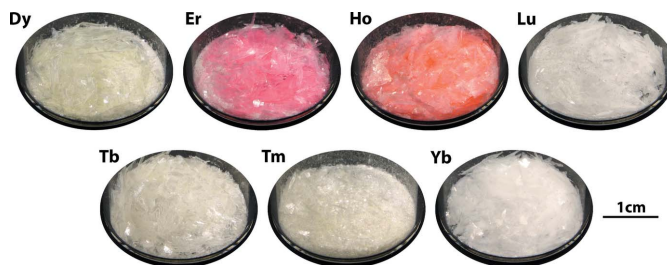
SEM micrographs of  $KRE(\text{MoO}_4)_2$ . Artifacts on the surface of several crystals shown resemble residual flux not fully removed during rinsing.

### Acknowledgements

The Pacific Northwest National Laboratory is operated by Battelle under Contract Number DE-AC05-76RL01830.

### References

- Axe, J., Dorner, B. & Shirane, G. (1971). *Phys. Rev. Lett.* **26**, 519–523.
- Borchardt, H. J. & Bierstedt, P. E. (1967). *J. Appl. Phys.* **38**, 2057–2060.
- Brixner, L. H., Barkley, J. R. & Jeitschko, W. (1979). *Handbook on the Physics and Chemistry of Rare Earths*, Vol. 3, pp. 609–654. Berlin, Heidelberg, New York: Springer.
- Bruker (2014). *APEX3*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chong, S., Riley, B. J., Nelson, Z. J. & Perry, S. N. (2020). *Acta Cryst.* **E76**, 339–343.
- Clark, R. C. & Reid, J. S. (1995). *Acta Cryst.* **A51**, 887–897.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Guzmán-Afonso, C., González-Silgo, C., Torres, M., Matesanz, E. & Mujica, A. (2013). *J. Phys. Condens. Matter*, **25**, 035902–1–03590, 2–10.
- Hanuza, J. & Fomitsev, V. V. (1980). *J. Mol. Struct.* **66**, 1–24.
- Hanuza, J., Macalik, L. & Hermanowicz, K. (1994). *J. Mol. Struct.* **319**, 17–30.
- Jeitschko, W. (1973). *Acta Cryst.* **B29**, 2074–2081.
- Kamenskyi, D., Poperezhai, S., Gogoi, P., Engelkamp, H., Maan, J., Wosnitza, J. & Kut'ko, V. (2014). *Phys. Rev. B*, **89**, 014410–1–01441.
- Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.


**Figure 5**

Pictures of recovered crystals of  $KRE(\text{MoO}_4)_2$  showing various sizes and colors.

- Kut'ko, V. I. (2005). *Low Temp. Phys.* **31**, 1–31.
- Leask, M. J. M., Tropper, A. C. & Wells, M. R. (1981). *J. Phys. C.: Solid State Phys.* **14**, 3481–3498.
- Mat'aš, S., Dudzik, E., Feyerherm, R., Gerischer, S., Klemke, S., Prokeš, K. & Orendáčová, A. (2010). *Phys. Rev. B*, **82**, 184427–1–18442, 7–8.
- Ponomarev, B. K., Ivanov, S. A., Popov, Y. F., Negrii, V. D. & Red'Kin, B. S. (1994). *Ferroelectrics*, **161**, 43–48.
- Ponomarev, B. K. & Zhukov, A. (2012). *Phys. Res. Int.* **2012**, 276348–1–27634, 8–22.
- Poperezhai, S., Gogoi, P., Zubenko, N., Kutko, K., Kutko, V. I., Kovalev, A. S. & Kamenskyi, D. (2017). *J. Phys. Condens. Matter*, **29**, 095402–1–09540, 2–6.
- Pratap, V., Gaur, K. & Lal, H. B. (1987). *Mater. Res. Bull.* **22**, 1381–1393.
- Rigaku OD (2019). *CrysAlis PRO*. Rigaku Oxford Diffraction, Yarnton, England.
- Shannon, R. D. (1976). *Acta Cryst.* **A32**, 751–767.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Shi, F., Meng, J. & Ren, Y. (1996). *J. Solid State Chem.* **121**, 236–239.
- Stedman, N. J., Cheetham, A. K. & Battle, P. D. (1994). *J. Mater. Chem.* **4**, 707–711.
- Voron'ko, Y. K., Zharikov, E. V., Lis, D., Sobol, A. A., Subbotin, K. A., Ushakov, S. N. & Shukshin, V. E. (2004). *Laser Optics 2003: Solid State Lasers and Nonlinear Frequency Conversion*, pp. 60–68. International Society for Optics and Photonics.
- Wang, Z., Liang, H., Gong, M. & Su, Q. (2007). *J. Alloys Compd.* **432**, 308–312.
- Wanklyn, B. M. & Wondre, F. R. (1978). *J. Cryst. Growth*, **43**, 93–100.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

## supporting information

*Acta Cryst.* (2020). E76, 1871-1875 [https://doi.org/10.1107/S205698902001542X]

## Crystal structures and comparisons of potassium rare-earth molybdates $KRE(\text{MoO}_4)_2$ ( $RE = \text{Tb, Dy, Ho, Er, Yb, and Lu}$ )

Saehwa Chong, Samuel Perry, Brian J. Riley and Zayne J. Nelson

### Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2019) for Tb\_Molybdate, Dy\_Molybdate, Er\_Molybdate, Lu\_Molybdate; *APEX3* (Bruker, 2014) for Ho\_Molybdate, Yb\_Molybdate. Cell refinement: *CrysAlis PRO* (Rigaku OD, 2019) for Tb\_Molybdate, Dy\_Molybdate, Er\_Molybdate, Lu\_Molybdate; *APEX3* (Bruker, 2014) for Ho\_Molybdate, Yb\_Molybdate. Data reduction: *CrysAlis PRO* (Rigaku OD, 2019) for Tb\_Molybdate, Dy\_Molybdate, Er\_Molybdate, Lu\_Molybdate; *APEX3* (Bruker, 2014) for Ho\_Molybdate, Yb\_Molybdate. For all structures, program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010).

### Potassium terbium bis(molybdate) (Tb\_Molybdate)

#### Crystal data

$\text{KTb}(\text{MoO}_4)_2$	$F(000) = 928$
$M_r = 517.90$	$D_x = 4.674 \text{ Mg m}^{-3}$
Orthorhombic, <i>Pbcn</i>	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2n 2ab	Cell parameters from 6923 reflections
$a = 5.0826 (1) \text{ \AA}$	$\theta = 2.3\text{--}33.5^\circ$
$b = 18.1273 (7) \text{ \AA}$	$\mu = 13.43 \text{ mm}^{-1}$
$c = 7.9875 (2) \text{ \AA}$	$T = 100 \text{ K}$
$V = 735.92 (4) \text{ \AA}^3$	Plate, white
$Z = 4$	$0.36 \times 0.20 \times 0.04 \text{ mm}$

#### Data collection

Rigaku XtaLAB Synergy-S, HyPix diffractometer	13792 measured reflections
Radiation source: micro-focus sealed X-ray tube	1350 independent reflections
Mirror monochromator	1238 reflections with $I > 2\sigma(I)$
Detector resolution: $10.0000 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.069$
$\omega$ scans	$\theta_{\text{max}} = 33.7^\circ$ , $\theta_{\text{min}} = 2.3^\circ$
Absorption correction: gaussian	$h = -7 \rightarrow 6$
(CrysAlisPro; Rigaku OD, 2019)	$k = -27 \rightarrow 27$
$T_{\text{min}} = 0.035$ , $T_{\text{max}} = 0.703$	$l = -12 \rightarrow 12$

#### Refinement

Refinement on $F^2$	1350 reflections
Least-squares matrix: full	56 parameters
$R[F^2 > 2\sigma(F^2)] = 0.033$	0 restraints
$wR(F^2) = 0.102$	0 constraints
$S = 1.08$	Primary atom site location: dual

$$w = 1/[\sigma^2(F_o^2) + (0.0705P)^2 + 1.3389P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.002$

$$\Delta\rho_{\max} = 2.63 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -2.78 \text{ e } \text{\AA}^{-3}$$

*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.** Reflections were merged by SHELXL according to the crystal class for the calculation of statistics and refinement.

*\_reflns\_Friedel\_fraction* is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Tb1	0.000000	0.49384 (2)	0.250000	0.00653 (12)
Mo1	0.48012 (7)	0.60289 (2)	0.48337 (5)	0.00701 (12)
K1	0.500000	0.77012 (8)	0.750000	0.0114 (2)
O2	0.7489 (5)	0.53496 (13)	0.5042 (2)	0.0089 (4)
O4	0.6111 (5)	0.69028 (13)	0.4759 (3)	0.0105 (5)
O1	0.2592 (5)	0.59503 (12)	0.3093 (3)	0.0099 (4)
O3	0.2716 (5)	0.60251 (12)	0.6603 (3)	0.0102 (4)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Tb1	0.00409 (18)	0.01076 (18)	0.00476 (17)	0.000	-0.00033 (5)	0.000
Mo1	0.00425 (19)	0.01069 (19)	0.00610 (19)	0.00024 (8)	-0.00066 (7)	-0.00020 (10)
K1	0.0118 (5)	0.0136 (5)	0.0089 (5)	0.000	-0.0008 (3)	0.000
O2	0.0056 (12)	0.0138 (9)	0.0073 (9)	0.0017 (9)	-0.0008 (6)	0.0002 (7)
O4	0.0096 (12)	0.0130 (10)	0.0089 (10)	-0.0036 (9)	0.0011 (7)	-0.0007 (8)
O1	0.0069 (10)	0.0143 (10)	0.0084 (10)	-0.0001 (8)	-0.0021 (8)	0.0002 (8)
O3	0.0089 (10)	0.0144 (10)	0.0073 (9)	-0.0003 (8)	0.0003 (8)	-0.0008 (7)

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

Tb1—Tb1 <sup>i</sup>	4.0000 (1)	Mo1—O3	1.767 (2)
Tb1—Tb1 <sup>ii</sup>	4.0000 (1)	K1—Mo1 <sup>xi</sup>	3.8378 (9)
Tb1—K1 <sup>iii</sup>	4.2788 (14)	K1—Mo1 <sup>xii</sup>	3.7062 (11)
Tb1—O2 <sup>iv</sup>	2.399 (2)	K1—Mo1 <sup>xiii</sup>	3.8378 (9)
Tb1—O2 <sup>v</sup>	2.399 (2)	K1—O4	2.684 (2)
Tb1—O2 <sup>vi</sup>	2.511 (2)	K1—O4 <sup>xiv</sup>	2.771 (2)
Tb1—O2 <sup>vii</sup>	2.511 (2)	K1—O4 <sup>xii</sup>	2.684 (2)
Tb1—O1 <sup>viii</sup>	2.308 (2)	K1—O4 <sup>xv</sup>	2.771 (2)
Tb1—O1	2.308 (2)	K1—O1 <sup>xiii</sup>	2.817 (3)
Tb1—O3 <sup>ix</sup>	2.339 (2)	K1—O1 <sup>xi</sup>	2.817 (3)
Tb1—O3 <sup>ii</sup>	2.339 (2)	K1—O3	3.330 (3)

