



**Keywords:** crystal structure; molybdate; phosphate; gadolinium; triangular dodecahedron.

**CCDC reference:** 2322198

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

# Crystal structure of a layered phosphate molybdate $K_2Gd(PO_4)(MoO_4)$

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