Mo1—K1	3.7062 (11)	K1—O3 <sup>xii</sup>	3.330 (3)
Mo1—K1 <sup>x</sup>	3.9694 (9)	O2—Tb1 <sup>iv</sup>	2.399 (2)
Mo1—K1 <sup>iii</sup>	3.8378 (9)	O2—Tb1 <sup>xvi</sup>	2.511 (2)
Mo1—O2	1.847 (2)	O4—K1 <sup>x</sup>	2.771 (2)
Mo1—O4	1.719 (2)	O1—K1 <sup>iii</sup>	2.817 (3)
Mo1—O1	1.793 (2)	O3—Tb1 <sup>ii</sup>	2.339 (2)
Tb1 <sup>ii</sup> —Tb1—Tb1 <sup>i</sup>	173.598 (18)	Mo1 <sup>xiii</sup> —K1—Mo1 <sup>xi</sup>	106.29 (4)
Tb1 <sup>ii</sup> —Tb1—K1 <sup>iii</sup>	86.799 (9)	Mo1—K1—Mo1 <sup>xiii</sup>	103.230 (11)
Tb1 <sup>i</sup> —Tb1—K1 <sup>iii</sup>	86.799 (9)	Mo1 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	138.798 (13)
O2 <sup>iv</sup> —Tb1—Tb1 <sup>i</sup>	146.02 (5)	Mo1—K1—Mo1 <sup>xii</sup>	70.24 (3)
O2 <sup>v</sup> —Tb1—Tb1 <sup>i</sup>	36.40 (5)	Mo1 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	103.230 (11)
O2 <sup>vi</sup> —Tb1—Tb1 <sup>i</sup>	34.54 (5)	O4 <sup>xii</sup> —K1—Mo1	91.25 (6)
O2 <sup>vii</sup> —Tb1—Tb1 <sup>i</sup>	142.24 (5)	O4—K1—Mo1 <sup>xii</sup>	91.25 (6)
O2 <sup>iv</sup> —Tb1—Tb1 <sup>ii</sup>	36.40 (5)	O4 <sup>xiv</sup> —K1—Mo1 <sup>xi</sup>	72.98 (5)
O2 <sup>v</sup> —Tb1—Tb1 <sup>ii</sup>	146.02 (5)	O4 <sup>xiv</sup> —K1—Mo1 <sup>xii</sup>	127.27 (6)
O2 <sup>vi</sup> —Tb1—Tb1 <sup>ii</sup>	142.24 (5)	O4 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	78.09 (5)
O2 <sup>vii</sup> —Tb1—Tb1 <sup>ii</sup>	34.54 (5)	O4—K1—Mo1	25.32 (5)
O2 <sup>vi</sup> —Tb1—K1 <sup>iii</sup>	72.73 (5)	O4 <sup>xiv</sup> —K1—Mo1	79.53 (5)
O2 <sup>vii</sup> —Tb1—K1 <sup>iii</sup>	72.73 (5)	O4—K1—Mo1 <sup>xiii</sup>	78.09 (5)
O2 <sup>v</sup> —Tb1—K1 <sup>iii</sup>	102.57 (6)	O4—K1—Mo1 <sup>xi</sup>	148.56 (6)
O2 <sup>iv</sup> —Tb1—K1 <sup>iii</sup>	102.57 (6)	O4 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	148.57 (6)
O2 <sup>vii</sup> —Tb1—O2 <sup>vi</sup>	145.47 (11)	O4 <sup>xv</sup> —K1—Mo1 <sup>xi</sup>	88.96 (6)
O2 <sup>iv</sup> —Tb1—O2 <sup>vi</sup>	117.12 (10)	O4 <sup>xv</sup> —K1—Mo1 <sup>xiii</sup>	72.98 (5)
O2 <sup>iv</sup> —Tb1—O2 <sup>vii</sup>	70.93 (9)	O4 <sup>xii</sup> —K1—Mo1 <sup>xii</sup>	25.32 (5)
O2 <sup>v</sup> —Tb1—O2 <sup>vi</sup>	70.93 (9)	O4 <sup>xiv</sup> —K1—Mo1 <sup>xiii</sup>	88.96 (6)
O2 <sup>v</sup> —Tb1—O2 <sup>vii</sup>	117.12 (10)	O4 <sup>xv</sup> —K1—Mo1	127.27 (6)
O2 <sup>v</sup> —Tb1—O2 <sup>iv</sup>	154.87 (11)	O4 <sup>xv</sup> —K1—Mo1 <sup>xii</sup>	79.53 (5)
O1—Tb1—Tb1 <sup>i</sup>	99.23 (6)	O4 <sup>xii</sup> —K1—O4 <sup>xv</sup>	76.03 (6)
O1 <sup>viii</sup> —Tb1—Tb1 <sup>i</sup>	75.57 (6)	O4—K1—O4 <sup>xv</sup>	121.38 (3)
O1—Tb1—Tb1 <sup>ii</sup>	75.57 (6)	O4 <sup>xv</sup> —K1—O4 <sup>xiv</sup>	149.97 (11)
O1 <sup>viii</sup> —Tb1—Tb1 <sup>ii</sup>	99.23 (6)	O4—K1—O4 <sup>xiv</sup>	76.03 (6)
O1 <sup>viii</sup> —Tb1—K1 <sup>iii</sup>	37.35 (6)	O4 <sup>xii</sup> —K1—O4 <sup>xiv</sup>	121.38 (3)
O1—Tb1—K1 <sup>iii</sup>	37.35 (6)	O4—K1—O4 <sup>xii</sup>	114.75 (11)
O1 <sup>viii</sup> —Tb1—O2 <sup>iv</sup>	130.14 (8)	O4 <sup>xii</sup> —K1—O1 <sup>xi</sup>	103.44 (7)
O1—Tb1—O2 <sup>vi</sup>	68.88 (7)	O4—K1—O1 <sup>xi</sup>	134.69 (7)
O1 <sup>viii</sup> —Tb1—O2 <sup>vii</sup>	68.88 (7)	O4—K1—O1 <sup>xiii</sup>	103.44 (7)
O1 <sup>viii</sup> —Tb1—O2 <sup>vi</sup>	83.59 (8)	O4 <sup>xiv</sup> —K1—O1 <sup>xiii</sup>	89.97 (8)
O1—Tb1—O2 <sup>iv</sup>	72.62 (8)	O4 <sup>xiv</sup> —K1—O1 <sup>xi</sup>	63.32 (7)
O1—Tb1—O2 <sup>vii</sup>	83.59 (8)	O4 <sup>xii</sup> —K1—O1 <sup>xiii</sup>	134.69 (7)
O1 <sup>viii</sup> —Tb1—O2 <sup>v</sup>	72.62 (8)	O4 <sup>xv</sup> —K1—O1 <sup>xiii</sup>	63.32 (7)
O1—Tb1—O2 <sup>v</sup>	130.14 (8)	O4 <sup>xv</sup> —K1—O1 <sup>xi</sup>	89.97 (8)
O1—Tb1—O1 <sup>viii</sup>	74.71 (11)	O4 <sup>xv</sup> —K1—O3	128.69 (7)
O1 <sup>viii</sup> —Tb1—O3 <sup>ix</sup>	150.21 (8)	O4 <sup>xv</sup> —K1—O3 <sup>xii</sup>	81.24 (7)
O1 <sup>viii</sup> —Tb1—O3 <sup>ii</sup>	108.63 (9)	O4 <sup>xii</sup> —K1—O3	67.06 (7)
O1—Tb1—O3 <sup>ix</sup>	108.63 (9)	O4 <sup>xiv</sup> —K1—O3	81.24 (7)
O1—Tb1—O3 <sup>ii</sup>	150.21 (8)	O4 <sup>xiv</sup> —K1—O3 <sup>xii</sup>	128.69 (7)
O3 <sup>ix</sup> —Tb1—Tb1 <sup>i</sup>	74.69 (5)	O4—K1—O3 <sup>xii</sup>	67.06 (7)



O3 <sup>ix</sup> —Tb1—Tb1 <sup>ii</sup>	110.34 (5)	O4—K1—O3	53.56 (7)
O3 <sup>ii</sup> —Tb1—Tb1 <sup>ii</sup>	74.69 (5)	O4 <sup>xii</sup> —K1—O3 <sup>xii</sup>	53.56 (7)
O3 <sup>ii</sup> —Tb1—Tb1 <sup>i</sup>	110.34 (5)	O1 <sup>xiii</sup> —K1—Mo1	128.75 (5)
O3 <sup>ii</sup> —Tb1—K1 <sup>iii</sup>	138.32 (6)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xi</sup>	81.87 (6)
O3 <sup>ix</sup> —Tb1—K1 <sup>iii</sup>	138.32 (6)	O1 <sup>xi</sup> —K1—Mo1 <sup>xii</sup>	128.75 (5)
O3 <sup>ix</sup> —Tb1—O2 <sup>vii</sup>	140.29 (7)	O1 <sup>xi</sup> —K1—Mo1	142.52 (5)
O3 <sup>ix</sup> —Tb1—O2 <sup>iv</sup>	76.95 (8)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xii</sup>	142.52 (5)
O3 <sup>ii</sup> —Tb1—O2 <sup>vii</sup>	70.98 (8)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xiii</sup>	25.90 (5)
O3 <sup>ii</sup> —Tb1—O2 <sup>v</sup>	76.95 (8)	O1 <sup>xi</sup> —K1—Mo1 <sup>xiii</sup>	81.87 (6)
O3 <sup>ix</sup> —Tb1—O2 <sup>vi</sup>	70.98 (8)	O1 <sup>xi</sup> —K1—Mo1 <sup>xi</sup>	25.90 (5)
O3 <sup>ii</sup> —Tb1—O2 <sup>vi</sup>	140.29 (7)	O1 <sup>xiii</sup> —K1—O1 <sup>xi</sup>	59.61 (10)
O3 <sup>ii</sup> —Tb1—O2 <sup>iv</sup>	84.31 (8)	O1 <sup>xiii</sup> —K1—O3 <sup>xii</sup>	131.66 (6)
O3 <sup>ix</sup> —Tb1—O2 <sup>v</sup>	84.31 (8)	O1 <sup>xi</sup> —K1—O3	131.66 (6)
O3 <sup>ii</sup> —Tb1—O3 <sup>ix</sup>	83.37 (11)	O1 <sup>xi</sup> —K1—O3 <sup>xii</sup>	156.71 (6)
K1—Mo1—K1 <sup>x</sup>	77.135 (10)	O1 <sup>xiii</sup> —K1—O3	156.71 (6)
K1 <sup>iii</sup> —Mo1—K1 <sup>x</sup>	81.22 (2)	O3—K1—Mo1 <sup>xiii</sup>	131.63 (4)
K1—Mo1—K1 <sup>iii</sup>	78.802 (10)	O3—K1—Mo1	28.44 (4)
O2—Mo1—K1 <sup>iii</sup>	156.08 (6)	O3 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	131.63 (4)
O2—Mo1—K1 <sup>x</sup>	86.36 (7)	O3 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	115.47 (4)
O2—Mo1—K1	118.26 (7)	O3 <sup>xii</sup> —K1—Mo1	52.19 (4)
O4—Mo1—K1 <sup>iii</sup>	71.14 (9)	O3—K1—Mo1 <sup>xi</sup>	115.47 (4)
O4—Mo1—K1 <sup>x</sup>	36.07 (8)	O3 <sup>xii</sup> —K1—Mo1 <sup>xii</sup>	28.44 (4)
O4—Mo1—K1	41.90 (7)	O3—K1—Mo1 <sup>xii</sup>	52.19 (4)
O4—Mo1—O2	109.34 (11)	O3—K1—O3 <sup>xii</sup>	48.36 (8)
O4—Mo1—O1	106.78 (11)	Tb1 <sup>iv</sup> —O2—Tb1 <sup>xvi</sup>	109.07 (9)
O4—Mo1—O3	105.28 (10)	Mo1—O2—Tb1 <sup>iv</sup>	127.76 (10)
O1—Mo1—K1	121.85 (7)	Mo1—O2—Tb1 <sup>xvi</sup>	120.07 (9)
O1—Mo1—K1 <sup>iii</sup>	43.35 (7)	Mo1—O4—K1	112.78 (11)
O1—Mo1—K1 <sup>x</sup>	95.66 (7)	Mo1—O4—K1 <sup>x</sup>	122.51 (11)
O1—Mo1—O2	118.69 (10)	K1—O4—K1 <sup>x</sup>	122.78 (9)
O3—Mo1—K1	63.88 (7)	Tb1—O1—K1 <sup>iii</sup>	112.84 (9)
O3—Mo1—K1 <sup>iii</sup>	90.54 (8)	Mo1—O1—Tb1	125.44 (11)
O3—Mo1—K1 <sup>x</sup>	141.01 (7)	Mo1—O1—K1 <sup>iii</sup>	110.75 (10)
O3—Mo1—O2	111.67 (10)	Tb1 <sup>ii</sup> —O3—K1	145.20 (9)
O3—Mo1—O1	104.16 (12)	Mo1—O3—Tb1 <sup>ii</sup>	127.02 (11)
Mo1—K1—Mo1 <sup>xi</sup>	138.799 (13)	Mo1—O3—K1	87.68 (8)

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

### Potassium dysprosium bis(molybdate) (Dy\_Molybdate)

#### Crystal data

KDy(MoO<sub>4</sub>)<sub>2</sub>

$M_r = 521.48$

Orthorhombic, *Pbcn*

Hall symbol:  $-P\ 2n\ 2ab$

$a = 5.0776$  (2) Å

$b = 18.1214$  (7) Å

$c = 7.9428$  (3) Å

$V = 730.84$  (5) Å<sup>3</sup>

$Z = 4$

$F(000) = 932$

$D_x = 4.739$  Mg m<sup>-3</sup>

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

Cell parameters from 5121 reflections  
 $\theta = 2.3\text{--}33.3^\circ$   
 $\mu = 14.07 \text{ mm}^{-1}$

$T = 100 \text{ K}$   
 Plate, light yellow  
 $0.11 \times 0.11 \times 0.03 \text{ mm}$

*Data collection*

Rigaku XtaLAB Synergy-S, HyPix  
 diffractometer  
 Radiation source: micro-focus sealed X-ray tube  
 Mirror monochromator  
 Detector resolution:  $10.0000 \text{ pixels mm}^{-1}$   
 $\omega$  scans  
 Absorption correction: gaussian  
 (CrysAlisPro; Rigaku OD, 2019)  
 $T_{\text{min}} = 0.247, T_{\text{max}} = 0.695$

17317 measured reflections  
 1388 independent reflections  
 1196 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.075$   
 $\theta_{\text{max}} = 34.0^\circ, \theta_{\text{min}} = 2.3^\circ$   
 $h = -7 \rightarrow 7$   
 $k = -27 \rightarrow 27$   
 $l = -11 \rightarrow 11$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.033$   
 $wR(F^2) = 0.088$   
 $S = 1.10$   
 1388 reflections  
 56 parameters  
 0 restraints

0 constraints  
 Primary atom site location: dual  
 $w = 1/[\sigma^2(F_o^2) + (0.0343P)^2 + 8.538P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 2.38 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\text{min}} = -2.65 \text{ e } \text{Å}^{-3}$

*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.** Reflections were merged by SHELXL according to the crystal class for the calculation of statistics and refinement.

$\_reflns\_Friedel\_fraction$  is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Dy1	0.000000	0.49408 (2)	0.250000	0.00749 (11)
Mo1	0.48037 (7)	0.60238 (2)	0.48347 (5)	0.00769 (11)
K1	0.500000	0.77076 (9)	0.750000	0.0118 (3)
O4	0.6101 (7)	0.6900 (2)	0.4770 (4)	0.0121 (6)
O2	0.7489 (7)	0.53449 (19)	0.5049 (4)	0.0100 (6)
O1	0.2588 (6)	0.59449 (19)	0.3083 (4)	0.0111 (6)
O3	0.2709 (6)	0.60172 (19)	0.6610 (4)	0.0109 (6)

*Atomic displacement parameters ( $\text{Å}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Dy1	0.00510 (15)	0.01210 (17)	0.00526 (16)	0.000	-0.00001 (8)	0.000
Mo1	0.00512 (18)	0.0115 (2)	0.00648 (18)	0.00032 (12)	-0.00022 (10)	-0.00023 (12)
K1	0.0125 (6)	0.0147 (6)	0.0083 (6)	0.000	-0.0006 (4)	0.000

O4	0.0098 (15)	0.0154 (16)	0.0109 (15)	-0.0020 (12)	0.0006 (11)	-0.0004 (12)
O2	0.0066 (13)	0.0161 (16)	0.0072 (14)	0.0018 (11)	-0.0009 (11)	-0.0015 (11)
O1	0.0081 (13)	0.0178 (16)	0.0074 (14)	-0.0013 (12)	-0.0010 (11)	0.0001 (12)
O3	0.0073 (13)	0.0171 (16)	0.0082 (14)	-0.0003 (12)	0.0008 (11)	0.0006 (11)

*Geometric parameters (Å, °)*

Dy1—Dy1 <sup>i</sup>	3.9772 (1)	Mo1—O3	1.766 (3)
Dy1—Dy1 <sup>ii</sup>	3.9772 (2)	K1—Mo1 <sup>xi</sup>	3.8305 (10)
Dy1—K1 <sup>iii</sup>	4.2614 (16)	K1—Mo1 <sup>xii</sup>	3.7150 (13)
Dy1—O2 <sup>iv</sup>	2.384 (3)	K1—Mo1 <sup>xiii</sup>	3.8305 (10)
Dy1—O2 <sup>v</sup>	2.384 (3)	K1—O4	2.676 (4)
Dy1—O2 <sup>vi</sup>	2.502 (3)	K1—O4 <sup>xiv</sup>	2.770 (3)
Dy1—O2 <sup>vii</sup>	2.502 (3)	K1—O4 <sup>xii</sup>	2.676 (4)
Dy1—O1	2.292 (3)	K1—O4 <sup>xv</sup>	2.770 (3)
Dy1—O1 <sup>viii</sup>	2.292 (3)	K1—O1 <sup>xiii</sup>	2.812 (4)
Dy1—O3 <sup>ix</sup>	2.325 (3)	K1—O1 <sup>xi</sup>	2.812 (4)
Dy1—O3 <sup>ii</sup>	2.325 (3)	K1—O3 <sup>xii</sup>	3.352 (4)
Mo1—K1	3.7150 (13)	K1—O3	3.352 (4)
Mo1—K1 <sup>x</sup>	3.9604 (10)	O4—K1 <sup>x</sup>	2.770 (3)
Mo1—K1 <sup>iii</sup>	3.8305 (10)	O2—Dy1 <sup>xvi</sup>	2.502 (3)
Mo1—O4	1.720 (4)	O2—Dy1 <sup>iv</sup>	2.384 (3)
Mo1—O2	1.845 (3)	O1—K1 <sup>iii</sup>	2.812 (4)
Mo1—O1	1.795 (3)	O3—Dy1 <sup>ii</sup>	2.325 (3)
Dy1 <sup>ii</sup> —Dy1—Dy1 <sup>i</sup>	173.818 (18)	Mo1—K1—Mo1 <sup>xii</sup>	69.57 (3)
Dy1 <sup>ii</sup> —Dy1—K1 <sup>iii</sup>	86.909 (9)	Mo1—K1—Mo1 <sup>xi</sup>	138.737 (15)
Dy1 <sup>i</sup> —Dy1—K1 <sup>iii</sup>	86.909 (9)	Mo1 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	138.737 (15)
O2 <sup>iv</sup> —Dy1—Dy1 <sup>i</sup>	145.80 (8)	Mo1 <sup>xiii</sup> —K1—Mo1 <sup>xi</sup>	106.24 (4)
O2 <sup>v</sup> —Dy1—Dy1 <sup>i</sup>	36.52 (8)	Mo1 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	103.540 (12)
O2 <sup>vi</sup> —Dy1—Dy1 <sup>i</sup>	34.53 (8)	O4 <sup>xv</sup> —K1—Mo1 <sup>xii</sup>	79.68 (8)
O2 <sup>vii</sup> —Dy1—Dy1 <sup>i</sup>	142.40 (8)	O4—K1—Mo1 <sup>xii</sup>	90.40 (9)
O2 <sup>iv</sup> —Dy1—Dy1 <sup>ii</sup>	36.52 (8)	O4 <sup>xiv</sup> —K1—Mo1 <sup>xi</sup>	72.90 (7)
O2 <sup>v</sup> —Dy1—Dy1 <sup>ii</sup>	145.80 (8)	O4 <sup>xii</sup> —K1—Mo1	90.40 (9)
O2 <sup>vi</sup> —Dy1—Dy1 <sup>ii</sup>	142.40 (8)	O4 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	78.63 (8)
O2 <sup>vii</sup> —Dy1—Dy1 <sup>ii</sup>	34.53 (8)	O4 <sup>xv</sup> —K1—Mo1	126.93 (8)
O2 <sup>vi</sup> —Dy1—K1 <sup>iii</sup>	72.98 (8)	O4 <sup>xiv</sup> —K1—Mo1	79.68 (8)
O2 <sup>vii</sup> —Dy1—K1 <sup>iii</sup>	72.98 (8)	O4—K1—Mo1 <sup>xiii</sup>	78.63 (8)
O2 <sup>v</sup> —Dy1—K1 <sup>iii</sup>	102.55 (8)	O4—K1—Mo1 <sup>xi</sup>	148.63 (8)
O2 <sup>iv</sup> —Dy1—K1 <sup>iii</sup>	102.55 (8)	O4 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	148.63 (8)
O2 <sup>vii</sup> —Dy1—O2 <sup>vi</sup>	145.97 (16)	O4 <sup>xv</sup> —K1—Mo1 <sup>xi</sup>	89.19 (8)
O2 <sup>iv</sup> —Dy1—O2 <sup>vi</sup>	116.87 (14)	O4 <sup>xv</sup> —K1—Mo1 <sup>xiii</sup>	72.90 (7)
O2 <sup>iv</sup> —Dy1—O2 <sup>vii</sup>	71.05 (13)	O4—K1—Mo1	25.09 (8)
O2 <sup>v</sup> —Dy1—O2 <sup>vi</sup>	71.05 (13)	O4 <sup>xiv</sup> —K1—Mo1 <sup>xiii</sup>	89.19 (8)
O2 <sup>v</sup> —Dy1—O2 <sup>vii</sup>	116.87 (14)	O4 <sup>xii</sup> —K1—Mo1 <sup>xii</sup>	25.09 (8)
O2 <sup>v</sup> —Dy1—O2 <sup>iv</sup>	154.91 (17)	O4 <sup>xiv</sup> —K1—Mo1 <sup>xii</sup>	126.93 (8)
O1 <sup>viii</sup> —Dy1—Dy1 <sup>i</sup>	75.84 (8)	O4 <sup>xii</sup> —K1—O4 <sup>xv</sup>	76.26 (9)
O1—Dy1—Dy1 <sup>i</sup>	99.15 (8)	O4—K1—O4 <sup>xv</sup>	121.24 (5)

O1 <sup>viii</sup> —Dy1—Dy1 <sup>ii</sup>	99.15 (8)	O4 <sup>xv</sup> —K1—O4 <sup>xiv</sup>	150.24 (16)
O1—Dy1—Dy1 <sup>ii</sup>	75.84 (8)	O4—K1—O4 <sup>xiv</sup>	76.25 (8)
O1—Dy1—K1 <sup>iii</sup>	37.45 (8)	O4 <sup>xii</sup> —K1—O4 <sup>xiv</sup>	121.24 (5)
O1 <sup>viii</sup> —Dy1—K1 <sup>iii</sup>	37.45 (8)	O4—K1—O4 <sup>xii</sup>	113.67 (16)
O1—Dy1—O2 <sup>iv</sup>	72.59 (12)	O4 <sup>xii</sup> —K1—O1 <sup>xi</sup>	104.12 (10)
O1 <sup>viii</sup> —Dy1—O2 <sup>vi</sup>	84.04 (12)	O4—K1—O1 <sup>xi</sup>	134.94 (11)
O1—Dy1—O2 <sup>vii</sup>	84.04 (12)	O4—K1—O1 <sup>xiii</sup>	104.12 (10)
O1—Dy1—O2 <sup>vi</sup>	68.85 (11)	O4 <sup>xiv</sup> —K1—O1 <sup>xiii</sup>	90.21 (11)
O1 <sup>viii</sup> —Dy1—O2 <sup>iv</sup>	130.11 (12)	O4 <sup>xiv</sup> —K1—O1 <sup>xi</sup>	63.27 (10)
O1 <sup>viii</sup> —Dy1—O2 <sup>vii</sup>	68.85 (11)	O4 <sup>xii</sup> —K1—O1 <sup>xiii</sup>	134.94 (11)
O1—Dy1—O2 <sup>v</sup>	130.11 (12)	O4 <sup>xv</sup> —K1—O1 <sup>xiii</sup>	63.27 (10)
O1 <sup>viii</sup> —Dy1—O2 <sup>v</sup>	72.59 (12)	O4 <sup>xv</sup> —K1—O1 <sup>xi</sup>	90.21 (11)
O1 <sup>viii</sup> —Dy1—O1	74.89 (17)	O4 <sup>xv</sup> —K1—O3 <sup>xii</sup>	81.35 (10)
O1—Dy1—O3 <sup>ix</sup>	108.36 (12)	O4 <sup>xv</sup> —K1—O3	128.30 (10)
O1—Dy1—O3 <sup>ii</sup>	150.54 (12)	O4 <sup>xii</sup> —K1—O3 <sup>xii</sup>	53.25 (10)
O1 <sup>viii</sup> —Dy1—O3 <sup>ix</sup>	150.54 (12)	O4 <sup>xiv</sup> —K1—O3 <sup>xii</sup>	128.30 (10)
O1 <sup>viii</sup> —Dy1—O3 <sup>ii</sup>	108.36 (12)	O4 <sup>xiv</sup> —K1—O3	81.35 (10)
O3 <sup>ix</sup> —Dy1—Dy1 <sup>i</sup>	74.74 (8)	O4—K1—O3	53.25 (10)
O3 <sup>ix</sup> —Dy1—Dy1 <sup>ii</sup>	110.11 (8)	O4—K1—O3 <sup>xii</sup>	66.32 (10)
O3 <sup>ii</sup> —Dy1—Dy1 <sup>ii</sup>	74.74 (8)	O4 <sup>xii</sup> —K1—O3	66.32 (10)
O3 <sup>ii</sup> —Dy1—Dy1 <sup>i</sup>	110.11 (8)	O1 <sup>xiii</sup> —K1—Mo1	129.20 (7)
O3 <sup>ii</sup> —Dy1—K1 <sup>iii</sup>	138.31 (8)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xi</sup>	81.73 (8)
O3 <sup>ix</sup> —Dy1—K1 <sup>iii</sup>	138.31 (8)	O1 <sup>xi</sup> —K1—Mo1 <sup>xii</sup>	129.20 (7)
O3 <sup>ix</sup> —Dy1—O2 <sup>vii</sup>	139.98 (11)	O1 <sup>xi</sup> —K1—Mo1	142.63 (7)
O3 <sup>ix</sup> —Dy1—O2 <sup>iv</sup>	76.68 (11)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xii</sup>	142.63 (7)
O3 <sup>ii</sup> —Dy1—O2 <sup>vii</sup>	70.80 (12)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xiii</sup>	26.02 (7)
O3 <sup>ii</sup> —Dy1—O2 <sup>v</sup>	76.68 (11)	O1 <sup>xi</sup> —K1—Mo1 <sup>xiii</sup>	81.73 (8)
O3 <sup>ix</sup> —Dy1—O2 <sup>v</sup>	84.61 (12)	O1 <sup>xi</sup> —K1—Mo1 <sup>xi</sup>	26.02 (7)
O3 <sup>ix</sup> —Dy1—O2 <sup>vi</sup>	70.80 (12)	O1 <sup>xiii</sup> —K1—O1 <sup>xi</sup>	59.42 (14)
O3 <sup>ii</sup> —Dy1—O2 <sup>iv</sup>	84.61 (12)	O1 <sup>xiii</sup> —K1—O3	157.10 (9)
O3 <sup>ii</sup> —Dy1—O2 <sup>vi</sup>	139.98 (11)	O1 <sup>xi</sup> —K1—O3 <sup>xii</sup>	157.10 (9)
O3 <sup>ii</sup> —Dy1—O3 <sup>ix</sup>	83.39 (16)	O1 <sup>xi</sup> —K1—O3	131.78 (9)
K1—Mo1—K1 <sup>iii</sup>	78.467 (11)	O1 <sup>xiii</sup> —K1—O3 <sup>xii</sup>	131.78 (9)
K1—Mo1—K1 <sup>x</sup>	76.833 (11)	O3 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	115.44 (6)
K1 <sup>iii</sup> —Mo1—K1 <sup>x</sup>	81.33 (3)	O3 <sup>xii</sup> —K1—Mo1	51.60 (6)
O4—Mo1—K1 <sup>x</sup>	36.37 (12)	O3—K1—Mo1 <sup>xi</sup>	115.44 (6)
O4—Mo1—K1	41.29 (11)	O3—K1—Mo1 <sup>xiii</sup>	131.86 (6)
O4—Mo1—K1 <sup>iii</sup>	71.06 (12)	O3—K1—Mo1	28.36 (6)
O4—Mo1—O2	109.59 (16)	O3 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	131.86 (6)
O4—Mo1—O1	106.89 (16)	O3—K1—Mo1 <sup>xii</sup>	51.60 (6)
O4—Mo1—O3	105.13 (16)	O3 <sup>xii</sup> —K1—Mo1 <sup>xii</sup>	28.36 (6)
O2—Mo1—K1 <sup>x</sup>	86.44 (11)	O3 <sup>xii</sup> —K1—O3	47.91 (11)
O2—Mo1—K1 <sup>iii</sup>	156.32 (9)	Mo1—O4—K1 <sup>x</sup>	122.04 (17)
O2—Mo1—K1	118.38 (10)	Mo1—O4—K1	113.61 (16)
O1—Mo1—K1 <sup>iii</sup>	43.41 (11)	K1—O4—K1 <sup>x</sup>	122.44 (13)
O1—Mo1—K1	121.60 (11)	Dy1 <sup>iv</sup> —O2—Dy1 <sup>xvi</sup>	108.95 (13)
O1—Mo1—K1 <sup>x</sup>	95.79 (11)	Mo1—O2—Dy1 <sup>xvi</sup>	119.80 (14)
O1—Mo1—O2	118.81 (15)	Mo1—O2—Dy1 <sup>iv</sup>	128.01 (15)

O3—Mo1—K1 <sup>iii</sup>	90.42 (11)	Dy1—O1—K1 <sup>iii</sup>	112.84 (12)
O3—Mo1—K1 <sup>x</sup>	141.16 (11)	Mo1—O1—Dy1	125.41 (17)
O3—Mo1—K1	64.32 (11)	Mo1—O1—K1 <sup>iii</sup>	110.57 (15)
O3—Mo1—O2	111.51 (15)	Dy1 <sup>ii</sup> —O3—K1	145.45 (13)
O3—Mo1—O1	103.94 (16)	Mo1—O3—Dy1 <sup>ii</sup>	127.15 (18)
Mo1—K1—Mo1 <sup>xiii</sup>	103.540 (12)	Mo1—O3—K1	87.32 (12)

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

## Potassium holmium bis(molybdate) (Ho\_Molybdate)

### Crystal data

KHo(MoO<sub>4</sub>)<sub>2</sub>

$M_r = 523.91$

Orthorhombic, *Pbcn*

Hall symbol: -P 2n 2ab

$a = 5.0770$  (15) Å

$b = 18.161$  (5) Å

$c = 7.934$  (2) Å

$V = 731.5$  (4) Å<sup>3</sup>

$Z = 4$

$F(000) = 936$

$D_x = 4.757$  Mg m<sup>-3</sup>

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

Cell parameters from 2917 reflections

$\theta = 3.4$ – $28.8^\circ$

$\mu = 14.66$  mm<sup>-1</sup>

$T = 273$  K

Plate, light red

$0.06 \times 0.06 \times 0.04$  mm

### Data collection

Bruker APEXII CCD

diffractometer

Radiation source: X-ray tube

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(SADABS; Krause et al., 2015)

$T_{\min} = 0.248$ ,  $T_{\max} = 0.343$

7665 measured reflections

936 independent reflections

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

$R_{\text{int}} = 0.032$

$\theta_{\max} = 28.9^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -6 \rightarrow 6$

$k = -24 \rightarrow 24$

$l = -10 \rightarrow 10$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.027$

$wR(F^2) = 0.063$

$S = 1.25$

936 reflections

56 parameters

0 restraints

0 constraints

Primary atom site location: dual

$w = 1/[\sigma^2(F_o^2) + 11.609P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 1.51$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -1.37$  e Å<sup>-3</sup>

### 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.** Reflections were merged by SHELXL according to the crystal class for the calculation of statistics and refinement.

\_reflns Friedel fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.



Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ho1	0.000000	0.49423 (2)	0.750000	0.00704 (12)
Mo1	0.47909 (9)	0.60179 (3)	0.51614 (6)	0.00726 (13)
K1	0.500000	0.77102 (11)	0.250000	0.0141 (4)
O4	0.6095 (9)	0.6894 (3)	0.5227 (6)	0.0122 (9)
O2	0.7503 (9)	0.5342 (2)	0.4941 (5)	0.0095 (8)
O1	0.2583 (8)	0.5939 (2)	0.6923 (5)	0.0096 (8)
O3	0.2700 (9)	0.6008 (2)	0.3389 (5)	0.0093 (8)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Ho1	0.00566 (18)	0.0094 (2)	0.00606 (18)	0.000	-0.00040 (14)	0.000
Mo1	0.0058 (2)	0.0084 (3)	0.0076 (2)	0.00040 (19)	-0.00009 (17)	0.00032 (17)
K1	0.0177 (9)	0.0132 (9)	0.0115 (8)	0.000	0.0013 (8)	0.000
O4	0.013 (2)	0.014 (2)	0.010 (2)	-0.0011 (18)	0.0002 (17)	0.0016 (17)
O2	0.0080 (19)	0.014 (2)	0.0061 (19)	0.0016 (17)	0.0037 (18)	-0.0005 (17)
O1	0.0072 (19)	0.013 (2)	0.0085 (19)	-0.0001 (17)	0.0015 (16)	0.0001 (17)
O3	0.013 (2)	0.012 (2)	0.0035 (19)	-0.0008 (19)	-0.0005 (15)	0.0000 (16)

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

Ho1—Ho1 <sup>i</sup>	3.9723 (11)	Mo1—O3	1.762 (4)
Ho1—Ho1 <sup>ii</sup>	3.9723 (11)	K1—Mo1 <sup>xi</sup>	3.8332 (14)
Ho1—K1 <sup>iii</sup>	4.263 (2)	K1—Mo1 <sup>xii</sup>	3.7303 (18)
Ho1—O2 <sup>iv</sup>	2.372 (4)	K1—Mo1 <sup>xiii</sup>	3.8332 (14)
Ho1—O2 <sup>v</sup>	2.372 (4)	K1—O4	2.680 (5)
Ho1—O2 <sup>vi</sup>	2.501 (4)	K1—O4 <sup>xiv</sup>	2.775 (5)
Ho1—O2 <sup>vii</sup>	2.501 (4)	K1—O4 <sup>xii</sup>	2.681 (5)
Ho1—O1 <sup>viii</sup>	2.282 (4)	K1—O4 <sup>xv</sup>	2.775 (5)
Ho1—O1	2.282 (4)	K1—O1 <sup>xiii</sup>	2.819 (5)
Ho1—O3 <sup>ix</sup>	2.314 (4)	K1—O1 <sup>xi</sup>	2.819 (5)
Ho1—O3 <sup>i</sup>	2.314 (4)	K1—O3	3.380 (5)
Mo1—K1	3.7303 (18)	K1—O3 <sup>xii</sup>	3.380 (5)
Mo1—K1 <sup>x</sup>	3.9713 (14)	O4—K1 <sup>x</sup>	2.775 (5)
Mo1—K1 <sup>iii</sup>	3.8332 (14)	O2—Ho1 <sup>xvi</sup>	2.501 (4)
Mo1—O4	1.724 (5)	O2—Ho1 <sup>v</sup>	2.372 (4)
Mo1—O2	1.853 (4)	O1—K1 <sup>iii</sup>	2.819 (5)
Mo1—O1	1.797 (4)	O3—Ho1 <sup>i</sup>	2.314 (4)
Ho1 <sup>ii</sup> —Ho1—Ho1 <sup>i</sup>	173.96 (2)	Mo1 <sup>xii</sup> —K1—Mo1	69.05 (4)
Ho1 <sup>ii</sup> —Ho1—K1 <sup>iii</sup>	86.979 (11)	Mo1 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	103.92 (2)
Ho1 <sup>i</sup> —Ho1—K1 <sup>iii</sup>	86.979 (11)	Mo1—K1—Mo1 <sup>xiii</sup>	103.92 (2)
O2 <sup>iv</sup> —Ho1—Ho1 <sup>i</sup>	145.79 (11)	Mo1 <sup>xiii</sup> —K1—Mo1 <sup>xi</sup>	105.89 (5)
O2 <sup>v</sup> —Ho1—Ho1 <sup>i</sup>	36.49 (11)	Mo1—K1—Mo1 <sup>xi</sup>	138.79 (2)
O2 <sup>vi</sup> —Ho1—Ho1 <sup>i</sup>	34.33 (10)	O4 <sup>xv</sup> —K1—Mo1	126.98 (10)

O2 <sup>vii</sup> —Ho1—Ho1 <sup>i</sup>	142.68 (10)	O4—K1—Mo1	24.98 (10)
O2 <sup>iv</sup> —Ho1—Ho1 <sup>ii</sup>	36.49 (11)	O4 <sup>xiv</sup> —K1—Mo1 <sup>xi</sup>	72.85 (10)
O2 <sup>v</sup> —Ho1—Ho1 <sup>ii</sup>	145.79 (11)	O4 <sup>xii</sup> —K1—Mo1 <sup>xii</sup>	24.98 (10)
O2 <sup>vi</sup> —Ho1—Ho1 <sup>ii</sup>	142.68 (10)	O4 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	79.11 (10)
O2 <sup>vii</sup> —Ho1—Ho1 <sup>ii</sup>	34.33 (10)	O4 <sup>xv</sup> —K1—Mo1 <sup>xii</sup>	79.93 (10)
O2 <sup>vi</sup> —Ho1—K1 <sup>iii</sup>	73.13 (10)	O4 <sup>xiv</sup> —K1—Mo1 <sup>xii</sup>	126.98 (10)
O2 <sup>vii</sup> —Ho1—K1 <sup>iii</sup>	73.13 (10)	O4—K1—Mo1 <sup>xiii</sup>	79.11 (10)
O2 <sup>v</sup> —Ho1—K1 <sup>iii</sup>	102.57 (11)	O4—K1—Mo1 <sup>xi</sup>	148.80 (10)
O2 <sup>iv</sup> —Ho1—K1 <sup>iii</sup>	102.57 (11)	O4 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	148.80 (10)
O2 <sup>vii</sup> —Ho1—O2 <sup>vi</sup>	146.3 (2)	O4 <sup>xv</sup> —K1—Mo1 <sup>xi</sup>	89.01 (10)
O2 <sup>iv</sup> —Ho1—O2 <sup>vi</sup>	117.06 (18)	O4 <sup>xv</sup> —K1—Mo1 <sup>xiii</sup>	72.85 (10)
O2 <sup>iv</sup> —Ho1—O2 <sup>vii</sup>	70.81 (17)	O4—K1—Mo1 <sup>xii</sup>	89.73 (11)
O2 <sup>v</sup> —Ho1—O2 <sup>vi</sup>	70.81 (17)	O4 <sup>xiv</sup> —K1—Mo1 <sup>xiii</sup>	89.01 (10)
O2 <sup>v</sup> —Ho1—O2 <sup>vii</sup>	117.06 (18)	O4 <sup>xii</sup> —K1—Mo1	89.73 (11)
O2 <sup>v</sup> —Ho1—O2 <sup>iv</sup>	154.9 (2)	O4 <sup>xiv</sup> —K1—Mo1	79.93 (10)
O1—Ho1—Ho1 <sup>i</sup>	75.99 (11)	O4 <sup>xii</sup> —K1—O4 <sup>xv</sup>	76.52 (11)
O1 <sup>viii</sup> —Ho1—Ho1 <sup>i</sup>	99.12 (11)	O4—K1—O4 <sup>xv</sup>	121.30 (6)
O1—Ho1—Ho1 <sup>ii</sup>	99.12 (11)	O4 <sup>xv</sup> —K1—O4 <sup>xiv</sup>	150.0 (2)
O1 <sup>viii</sup> —Ho1—Ho1 <sup>ii</sup>	75.99 (11)	O4—K1—O4 <sup>xiv</sup>	76.52 (11)
O1 <sup>viii</sup> —Ho1—K1 <sup>iii</sup>	37.49 (11)	O4 <sup>xii</sup> —K1—O4 <sup>xiv</sup>	121.30 (6)
O1—Ho1—K1 <sup>iii</sup>	37.50 (11)	O4—K1—O4 <sup>xii</sup>	112.9 (2)
O1 <sup>viii</sup> —Ho1—O2 <sup>iv</sup>	72.64 (15)	O4 <sup>xii</sup> —K1—O1 <sup>xi</sup>	104.69 (13)
O1—Ho1—O2 <sup>vi</sup>	84.17 (15)	O4—K1—O1 <sup>xi</sup>	135.12 (13)
O1 <sup>viii</sup> —Ho1—O2 <sup>vii</sup>	84.17 (15)	O4—K1—O1 <sup>xiii</sup>	104.69 (13)
O1 <sup>viii</sup> —Ho1—O2 <sup>vi</sup>	68.97 (15)	O4 <sup>xiv</sup> —K1—O1 <sup>xiii</sup>	90.09 (14)
O1—Ho1—O2 <sup>iv</sup>	130.07 (15)	O4 <sup>xiv</sup> —K1—O1 <sup>xi</sup>	63.11 (13)
O1—Ho1—O2 <sup>vii</sup>	68.97 (15)	O4 <sup>xii</sup> —K1—O1 <sup>xiii</sup>	135.13 (13)
O1 <sup>viii</sup> —Ho1—O2 <sup>v</sup>	130.06 (15)	O4 <sup>xv</sup> —K1—O1 <sup>xiii</sup>	63.11 (13)
O1—Ho1—O2 <sup>v</sup>	72.64 (15)	O4 <sup>xv</sup> —K1—O1 <sup>xi</sup>	90.09 (14)
O1—Ho1—O1 <sup>viii</sup>	75.0 (2)	O4 <sup>xv</sup> —K1—O3	128.28 (13)
O1 <sup>viii</sup> —Ho1—O3 <sup>ix</sup>	150.57 (15)	O4 <sup>xv</sup> —K1—O3 <sup>xii</sup>	81.63 (12)
O1 <sup>viii</sup> —Ho1—O3 <sup>i</sup>	108.19 (16)	O4 <sup>xii</sup> —K1—O3	65.85 (13)
O1—Ho1—O3 <sup>ix</sup>	108.19 (16)	O4 <sup>xiv</sup> —K1—O3	81.63 (12)
O1—Ho1—O3 <sup>i</sup>	150.57 (15)	O4 <sup>xiv</sup> —K1—O3 <sup>xii</sup>	128.28 (13)
O3 <sup>ix</sup> —Ho1—Ho1 <sup>i</sup>	110.11 (10)	O4—K1—O3 <sup>xii</sup>	65.85 (13)
O3 <sup>ix</sup> —Ho1—Ho1 <sup>ii</sup>	74.62 (10)	O4—K1—O3	52.94 (12)
O3 <sup>i</sup> —Ho1—Ho1 <sup>ii</sup>	110.11 (10)	O4 <sup>xii</sup> —K1—O3 <sup>xii</sup>	52.93 (12)
O3 <sup>i</sup> —Ho1—Ho1 <sup>i</sup>	74.62 (10)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xii</sup>	142.71 (9)
O3 <sup>i</sup> —Ho1—K1 <sup>iii</sup>	138.22 (11)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xi</sup>	81.34 (10)
O3 <sup>ix</sup> —Ho1—K1 <sup>iii</sup>	138.21 (11)	O1 <sup>xi</sup> —K1—Mo1	142.71 (9)
O3 <sup>ix</sup> —Ho1—O2 <sup>vii</sup>	70.64 (15)	O1 <sup>xi</sup> —K1—Mo1 <sup>xii</sup>	129.66 (9)
O3 <sup>ix</sup> —Ho1—O2 <sup>iv</sup>	84.58 (15)	O1 <sup>xiii</sup> —K1—Mo1	129.66 (9)
O3 <sup>i</sup> —Ho1—O2 <sup>vii</sup>	139.83 (14)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xiii</sup>	26.09 (9)
O3 <sup>i</sup> —Ho1—O2 <sup>v</sup>	84.58 (15)	O1 <sup>xi</sup> —K1—Mo1 <sup>xiii</sup>	81.34 (10)
O3 <sup>ix</sup> —Ho1—O2 <sup>v</sup>	76.70 (15)	O1 <sup>xi</sup> —K1—Mo1 <sup>xi</sup>	26.09 (9)
O3 <sup>ix</sup> —Ho1—O2 <sup>vi</sup>	139.83 (14)	O1 <sup>xiii</sup> —K1—O1 <sup>xi</sup>	59.05 (18)
O3 <sup>i</sup> —Ho1—O2 <sup>iv</sup>	76.70 (15)	O1 <sup>xiii</sup> —K1—O3 <sup>xii</sup>	132.01 (12)
O3 <sup>i</sup> —Ho1—O2 <sup>vi</sup>	70.64 (15)	O1 <sup>xi</sup> —K1—O3	132.01 (12)

O3 <sup>i</sup> —Ho1—O3 <sup>ix</sup>	83.6 (2)	O1 <sup>xi</sup> —K1—O3 <sup>xii</sup>	157.36 (11)
K1—Mo1—K1 <sup>iii</sup>	78.20 (2)	O1 <sup>xiii</sup> —K1—O3	157.36 (11)
K1—Mo1—K1 <sup>x</sup>	76.49 (2)	O3—K1—Mo1 <sup>xiii</sup>	132.03 (7)
K1 <sup>iii</sup> —Mo1—K1 <sup>x</sup>	81.14 (4)	O3—K1—Mo1 <sup>xii</sup>	51.26 (8)
O4—Mo1—K1 <sup>x</sup>	36.25 (15)	O3 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	132.03 (7)
O4—Mo1—K1	41.04 (15)	O3 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	115.66 (8)
O4—Mo1—K1 <sup>iii</sup>	70.92 (15)	O3 <sup>xii</sup> —K1—Mo1 <sup>xii</sup>	28.14 (7)
O4—Mo1—O2	109.2 (2)	O3—K1—Mo1 <sup>xi</sup>	115.66 (8)
O4—Mo1—O1	106.8 (2)	O3 <sup>xii</sup> —K1—Mo1	51.26 (8)
O4—Mo1—O3	105.4 (2)	O3—K1—Mo1	28.14 (7)
O2—Mo1—K1 <sup>x</sup>	86.25 (14)	O3—K1—O3 <sup>xii</sup>	47.61 (15)
O2—Mo1—K1 <sup>iii</sup>	156.40 (13)	Mo1—O4—K1 <sup>x</sup>	122.2 (2)
O2—Mo1—K1	118.13 (13)	Mo1—O4—K1	114.0 (2)
O1—Mo1—K1 <sup>iii</sup>	43.60 (14)	K1—O4—K1 <sup>x</sup>	121.97 (17)
O1—Mo1—K1	121.56 (14)	Ho1 <sup>v</sup> —O2—Ho1 <sup>xvi</sup>	109.19 (17)
O1—Mo1—K1 <sup>x</sup>	95.64 (14)	Mo1—O2—Ho1 <sup>xvi</sup>	119.50 (19)
O1—Mo1—O2	118.95 (19)	Mo1—O2—Ho1 <sup>v</sup>	128.2 (2)
O3—Mo1—K1 <sup>iii</sup>	90.59 (14)	Ho1—O1—K1 <sup>iii</sup>	112.98 (16)
O3—Mo1—K1 <sup>x</sup>	141.28 (15)	Mo1—O1—Ho1	125.3 (2)
O3—Mo1—K1	64.79 (14)	Mo1—O1—K1 <sup>iii</sup>	110.32 (19)
O3—Mo1—O2	111.44 (19)	Ho1 <sup>i</sup> —O3—K1	145.41 (16)
O3—Mo1—O1	104.1 (2)	Mo1—O3—Ho1 <sup>i</sup>	127.5 (2)
Mo1 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	138.79 (2)	Mo1—O3—K1	87.06 (16)

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

### Potassium erbium bis(molybdate) (Er\_Molybdate)

#### Crystal data

KEr(MoO<sub>4</sub>)<sub>2</sub>

$M_r = 526.24$

Orthorhombic, *Pbcn*

Hall symbol:  $-P\ 2n\ 2ab$

$a = 5.0602$  (2) Å

$b = 18.1965$  (8) Å

$c = 7.8920$  (3) Å

$V = 726.68$  (5) Å<sup>3</sup>

$Z = 4$

$F(000) = 940$

$D_x = 4.810$  Mg m<sup>-3</sup>

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

Cell parameters from 7328 reflections

$\theta = 3.4\text{--}34.0^\circ$

$\mu = 15.42$  mm<sup>-1</sup>

$T = 100$  K

Plate, light pink

$0.28 \times 0.15 \times 0.02$  mm

#### Data collection

Rigaku XtaLAB Synergy-S, HyPix  
diffractometer

Radiation source: micro-focus sealed X-ray tube

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlisPro; Rigaku OD, 2019)

$T_{\min} = 0.091, T_{\max} = 0.911$

21246 measured reflections

1386 independent reflections

1255 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.072$

$\theta_{\text{max}} = 34.0^\circ, \theta_{\text{min}} = 2.2^\circ$

$h = -7 \rightarrow 7$

$k = -28 \rightarrow 27$

$l = -12 \rightarrow 10$

Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.027$   
 $wR(F^2) = 0.073$   
 $S = 1.06$   
 1386 reflections  
 56 parameters  
 0 restraints

0 constraints  
 Primary atom site location: dual  
 $w = 1/[\sigma^2(F_o^2) + (0.0345P)^2 + 4.1539P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.002$   
 $\Delta\rho_{\max} = 2.04 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -1.62 \text{ e } \text{\AA}^{-3}$

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Er1	1.000000	0.50535 (2)	0.750000	0.00602 (9)
Mo1	0.52026 (6)	0.39859 (2)	0.51517 (4)	0.00637 (9)
K1	0.500000	0.22821 (7)	0.250000	0.0107 (2)
O4	0.3903 (5)	0.31111 (15)	0.5213 (3)	0.0102 (5)
O2	0.2497 (5)	0.46619 (15)	0.4954 (3)	0.0084 (5)
O1	0.7417 (5)	0.40581 (14)	0.6914 (3)	0.0100 (4)
O3	0.7315 (5)	0.39970 (14)	0.3369 (3)	0.0099 (5)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Er1	0.00367 (12)	0.00995 (13)	0.00443 (13)	0.000	-0.00030 (6)	0.000
Mo1	0.00374 (14)	0.00961 (16)	0.00575 (15)	0.00027 (9)	-0.00044 (8)	-0.00032 (9)
K1	0.0112 (5)	0.0124 (5)	0.0085 (5)	0.000	-0.0007 (3)	0.000
O4	0.0079 (11)	0.0127 (12)	0.0100 (11)	-0.0029 (9)	0.0009 (9)	-0.0003 (9)
O2	0.0052 (10)	0.0131 (12)	0.0067 (10)	0.0007 (9)	-0.0008 (8)	-0.0006 (8)
O1	0.0077 (10)	0.0142 (12)	0.0080 (10)	-0.0014 (9)	-0.0019 (9)	-0.0004 (9)
O3	0.0068 (10)	0.0153 (12)	0.0074 (10)	-0.0014 (9)	0.0006 (8)	-0.0003 (8)

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

Er1—Er1 <sup>i</sup>	3.9508 (2)	Mo1—O3	1.767 (2)
Er1—Er1 <sup>ii</sup>	3.9508 (1)	K1—Mo1 <sup>xi</sup>	3.8277 (8)
Er1—K1 <sup>iii</sup>	4.2499 (13)	K1—Mo1 <sup>xii</sup>	3.7419 (10)
Er1—O2 <sup>iv</sup>	2.370 (2)	K1—Mo1 <sup>xiii</sup>	3.8277 (8)
Er1—O2 <sup>v</sup>	2.370 (2)	K1—O4	2.677 (3)
Er1—O2 <sup>vi</sup>	2.478 (2)	K1—O4 <sup>xiv</sup>	2.770 (3)
Er1—O2 <sup>vii</sup>	2.478 (2)	K1—O4 <sup>xii</sup>	2.677 (3)
Er1—O1 <sup>viii</sup>	2.281 (2)	K1—O4 <sup>xv</sup>	2.770 (3)
Er1—O1	2.281 (3)	K1—O1 <sup>xiii</sup>	2.805 (3)
Er1—O3 <sup>ix</sup>	2.303 (3)	K1—O1 <sup>xi</sup>	2.805 (3)

Er1—O3 <sup>ii</sup>	2.303 (3)	K1—O3	3.403 (3)
Mo1—K1	3.7419 (10)	K1—O3 <sup>xii</sup>	3.403 (3)
Mo1—K1 <sup>x</sup>	3.9610 (8)	O4—K1 <sup>x</sup>	2.770 (3)
Mo1—K1 <sup>iii</sup>	3.8278 (8)	O2—Er1 <sup>xvi</sup>	2.478 (2)
Mo1—O4	1.723 (3)	O2—Er1 <sup>iv</sup>	2.370 (2)
Mo1—O2	1.847 (3)	O1—K1 <sup>iii</sup>	2.805 (3)
Mo1—O1	1.791 (2)	O3—Er1 <sup>ii</sup>	2.303 (3)
Er1 <sup>ii</sup> —Er1—Er1 <sup>i</sup>	174.354 (13)	Mo1 <sup>xii</sup> —K1—Mo1	68.10 (2)
Er1 <sup>ii</sup> —Er1—K1 <sup>iii</sup>	87.177 (7)	Mo1 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	104.243 (10)
Er1 <sup>i</sup> —Er1—K1 <sup>iii</sup>	87.177 (7)	Mo1—K1—Mo1 <sup>xiii</sup>	104.243 (10)
O2 <sup>iv</sup> —Er1—Er1 <sup>i</sup>	145.80 (6)	Mo1 <sup>xiii</sup> —K1—Mo1 <sup>xi</sup>	105.86 (3)
O2 <sup>v</sup> —Er1—Er1 <sup>i</sup>	36.34 (6)	Mo1—K1—Mo1 <sup>xi</sup>	138.837 (12)
O2 <sup>vi</sup> —Er1—Er1 <sup>i</sup>	34.52 (6)	O4 <sup>xv</sup> —K1—Mo1	126.73 (6)
O2 <sup>vii</sup> —Er1—Er1 <sup>i</sup>	142.71 (6)	O4—K1—Mo1	24.71 (6)
O2 <sup>iv</sup> —Er1—Er1 <sup>ii</sup>	36.34 (6)	O4 <sup>xiv</sup> —K1—Mo1 <sup>xi</sup>	73.00 (5)
O2 <sup>v</sup> —Er1—Er1 <sup>ii</sup>	145.80 (6)	O4 <sup>xii</sup> —K1—Mo1 <sup>xii</sup>	24.71 (6)
O2 <sup>vi</sup> —Er1—Er1 <sup>ii</sup>	142.71 (6)	O4 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	79.69 (6)
O2 <sup>vii</sup> —Er1—Er1 <sup>ii</sup>	34.52 (6)	O4 <sup>xv</sup> —K1—Mo1 <sup>xii</sup>	80.21 (6)
O2 <sup>vi</sup> —Er1—K1 <sup>iii</sup>	73.29 (6)	O4 <sup>xiv</sup> —K1—Mo1 <sup>xiii</sup>	126.73 (6)
O2 <sup>vii</sup> —Er1—K1 <sup>iii</sup>	73.29 (6)	O4—K1—Mo1 <sup>xiii</sup>	79.69 (6)
O2 <sup>v</sup> —Er1—K1 <sup>iii</sup>	102.63 (6)	O4—K1—Mo1 <sup>xi</sup>	149.13 (6)
O2 <sup>iv</sup> —Er1—K1 <sup>iii</sup>	102.63 (6)	O4 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	149.13 (6)
O2 <sup>vii</sup> —Er1—O2 <sup>vi</sup>	146.57 (12)	O4 <sup>xv</sup> —K1—Mo1 <sup>xi</sup>	88.91 (6)
O2 <sup>iv</sup> —Er1—O2 <sup>vi</sup>	116.98 (11)	O4 <sup>xv</sup> —K1—Mo1 <sup>xiii</sup>	73.00 (5)
O2 <sup>iv</sup> —Er1—O2 <sup>vii</sup>	70.86 (10)	O4—K1—Mo1 <sup>xii</sup>	88.55 (6)
O2 <sup>v</sup> —Er1—O2 <sup>vi</sup>	70.86 (10)	O4 <sup>xiv</sup> —K1—Mo1 <sup>xiii</sup>	88.91 (6)
O2 <sup>v</sup> —Er1—O2 <sup>vii</sup>	116.98 (11)	O4 <sup>xii</sup> —K1—Mo1	88.55 (6)
O2 <sup>v</sup> —Er1—O2 <sup>iv</sup>	154.75 (13)	O4 <sup>xiv</sup> —K1—Mo1	80.21 (6)
O1—Er1—Er1 <sup>i</sup>	99.40 (6)	O4 <sup>xii</sup> —K1—O4 <sup>xv</sup>	76.83 (6)
O1 <sup>viii</sup> —Er1—Er1 <sup>i</sup>	76.03 (6)	O4—K1—O4 <sup>xv</sup>	121.26 (4)
O1—Er1—Er1 <sup>ii</sup>	76.03 (6)	O4 <sup>xv</sup> —K1—O4 <sup>xiv</sup>	150.06 (12)
O1 <sup>viii</sup> —Er1—Er1 <sup>ii</sup>	99.39 (6)	O4—K1—O4 <sup>xiv</sup>	76.83 (6)
O1 <sup>viii</sup> —Er1—K1 <sup>iii</sup>	37.43 (6)	O4 <sup>xii</sup> —K1—O4 <sup>xiv</sup>	121.26 (4)
O1—Er1—K1 <sup>iii</sup>	37.43 (6)	O4—K1—O4 <sup>xii</sup>	111.41 (12)
O1 <sup>viii</sup> —Er1—O2 <sup>iv</sup>	130.14 (9)	O4 <sup>xii</sup> —K1—O1 <sup>xi</sup>	105.16 (8)
O1—Er1—O2 <sup>vi</sup>	69.13 (8)	O4—K1—O1 <sup>xi</sup>	135.91 (8)
O1 <sup>viii</sup> —Er1—O2 <sup>vii</sup>	69.13 (8)	O4—K1—O1 <sup>xiii</sup>	105.16 (8)
O1 <sup>viii</sup> —Er1—O2 <sup>vi</sup>	84.23 (9)	O4 <sup>xiv</sup> —K1—O1 <sup>xiii</sup>	90.02 (8)
O1—Er1—O2 <sup>iv</sup>	72.69 (9)	O4 <sup>xiv</sup> —K1—O1 <sup>xi</sup>	63.29 (8)
O1—Er1—O2 <sup>vii</sup>	84.23 (9)	O4 <sup>xii</sup> —K1—O1 <sup>xiii</sup>	135.91 (8)
O1 <sup>viii</sup> —Er1—O2 <sup>v</sup>	72.70 (9)	O4 <sup>xv</sup> —K1—O1 <sup>xiii</sup>	63.29 (8)
O1—Er1—O2 <sup>v</sup>	130.14 (9)	O4 <sup>xv</sup> —K1—O1 <sup>xi</sup>	90.02 (8)
O1—Er1—O1 <sup>viii</sup>	74.85 (13)	O4 <sup>xv</sup> —K1—O3	127.86 (8)
O1 <sup>viii</sup> —Er1—O3 <sup>ix</sup>	150.89 (9)	O4 <sup>xv</sup> —K1—O3 <sup>xii</sup>	81.96 (7)
O1 <sup>viii</sup> —Er1—O3 <sup>ii</sup>	108.55 (10)	O4 <sup>xii</sup> —K1—O3	64.73 (7)
O1—Er1—O3 <sup>ix</sup>	108.55 (10)	O4 <sup>xiv</sup> —K1—O3	81.96 (7)
O1—Er1—O3 <sup>ii</sup>	150.89 (9)	O4 <sup>xiv</sup> —K1—O3 <sup>xii</sup>	127.86 (8)



O3 <sup>ix</sup> —Er1—Er1 <sup>i</sup>	74.90 (6)	O4—K1—O3 <sup>xii</sup>	64.73 (7)
O3 <sup>ix</sup> —Er1—Er1 <sup>ii</sup>	109.54 (6)	O4—K1—O3	52.66 (7)
O3 <sup>ii</sup> —Er1—Er1 <sup>ii</sup>	74.90 (6)	O4 <sup>xii</sup> —K1—O3 <sup>xii</sup>	52.66 (7)
O3 <sup>ii</sup> —Er1—Er1 <sup>i</sup>	109.54 (6)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xii</sup>	143.10 (5)
O3 <sup>ii</sup> —Er1—K1 <sup>iii</sup>	138.62 (6)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xi</sup>	81.44 (6)
O3 <sup>ix</sup> —Er1—K1 <sup>iii</sup>	138.62 (6)	O1 <sup>xi</sup> —K1—Mo1	143.10 (5)
O3 <sup>ix</sup> —Er1—O2 <sup>vii</sup>	139.31 (8)	O1 <sup>xi</sup> —K1—Mo1 <sup>xii</sup>	129.86 (5)
O3 <sup>ix</sup> —Er1—O2 <sup>iv</sup>	76.40 (8)	O1 <sup>xiii</sup> —K1—Mo1	129.86 (5)
O3 <sup>ii</sup> —Er1—O2 <sup>vii</sup>	70.94 (9)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xiii</sup>	25.94 (5)
O3 <sup>ii</sup> —Er1—O2 <sup>v</sup>	76.40 (8)	O1 <sup>xi</sup> —K1—Mo1 <sup>xiii</sup>	81.44 (6)
O3 <sup>ix</sup> —Er1—O2 <sup>v</sup>	84.67 (9)	O1 <sup>xi</sup> —K1—Mo1 <sup>xi</sup>	25.94 (5)
O3 <sup>ix</sup> —Er1—O2 <sup>vi</sup>	70.94 (9)	O1 <sup>xiii</sup> —K1—O1 <sup>xi</sup>	59.23 (10)
O3 <sup>ii</sup> —Er1—O2 <sup>iv</sup>	84.67 (9)	O1 <sup>xiii</sup> —K1—O3 <sup>xii</sup>	132.07 (6)
O3 <sup>ii</sup> —Er1—O2 <sup>vi</sup>	139.31 (8)	O1 <sup>xi</sup> —K1—O3	132.07 (6)
O3 <sup>ii</sup> —Er1—O3 <sup>ix</sup>	82.76 (13)	O1 <sup>xi</sup> —K1—O3 <sup>xii</sup>	157.57 (7)
K1—Mo1—K1 <sup>iii</sup>	77.802 (9)	O1 <sup>xiii</sup> —K1—O3	157.57 (7)
K1—Mo1—K1 <sup>x</sup>	76.162 (9)	O3—K1—Mo1 <sup>xiii</sup>	132.33 (4)
K1 <sup>iii</sup> —Mo1—K1 <sup>x</sup>	81.02 (2)	O3—K1—Mo1 <sup>xii</sup>	50.39 (4)
O4—Mo1—K1 <sup>x</sup>	36.40 (9)	O3 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	132.33 (4)
O4—Mo1—K1	40.51 (8)	O3 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	115.60 (4)
O4—Mo1—K1 <sup>iii</sup>	70.83 (9)	O3 <sup>xii</sup> —K1—Mo1 <sup>xii</sup>	28.12 (4)
O4—Mo1—O2	109.56 (12)	O3—K1—Mo1 <sup>xi</sup>	115.60 (4)
O4—Mo1—O1	106.55 (12)	O3 <sup>xii</sup> —K1—Mo1	50.39 (4)
O4—Mo1—O3	105.29 (12)	O3—K1—Mo1	28.12 (4)
O2—Mo1—K1 <sup>x</sup>	86.26 (8)	O3—K1—O3 <sup>xii</sup>	47.02 (9)
O2—Mo1—K1 <sup>iii</sup>	155.83 (7)	Mo1—O4—K1 <sup>x</sup>	121.94 (12)
O2—Mo1—K1	118.97 (8)	Mo1—O4—K1	114.78 (12)
O1—Mo1—K1 <sup>iii</sup>	43.23 (8)	K1—O4—K1 <sup>x</sup>	121.57 (10)
O1—Mo1—K1	120.81 (8)	Er1 <sup>iv</sup> —O2—Er1 <sup>xvi</sup>	109.14 (10)
O1—Mo1—K1 <sup>x</sup>	95.46 (8)	Mo1—O2—Er1 <sup>xvi</sup>	120.07 (10)
O1—Mo1—O2	118.71 (11)	Mo1—O2—Er1 <sup>iv</sup>	127.56 (11)
O3—Mo1—K1 <sup>iii</sup>	90.50 (8)	Er1—O1—K1 <sup>iii</sup>	112.96 (10)
O3—Mo1—K1 <sup>x</sup>	141.37 (9)	Mo1—O1—Er1	125.03 (13)
O3—Mo1—K1	65.21 (8)	Mo1—O1—K1 <sup>iii</sup>	110.84 (11)
O3—Mo1—O2	111.94 (11)	Er1 <sup>ii</sup> —O3—K1	146.22 (10)
O3—Mo1—O1	103.83 (12)	Mo1—O3—Er1 <sup>ii</sup>	127.06 (13)
Mo1 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	138.837 (12)	Mo1—O3—K1	86.67 (9)

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

### Potassium ytterbium bis(molybdate) (Yb\_Molybdate)

#### Crystal data

KYb(MoO<sub>4</sub>)<sub>2</sub>

$M_r = 532.02$

Orthorhombic, *Pbcn*

Hall symbol:  $-P\ 2n\ 2ab$

$a = 5.0417\ (5)\ \text{\AA}$

$b = 18.3039\ (19)\ \text{\AA}$

$c = 7.8693\ (8)\ \text{\AA}$

$V = 726.20\ (13)\ \text{\AA}^3$

$Z = 4$

$F(000) = 948$

$D_x = 4.866 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 3007 reflections  
 $\theta = 2.2\text{--}29.2^\circ$

$\mu = 16.75 \text{ mm}^{-1}$   
 $T = 273 \text{ K}$   
 Plate, white  
 $0.06 \times 0.06 \times 0.04 \text{ mm}$

*Data collection*

Bruker APEXII CCD  
 diffractometer  
 Radiation source: X-ray tube  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan  
 (SADABS; Krause et al., 2015)  
 $T_{\min} = 0.198$ ,  $T_{\max} = 0.301$   
 8164 measured reflections

978 independent reflections  
 845 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.038$   
 $\theta_{\max} = 29.5^\circ$ ,  $\theta_{\min} = 2.2^\circ$   
 $h = -6 \rightarrow 6$   
 $k = -24 \rightarrow 25$   
 $l = -10 \rightarrow 10$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.022$   
 $wR(F^2) = 0.063$   
 $S = 1.11$   
 978 reflections  
 56 parameters  
 0 restraints

0 constraints  
 Primary atom site location: dual  
 $w = 1/[\sigma^2(F_o^2) + (0.0244P)^2 + 3.0168P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 1.55 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -1.14 \text{ e \AA}^{-3}$

*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.** Reflections were merged by SHELXL according to the crystal class for the calculation of statistics and refinement.

\_reflns\_Friedel\_fraction is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Yb1	0.000000	0.49506 (2)	0.750000	0.01036 (11)
Mo1	0.47826 (7)	0.60027 (2)	0.51395 (5)	0.01066 (12)
K1	0.500000	0.77252 (9)	0.250000	0.0230 (3)
O4	0.6053 (7)	0.68778 (18)	0.5206 (4)	0.0188 (7)
O2	0.7521 (6)	0.53396 (17)	0.4939 (3)	0.0128 (6)
O1	0.2572 (6)	0.59262 (17)	0.6912 (4)	0.0149 (6)
O3	0.2661 (6)	0.59860 (17)	0.3360 (4)	0.0152 (6)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Yb1	0.00810 (17)	0.01404 (17)	0.00894 (16)	0.000	0.00084 (9)	0.000
Mo1	0.0085 (2)	0.0117 (2)	0.0118 (2)	0.00068 (12)	0.00134 (12)	0.00054 (13)
K1	0.0306 (9)	0.0195 (7)	0.0189 (7)	0.000	0.0019 (6)	0.000

O4	0.0212 (18)	0.0179 (17)	0.0173 (16)	-0.0025 (14)	-0.0044 (13)	0.0007 (13)
O2	0.0112 (15)	0.0184 (17)	0.0086 (14)	0.0033 (12)	0.0023 (12)	-0.0010 (11)
O1	0.0131 (14)	0.0175 (15)	0.0143 (15)	-0.0011 (12)	0.0039 (12)	-0.0038 (13)
O3	0.0142 (15)	0.0177 (16)	0.0137 (15)	-0.0024 (13)	0.0006 (12)	0.0003 (12)

*Geometric parameters (Å, °)*

Yb1—Mo1 <sup>i</sup>	3.6018 (5)	Mo1—O2	1.845 (3)
Yb1—Mo1 <sup>ii</sup>	3.6294 (5)	Mo1—O1	1.791 (3)
Yb1—Mo1 <sup>iii</sup>	3.6294 (5)	Mo1—O3	1.762 (3)
Yb1—Mo1	3.6018 (5)	K1—Mo1 <sup>xi</sup>	3.9738 (11)
Yb1—O2 <sup>iv</sup>	2.351 (3)	K1—Mo1 <sup>xii</sup>	3.7772 (14)
Yb1—O2 <sup>v</sup>	2.476 (3)	K1—Mo1 <sup>xiii</sup>	3.8322 (11)
Yb1—O2 <sup>vi</sup>	2.351 (3)	K1—Mo1 <sup>xiv</sup>	3.8322 (11)
Yb1—O2 <sup>vii</sup>	2.476 (3)	K1—Mo1 <sup>xv</sup>	3.9738 (11)
Yb1—O1	2.255 (3)	K1—O4 <sup>xii</sup>	2.687 (3)
Yb1—O1 <sup>i</sup>	2.255 (3)	K1—O4	2.687 (3)
Yb1—O3 <sup>ii</sup>	2.280 (3)	K1—O4 <sup>xi</sup>	2.784 (3)
Yb1—O3 <sup>iii</sup>	2.280 (3)	K1—O4 <sup>xv</sup>	2.784 (3)
Mo1—Yb1 <sup>iii</sup>	3.6294 (5)	K1—O1 <sup>xiii</sup>	2.826 (3)
Mo1—Yb1 <sup>iv</sup>	3.7786 (5)	K1—O1 <sup>xiv</sup>	2.826 (3)
Mo1—Yb1 <sup>viii</sup>	3.7522 (5)	O4—K1 <sup>x</sup>	2.784 (3)
Mo1—K1 <sup>ix</sup>	3.8322 (11)	O2—Yb1 <sup>iv</sup>	2.351 (3)
Mo1—K1 <sup>x</sup>	3.9738 (11)	O2—Yb1 <sup>viii</sup>	2.476 (3)
Mo1—K1	3.7772 (14)	O1—K1 <sup>ix</sup>	2.826 (3)
Mo1—O4	1.726 (3)	O3—Yb1 <sup>iii</sup>	2.280 (3)
Mo1—Yb1—Mo1 <sup>i</sup>	115.356 (17)	O2—Mo1—Yb1	101.15 (10)
Mo1 <sup>i</sup> —Yb1—Mo1 <sup>iii</sup>	96.169 (13)	O2—Mo1—Yb1 <sup>iii</sup>	97.58 (10)
Mo1—Yb1—Mo1 <sup>iii</sup>	113.994 (10)	O2—Mo1—K1 <sup>ix</sup>	155.78 (8)
Mo1—Yb1—Mo1 <sup>ii</sup>	96.168 (13)	O2—Mo1—K1 <sup>x</sup>	85.99 (10)
Mo1 <sup>iii</sup> —Yb1—Mo1 <sup>ii</sup>	122.531 (16)	O2—Mo1—K1	118.71 (10)
Mo1 <sup>i</sup> —Yb1—Mo1 <sup>ii</sup>	113.994 (10)	O1—Mo1—Yb1 <sup>iii</sup>	89.70 (10)
O2 <sup>v</sup> —Yb1—Mo1 <sup>i</sup>	85.88 (7)	O1—Mo1—Yb1 <sup>viii</sup>	90.61 (10)
O2 <sup>iv</sup> —Yb1—Mo1 <sup>ii</sup>	90.31 (8)	O1—Mo1—Yb1 <sup>iv</sup>	145.62 (10)
O2 <sup>vii</sup> —Yb1—Mo1 <sup>i</sup>	76.37 (8)	O1—Mo1—Yb1	30.68 (10)
O2 <sup>iv</sup> —Yb1—Mo1	49.00 (8)	O1—Mo1—K1	120.77 (10)
O2 <sup>vii</sup> —Yb1—Mo1 <sup>ii</sup>	48.48 (8)	O1—Mo1—K1 <sup>x</sup>	95.37 (10)
O2 <sup>iv</sup> —Yb1—Mo1 <sup>iii</sup>	77.14 (8)	O1—Mo1—K1 <sup>ix</sup>	43.81 (10)
O2 <sup>vi</sup> —Yb1—Mo1 <sup>i</sup>	49.00 (8)	O1—Mo1—O2	118.75 (14)
O2 <sup>vi</sup> —Yb1—Mo1	153.85 (8)	O3—Mo1—Yb1	89.67 (10)
O2 <sup>vi</sup> —Yb1—Mo1 <sup>ii</sup>	77.14 (8)	O3—Mo1—Yb1 <sup>viii</sup>	144.09 (11)
O2 <sup>vii</sup> —Yb1—Mo1	85.88 (7)	O3—Mo1—Yb1 <sup>iii</sup>	29.97 (10)
O2 <sup>v</sup> —Yb1—Mo1 <sup>ii</sup>	159.95 (7)	O3—Mo1—Yb1 <sup>iv</sup>	88.72 (10)
O2 <sup>v</sup> —Yb1—Mo1 <sup>iii</sup>	48.48 (8)	O3—Mo1—K1	66.11 (10)
O2 <sup>iv</sup> —Yb1—Mo1 <sup>i</sup>	153.85 (8)	O3—Mo1—K1 <sup>x</sup>	141.56 (11)
O2 <sup>vi</sup> —Yb1—Mo1 <sup>iii</sup>	90.31 (8)	O3—Mo1—K1 <sup>ix</sup>	90.79 (10)
O2 <sup>vii</sup> —Yb1—Mo1 <sup>iii</sup>	159.95 (7)	O3—Mo1—O2	112.01 (14)

O2 <sup>v</sup> —Yb1—Mo1	76.37 (8)	O3—Mo1—O1	103.88 (15)
O2 <sup>iv</sup> —Yb1—O2 <sup>v</sup>	70.66 (12)	Mo1 <sup>xiv</sup> —K1—Mo1 <sup>xv</sup>	80.44 (3)
O2 <sup>iv</sup> —Yb1—O2 <sup>vii</sup>	117.45 (14)	Mo1 <sup>xii</sup> —K1—Mo1	66.83 (3)
O2 <sup>vi</sup> —Yb1—O2 <sup>vii</sup>	70.66 (12)	Mo1—K1—Mo1 <sup>xv</sup>	139.938 (17)
O2 <sup>vi</sup> —Yb1—O2 <sup>v</sup>	117.45 (14)	Mo1 <sup>xiii</sup> —K1—Mo1 <sup>xiv</sup>	105.17 (4)
O2 <sup>vii</sup> —Yb1—O2 <sup>v</sup>	146.58 (15)	Mo1—K1—Mo1 <sup>xi</sup>	102.288 (13)
O2 <sup>iv</sup> —Yb1—O2 <sup>vi</sup>	153.89 (16)	Mo1—K1—Mo1 <sup>xiii</sup>	105.003 (13)
O1—Yb1—Mo1	23.91 (8)	Mo1 <sup>xii</sup> —K1—Mo1 <sup>xv</sup>	102.287 (13)
O1 <sup>i</sup> —Yb1—Mo1 <sup>iii</sup>	96.67 (8)	Mo1 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	139.065 (16)
O1—Yb1—Mo1 <sup>ii</sup>	96.67 (8)	Mo1 <sup>xiii</sup> —K1—Mo1 <sup>xv</sup>	56.91 (2)
O1 <sup>i</sup> —Yb1—Mo1	93.86 (8)	Mo1 <sup>xiv</sup> —K1—Mo1 <sup>xi</sup>	56.91 (2)
O1 <sup>i</sup> —Yb1—Mo1 <sup>ii</sup>	130.19 (8)	Mo1 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	139.938 (17)
O1—Yb1—Mo1 <sup>i</sup>	93.86 (8)	Mo1 <sup>xii</sup> —K1—Mo1 <sup>xiv</sup>	105.003 (13)
O1 <sup>i</sup> —Yb1—Mo1 <sup>i</sup>	23.91 (8)	Mo1 <sup>xiii</sup> —K1—Mo1 <sup>xi</sup>	80.44 (3)
O1—Yb1—Mo1 <sup>iii</sup>	130.19 (8)	Mo1—K1—Mo1 <sup>xiv</sup>	139.065 (16)
O1 <sup>i</sup> —Yb1—O2 <sup>v</sup>	69.45 (10)	Mo1 <sup>xv</sup> —K1—Mo1 <sup>xi</sup>	108.26 (4)
O1—Yb1—O2 <sup>vii</sup>	69.45 (10)	O4—K1—Mo1 <sup>xiv</sup>	149.21 (8)
O1 <sup>i</sup> —Yb1—O2 <sup>iv</sup>	130.70 (11)	O4 <sup>xi</sup> —K1—Mo1 <sup>xi</sup>	21.66 (7)
O1—Yb1—O2 <sup>iv</sup>	72.89 (11)	O4—K1—Mo1 <sup>xiii</sup>	80.93 (7)
O1 <sup>i</sup> —Yb1—O2 <sup>vii</sup>	84.00 (11)	O4 <sup>xi</sup> —K1—Mo1 <sup>xiv</sup>	72.90 (7)
O1—Yb1—O2 <sup>v</sup>	84.00 (11)	O4 <sup>xi</sup> —K1—Mo1 <sup>xii</sup>	126.53 (8)
O1—Yb1—O2 <sup>vi</sup>	130.70 (11)	O4 <sup>xii</sup> —K1—Mo1	87.03 (8)
O1 <sup>i</sup> —Yb1—O2 <sup>vi</sup>	72.89 (11)	O4 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	125.30 (8)
O1—Yb1—O1 <sup>i</sup>	75.25 (16)	O4—K1—Mo1	24.24 (7)
O1 <sup>i</sup> —Yb1—O3 <sup>iii</sup>	108.54 (12)	O4 <sup>xv</sup> —K1—Mo1 <sup>xiii</sup>	72.90 (7)
O1—Yb1—O3 <sup>iii</sup>	150.82 (12)	O4 <sup>xi</sup> —K1—Mo1	80.82 (7)
O1 <sup>i</sup> —Yb1—O3 <sup>ii</sup>	150.82 (12)	O4 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	149.21 (8)
O1—Yb1—O3 <sup>ii</sup>	108.54 (12)	O4 <sup>xv</sup> —K1—Mo1	126.53 (8)
O3 <sup>iii</sup> —Yb1—Mo1	130.01 (7)	O4 <sup>xi</sup> —K1—Mo1 <sup>xiii</sup>	88.68 (8)
O3 <sup>ii</sup> —Yb1—Mo1 <sup>iii</sup>	101.50 (8)	O4 <sup>xii</sup> —K1—Mo1 <sup>xv</sup>	95.65 (7)
O3 <sup>ii</sup> —Yb1—Mo1 <sup>i</sup>	130.01 (7)	O4—K1—Mo1 <sup>xi</sup>	95.65 (7)
O3 <sup>ii</sup> —Yb1—Mo1	99.27 (8)	O4—K1—Mo1 <sup>xv</sup>	125.30 (8)
O3 <sup>iii</sup> —Yb1—Mo1 <sup>i</sup>	99.27 (8)	O4 <sup>xv</sup> —K1—Mo1 <sup>xi</sup>	128.58 (9)
O3 <sup>iii</sup> —Yb1—Mo1 <sup>iii</sup>	22.72 (8)	O4 <sup>xi</sup> —K1—Mo1 <sup>xv</sup>	128.58 (9)
O3 <sup>iii</sup> —Yb1—Mo1 <sup>ii</sup>	101.50 (8)	O4—K1—Mo1 <sup>xii</sup>	87.03 (8)
O3 <sup>ii</sup> —Yb1—Mo1 <sup>ii</sup>	22.72 (8)	O4 <sup>xv</sup> —K1—Mo1 <sup>xv</sup>	21.66 (7)
O3 <sup>iii</sup> —Yb1—O2 <sup>iv</sup>	84.30 (11)	O4 <sup>xv</sup> —K1—Mo1 <sup>xii</sup>	80.82 (7)
O3 <sup>ii</sup> —Yb1—O2 <sup>v</sup>	139.02 (10)	O4 <sup>xii</sup> —K1—Mo1 <sup>xii</sup>	24.25 (7)
O3 <sup>iii</sup> —Yb1—O2 <sup>v</sup>	71.18 (11)	O4 <sup>xv</sup> —K1—Mo1 <sup>xiv</sup>	88.68 (8)
O3 <sup>ii</sup> —Yb1—O2 <sup>iv</sup>	76.09 (10)	O4 <sup>xii</sup> —K1—Mo1 <sup>xiv</sup>	80.93 (7)
O3 <sup>ii</sup> —Yb1—O2 <sup>vi</sup>	84.30 (11)	O4 <sup>xii</sup> —K1—O4 <sup>xi</sup>	121.56 (5)
O3 <sup>iii</sup> —Yb1—O2 <sup>vi</sup>	76.09 (10)	O4 <sup>xii</sup> —K1—O4	109.49 (15)
O3 <sup>iii</sup> —Yb1—O2 <sup>vii</sup>	139.02 (10)	O4—K1—O4 <sup>xv</sup>	121.56 (5)
O3 <sup>ii</sup> —Yb1—O2 <sup>vii</sup>	71.18 (11)	O4—K1—O4 <sup>xi</sup>	77.17 (8)
O3 <sup>iii</sup> —Yb1—O3 <sup>ii</sup>	82.48 (16)	O4 <sup>xii</sup> —K1—O4 <sup>xv</sup>	77.17 (8)
Yb1 <sup>viii</sup> —Mo1—Yb1 <sup>iv</sup>	63.070 (9)	O4 <sup>xi</sup> —K1—O4 <sup>xv</sup>	149.74 (15)
Yb1 <sup>iii</sup> —Mo1—Yb1 <sup>viii</sup>	120.155 (13)	O4 <sup>xv</sup> —K1—O1 <sup>xiv</sup>	89.65 (10)
Yb1 <sup>iii</sup> —Mo1—Yb1 <sup>iv</sup>	85.752 (11)	O4 <sup>xv</sup> —K1—O1 <sup>xiii</sup>	63.27 (9)

Yb1—Mo1—Yb1 <sup>iii</sup>	66.007 (10)	O4 <sup>xii</sup> —K1—O1 <sup>xiv</sup>	106.49 (10)
Yb1—Mo1—Yb1 <sup>viii</sup>	86.536 (12)	O4 <sup>xi</sup> —K1—O1 <sup>xiii</sup>	89.65 (10)
Yb1—Mo1—Yb1 <sup>iv</sup>	120.176 (13)	O4 <sup>xi</sup> —K1—O1 <sup>xiv</sup>	63.27 (9)
Yb1—Mo1—K1	138.532 (12)	O4 <sup>xii</sup> —K1—O1 <sup>xiii</sup>	136.42 (10)
Yb1 <sup>iii</sup> —Mo1—K1 <sup>x</sup>	171.48 (2)	O4—K1—O1 <sup>xiii</sup>	106.49 (10)
Yb1 <sup>iv</sup> —Mo1—K1 <sup>ix</sup>	170.07 (2)	O4—K1—O1 <sup>xiv</sup>	136.42 (10)
Yb1 <sup>iii</sup> —Mo1—K1	96.075 (15)	O1 <sup>xiv</sup> —K1—Mo1 <sup>xiv</sup>	26.02 (6)
Yb1 <sup>viii</sup> —Mo1—K1 <sup>ix</sup>	120.864 (13)	O1 <sup>xiv</sup> —K1—Mo1 <sup>xiii</sup>	80.64 (7)
Yb1 <sup>viii</sup> —Mo1—K1	132.869 (12)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xi</sup>	73.47 (7)
Yb1—Mo1—K1 <sup>x</sup>	121.021 (14)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xiv</sup>	80.64 (7)
Yb1 <sup>iv</sup> —Mo1—K1 <sup>x</sup>	93.824 (14)	O1 <sup>xiii</sup> —K1—Mo1	130.72 (6)
Yb1 <sup>viii</sup> —Mo1—K1 <sup>x</sup>	66.75 (2)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xii</sup>	143.68 (7)
Yb1—Mo1—K1 <sup>ix</sup>	69.74 (2)	O1 <sup>xiv</sup> —K1—Mo1 <sup>xi</sup>	42.36 (7)
Yb1 <sup>iii</sup> —Mo1—K1 <sup>ix</sup>	98.712 (15)	O1 <sup>xiv</sup> —K1—Mo1 <sup>xii</sup>	130.72 (6)
K1—Mo1—Yb1 <sup>iv</sup>	93.608 (16)	O1 <sup>xiv</sup> —K1—Mo1 <sup>xv</sup>	73.47 (7)
K1—Mo1—K1 <sup>ix</sup>	77.153 (12)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xv</sup>	42.36 (7)
K1—Mo1—K1 <sup>x</sup>	75.448 (12)	O1 <sup>xiv</sup> —K1—Mo1	143.68 (7)
K1 <sup>ix</sup> —Mo1—K1 <sup>x</sup>	80.44 (3)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xiii</sup>	26.02 (6)
O4—Mo1—Yb1	136.81 (11)	O1 <sup>xiii</sup> —K1—O1 <sup>xiv</sup>	58.30 (13)
O4—Mo1—Yb1 <sup>viii</sup>	101.62 (11)	Mo1—O4—K1	116.01 (15)
O4—Mo1—Yb1 <sup>iv</sup>	100.77 (12)	Mo1—O4—K1 <sup>x</sup>	121.81 (16)
O4—Mo1—Yb1 <sup>iii</sup>	135.23 (11)	K1—O4—K1 <sup>x</sup>	120.30 (12)
O4—Mo1—K1 <sup>x</sup>	36.53 (11)	Yb1 <sup>iv</sup> —O2—Yb1 <sup>viii</sup>	109.33 (12)
O4—Mo1—K1 <sup>ix</sup>	69.81 (12)	Mo1—O2—Yb1 <sup>iv</sup>	128.04 (14)
O4—Mo1—K1	39.74 (10)	Mo1—O2—Yb1 <sup>viii</sup>	119.82 (13)
O4—Mo1—O2	109.61 (16)	Yb1—O1—K1 <sup>ix</sup>	113.22 (12)
O4—Mo1—O1	106.25 (15)	Mo1—O1—Yb1	125.42 (16)
O4—Mo1—O3	105.38 (15)	Mo1—O1—K1 <sup>ix</sup>	110.17 (14)
O2—Mo1—Yb1 <sup>iv</sup>	29.34 (8)	Mo1—O3—Yb1 <sup>iii</sup>	127.31 (16)
O2—Mo1—Yb1 <sup>viii</sup>	34.93 (8)		

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

### Potassium lutetium bis(molybdate) (Lu\_Molybdate)

#### Crystal data

KLu(MoO<sub>4</sub>)<sub>2</sub>

$M_r = 533.95$

Orthorhombic, *Pbcn*

Hall symbol:  $-P\ 2n\ 2ab$

$a = 5.0292$  (2) Å

$b = 18.2519$  (10) Å

$c = 7.8174$  (4) Å

$V = 717.58$  (6) Å<sup>3</sup>

$Z = 4$

$F(000) = 952$

$D_x = 4.942$  Mg m<sup>-3</sup>

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

Cell parameters from 6634 reflections

$\theta = 3.4\text{--}30.5^\circ$

$\mu = 17.68$  mm<sup>-1</sup>

$T = 100$  K

Plate, white

$0.44 \times 0.14 \times 0.04$  mm



*Data collection*

Rigaku XtaLAB Synergy-S, HyPix  
diffractometer

Radiation source: micro-focus sealed X-ray tube

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: multi-scan  
(CrysAlisPro; Rigaku OD, 2019)

$T_{\min} = 0.240$ ,  $T_{\max} = 1.000$

19857 measured reflections

1105 independent reflections

1004 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.087$

$\theta_{\max} = 30.5^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -7 \rightarrow 7$

$k = -26 \rightarrow 26$

$l = -11 \rightarrow 11$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.027$

$wR(F^2) = 0.077$

$S = 1.17$

1105 reflections

57 parameters

0 restraints

0 constraints

Primary atom site location: dual

$w = 1/[\sigma^2(F_o^2) + (0.0305P)^2 + 4.6788P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 2.05 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -3.03 \text{ e } \text{\AA}^{-3}$

*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.** Reflections were merged by SHELXL according to the crystal class for the calculation of statistics and refinement.

$\text{\_reflns\_Friedel\_fraction}$  is defined as the number of unique Friedel pairs measured divided by the number that would be possible theoretically, ignoring centric projections and systematic absences.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Lu1	1.000000	0.50463 (2)	0.750000	0.00674 (13)
Mo1	0.52103 (8)	0.39975 (3)	0.51329 (5)	0.00699 (13)
K1	0.500000	0.22690 (9)	0.250000	0.0116 (3)
O4	0.3935 (7)	0.31279 (19)	0.5200 (4)	0.0119 (7)
O2	0.2476 (7)	0.4667 (2)	0.4950 (4)	0.0098 (7)
O1	0.7429 (6)	0.40718 (19)	0.6920 (4)	0.0101 (6)
O3	0.7347 (6)	0.40152 (19)	0.3343 (4)	0.0098 (6)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Lu1	0.00628 (18)	0.0100 (2)	0.00389 (17)	0.000	0.00006 (8)	0.000
Mo1	0.0067 (2)	0.0091 (2)	0.0051 (2)	0.00028 (12)	-0.00024 (11)	-0.00031 (13)
K1	0.0142 (7)	0.0132 (8)	0.0073 (6)	0.000	-0.0006 (4)	0.000
O4	0.0128 (17)	0.0141 (17)	0.0087 (14)	-0.0011 (13)	0.0006 (12)	-0.0018 (12)
O2	0.0079 (15)	0.0168 (18)	0.0046 (14)	0.0006 (13)	-0.0051 (11)	0.0004 (11)
O1	0.0100 (14)	0.0130 (16)	0.0074 (14)	-0.0011 (13)	-0.0028 (12)	0.0009 (12)
O3	0.0118 (15)	0.0126 (16)	0.0051 (13)	0.0008 (13)	0.0002 (11)	-0.0001 (11)

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

Lu1—Lu1 <sup>i</sup>	3.9123 (2)	Mo1—O3	1.765 (3)
Lu1—Lu1 <sup>ii</sup>	3.9123 (2)	K1—Mo1 <sup>xi</sup>	3.9540 (11)
Lu1—K1 <sup>iii</sup>	4.2257 (17)	K1—Mo1 <sup>xii</sup>	3.7684 (14)
Lu1—O2 <sup>iv</sup>	2.344 (3)	K1—Mo1 <sup>xiii</sup>	3.8171 (11)
Lu1—O2 <sup>v</sup>	2.344 (3)	K1—Mo1 <sup>xiv</sup>	3.8171 (11)
Lu1—O2 <sup>vi</sup>	2.450 (3)	K1—Mo1 <sup>xv</sup>	3.9540 (11)
Lu1—O2 <sup>vii</sup>	2.450 (3)	K1—O4	2.683 (3)
Lu1—O1	2.245 (3)	K1—O4 <sup>xii</sup>	2.683 (3)
Lu1—O1 <sup>viii</sup>	2.245 (3)	K1—O4 <sup>xv</sup>	2.770 (3)
Lu1—O3 <sup>ix</sup>	2.269 (3)	K1—O4 <sup>xi</sup>	2.770 (3)
Lu1—O3 <sup>ii</sup>	2.269 (3)	K1—O1 <sup>xiii</sup>	2.805 (4)
Mo1—K1	3.7684 (14)	K1—O1 <sup>xiv</sup>	2.805 (4)
Mo1—K1 <sup>x</sup>	3.9540 (11)	O4—K1 <sup>x</sup>	2.770 (3)
Mo1—K1 <sup>iii</sup>	3.8171 (11)	O2—Lu1 <sup>iv</sup>	2.344 (3)
Mo1—O4	1.713 (4)	O2—Lu1 <sup>xvi</sup>	2.450 (3)
Mo1—O2	1.845 (4)	O1—K1 <sup>iii</sup>	2.805 (4)
Mo1—O1	1.793 (3)	O3—Lu1 <sup>ii</sup>	2.269 (3)
Lu1 <sup>ii</sup> —Lu1—Lu1 <sup>i</sup>	175.051 (17)	Mo1 <sup>xiv</sup> —K1—Mo1 <sup>xv</sup>	80.64 (3)
Lu1 <sup>ii</sup> —Lu1—K1 <sup>iii</sup>	87.526 (9)	Mo1—K1—Mo1 <sup>xv</sup>	139.788 (18)
Lu1 <sup>i</sup> —Lu1—K1 <sup>iii</sup>	87.526 (9)	Mo1 <sup>xiii</sup> —K1—Mo1 <sup>xiv</sup>	105.46 (4)
O2 <sup>vii</sup> —Lu1—Lu1 <sup>ii</sup>	34.41 (8)	Mo1 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	139.788 (18)
O2 <sup>vii</sup> —Lu1—Lu1 <sup>i</sup>	143.18 (8)	Mo1 <sup>xii</sup> —K1—Mo1	66.31 (3)
O2 <sup>iv</sup> —Lu1—Lu1 <sup>ii</sup>	36.22 (8)	Mo1 <sup>xiii</sup> —K1—Mo1 <sup>xi</sup>	80.64 (3)
O2 <sup>v</sup> —Lu1—Lu1 <sup>ii</sup>	145.70 (8)	Mo1 <sup>xii</sup> —K1—Mo1 <sup>xv</sup>	102.429 (13)
O2 <sup>vi</sup> —Lu1—Lu1 <sup>i</sup>	34.41 (8)	Mo1—K1—Mo1 <sup>xiii</sup>	105.066 (13)
O2 <sup>v</sup> —Lu1—Lu1 <sup>i</sup>	36.22 (8)	Mo1 <sup>xiii</sup> —K1—Mo1 <sup>xv</sup>	56.95 (2)
O2 <sup>iv</sup> —Lu1—Lu1 <sup>i</sup>	145.70 (8)	Mo1 <sup>xii</sup> —K1—Mo1 <sup>xiv</sup>	105.066 (14)
O2 <sup>vi</sup> —Lu1—Lu1 <sup>ii</sup>	143.18 (8)	Mo1—K1—Mo1 <sup>xiv</sup>	138.944 (16)
O2 <sup>vii</sup> —Lu1—K1 <sup>iii</sup>	73.58 (8)	Mo1—K1—Mo1 <sup>xi</sup>	102.429 (13)
O2 <sup>iv</sup> —Lu1—K1 <sup>iii</sup>	102.92 (9)	Mo1 <sup>xiv</sup> —K1—Mo1 <sup>xi</sup>	56.95 (2)
O2 <sup>v</sup> —Lu1—K1 <sup>iii</sup>	102.92 (9)	Mo1 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	138.944 (16)
O2 <sup>vi</sup> —Lu1—K1 <sup>iii</sup>	73.58 (8)	Mo1 <sup>xv</sup> —K1—Mo1 <sup>xi</sup>	108.45 (4)
O2 <sup>iv</sup> —Lu1—O2 <sup>vi</sup>	117.26 (15)	O4 <sup>xii</sup> —K1—Mo1 <sup>xiv</sup>	81.17 (8)
O2 <sup>v</sup> —Lu1—O2 <sup>vii</sup>	117.26 (15)	O4 <sup>xv</sup> —K1—Mo1 <sup>xi</sup>	128.63 (9)
O2 <sup>v</sup> —Lu1—O2 <sup>iv</sup>	154.16 (18)	O4 <sup>xv</sup> —K1—Mo1 <sup>xii</sup>	81.05 (7)
O2 <sup>v</sup> —Lu1—O2 <sup>vi</sup>	70.63 (14)	O4 <sup>xv</sup> —K1—Mo1 <sup>xiv</sup>	88.73 (8)
O2 <sup>vii</sup> —Lu1—O2 <sup>vi</sup>	147.15 (17)	O4—K1—Mo1	24.06 (8)
O2 <sup>iv</sup> —Lu1—O2 <sup>vii</sup>	70.63 (14)	O4 <sup>xi</sup> —K1—Mo1 <sup>xiv</sup>	72.87 (7)
O1—Lu1—Lu1 <sup>i</sup>	99.65 (8)	O4—K1—Mo1 <sup>xi</sup>	96.06 (8)
O1—Lu1—Lu1 <sup>ii</sup>	76.35 (8)	O4 <sup>xii</sup> —K1—Mo1	86.27 (8)
O1 <sup>viii</sup> —Lu1—Lu1 <sup>i</sup>	76.35 (8)	O4 <sup>xi</sup> —K1—Mo1 <sup>xiii</sup>	88.73 (8)
O1 <sup>viii</sup> —Lu1—Lu1 <sup>ii</sup>	99.65 (8)	O4 <sup>xv</sup> —K1—Mo1	126.40 (8)
O1 <sup>viii</sup> —Lu1—K1 <sup>iii</sup>	37.61 (8)	O4 <sup>xii</sup> —K1—Mo1 <sup>xii</sup>	24.06 (8)
O1—Lu1—K1 <sup>iii</sup>	37.61 (8)	O4 <sup>xi</sup> —K1—Mo1	81.05 (7)
O1 <sup>viii</sup> —Lu1—O2 <sup>iv</sup>	130.40 (12)	O4 <sup>xv</sup> —K1—Mo1 <sup>xiii</sup>	72.87 (7)

O1—Lu1—O2 <sup>vi</sup>	69.37 (11)	O4—K1—Mo1 <sup>xv</sup>	125.27 (8)
O1 <sup>viii</sup> —Lu1—O2 <sup>vii</sup>	69.37 (11)	O4 <sup>xii</sup> —K1—Mo1 <sup>xi</sup>	125.27 (8)
O1—Lu1—O2 <sup>iv</sup>	72.91 (12)	O4 <sup>xii</sup> —K1—Mo1 <sup>xv</sup>	96.06 (8)
O1 <sup>viii</sup> —Lu1—O2 <sup>v</sup>	72.91 (12)	O4 <sup>xi</sup> —K1—Mo1 <sup>xi</sup>	21.55 (7)
O1—Lu1—O2 <sup>vii</sup>	84.51 (12)	O4 <sup>xv</sup> —K1—Mo1 <sup>xv</sup>	21.55 (7)
O1—Lu1—O2 <sup>v</sup>	130.40 (12)	O4 <sup>xi</sup> —K1—Mo1 <sup>xii</sup>	126.40 (8)
O1 <sup>viii</sup> —Lu1—O2 <sup>vii</sup>	84.51 (12)	O4 <sup>xi</sup> —K1—Mo1 <sup>xv</sup>	128.63 (9)
O1 <sup>viii</sup> —Lu1—O1	75.22 (17)	O4—K1—Mo1 <sup>xiv</sup>	149.44 (8)
O1—Lu1—O3 <sup>ix</sup>	108.54 (13)	O4—K1—Mo1 <sup>xii</sup>	86.27 (8)
O1 <sup>viii</sup> —Lu1—O3 <sup>ix</sup>	151.41 (12)	O4 <sup>xii</sup> —K1—Mo1 <sup>xiii</sup>	149.44 (8)
O1 <sup>viii</sup> —Lu1—O3 <sup>ii</sup>	108.54 (13)	O4—K1—Mo1 <sup>xiii</sup>	81.17 (8)
O1—Lu1—O3 <sup>ii</sup>	151.41 (12)	O4—K1—O4 <sup>xv</sup>	121.38 (5)
O3 <sup>ii</sup> —Lu1—Lu1 <sup>ii</sup>	75.08 (8)	O4—K1—O4 <sup>xii</sup>	108.51 (16)
O3 <sup>ix</sup> —Lu1—Lu1 <sup>ii</sup>	108.82 (8)	O4 <sup>xii</sup> —K1—O4 <sup>xi</sup>	121.38 (5)
O3 <sup>ii</sup> —Lu1—Lu1 <sup>i</sup>	108.82 (8)	O4 <sup>xii</sup> —K1—O4 <sup>xv</sup>	77.57 (9)
O3 <sup>ix</sup> —Lu1—Lu1 <sup>i</sup>	75.08 (8)	O4—K1—O4 <sup>xi</sup>	77.57 (9)
O3 <sup>ii</sup> —Lu1—K1 <sup>iii</sup>	139.02 (8)	O4 <sup>xv</sup> —K1—O4 <sup>xi</sup>	149.68 (16)
O3 <sup>ix</sup> —Lu1—K1 <sup>iii</sup>	139.02 (8)	O4 <sup>xi</sup> —K1—O1 <sup>xiv</sup>	63.09 (10)
O3 <sup>ii</sup> —Lu1—O2 <sup>vii</sup>	71.24 (12)	O4 <sup>xi</sup> —K1—O1 <sup>xiii</sup>	89.78 (11)
O3 <sup>ii</sup> —Lu1—O2 <sup>iv</sup>	84.63 (12)	O4—K1—O1 <sup>xiv</sup>	136.80 (10)
O3 <sup>ix</sup> —Lu1—O2 <sup>v</sup>	84.63 (12)	O4 <sup>xv</sup> —K1—O1 <sup>xiii</sup>	63.09 (10)
O3 <sup>ii</sup> —Lu1—O2 <sup>v</sup>	75.88 (11)	O4 <sup>xv</sup> —K1—O1 <sup>xiv</sup>	89.78 (11)
O3 <sup>ix</sup> —Lu1—O2 <sup>vii</sup>	138.46 (11)	O4—K1—O1 <sup>xiii</sup>	106.89 (10)
O3 <sup>ii</sup> —Lu1—O2 <sup>vi</sup>	138.46 (11)	O4 <sup>xii</sup> —K1—O1 <sup>xiii</sup>	136.80 (10)
O3 <sup>ix</sup> —Lu1—O2 <sup>vi</sup>	71.24 (12)	O4 <sup>xii</sup> —K1—O1 <sup>xiv</sup>	106.89 (10)
O3 <sup>ix</sup> —Lu1—O2 <sup>iv</sup>	75.88 (11)	O1 <sup>xiv</sup> —K1—Mo1 <sup>xiv</sup>	26.14 (7)
O3 <sup>ii</sup> —Lu1—O3 <sup>ix</sup>	81.96 (17)	O1 <sup>xiv</sup> —K1—Mo1 <sup>xiii</sup>	80.84 (7)
K1—Mo1—K1 <sup>x</sup>	75.379 (13)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xi</sup>	73.72 (7)
K1—Mo1—K1 <sup>iii</sup>	77.027 (12)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xiv</sup>	80.84 (7)
K1 <sup>iii</sup> —Mo1—K1 <sup>x</sup>	80.64 (3)	O1 <sup>xiii</sup> —K1—Mo1	130.93 (6)
O4—Mo1—K1	39.69 (11)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xii</sup>	143.70 (7)
O4—Mo1—K1 <sup>x</sup>	36.45 (11)	O1 <sup>xiv</sup> —K1—Mo1 <sup>xi</sup>	42.27 (7)
O4—Mo1—K1 <sup>iii</sup>	70.14 (12)	O1 <sup>xiv</sup> —K1—Mo1 <sup>xii</sup>	130.93 (6)
O4—Mo1—O2	109.68 (17)	O1 <sup>xiv</sup> —K1—Mo1 <sup>xv</sup>	73.72 (7)
O4—Mo1—O1	106.21 (16)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xv</sup>	42.27 (7)
O4—Mo1—O3	105.63 (16)	O1 <sup>xiv</sup> —K1—Mo1	143.70 (7)
O2—Mo1—K1 <sup>iii</sup>	155.36 (9)	O1 <sup>xiii</sup> —K1—Mo1 <sup>xiii</sup>	26.14 (7)
O2—Mo1—K1	119.41 (10)	O1 <sup>xiii</sup> —K1—O1 <sup>xiv</sup>	58.48 (14)
O2—Mo1—K1 <sup>x</sup>	85.95 (11)	Mo1—O4—K1	116.25 (16)
O1—Mo1—K1 <sup>iii</sup>	43.56 (11)	Mo1—O4—K1 <sup>x</sup>	122.00 (16)
O1—Mo1—K1 <sup>x</sup>	95.28 (11)	K1—O4—K1 <sup>x</sup>	120.03 (13)
O1—Mo1—K1	120.42 (11)	Lu1 <sup>iv</sup> —O2—Lu1 <sup>xvi</sup>	109.37 (14)
O1—Mo1—O2	118.31 (14)	Mo1—O2—Lu1 <sup>iv</sup>	127.40 (15)
O3—Mo1—K1 <sup>x</sup>	141.76 (12)	Mo1—O2—Lu1 <sup>xvi</sup>	120.19 (14)
O3—Mo1—K1 <sup>iii</sup>	90.65 (11)	Lu1—O1—K1 <sup>iii</sup>	113.15 (12)
O3—Mo1—K1	66.38 (11)	Mo1—O1—Lu1	125.15 (17)

---

O3—Mo1—O2	112.35 (14)	Mo1—O1—K1 <sup>iii</sup>	110.29 (15)
O3—Mo1—O1	103.74 (16)	Mo1—O3—Lu1 <sup>ii</sup>	127.02 (17)

---

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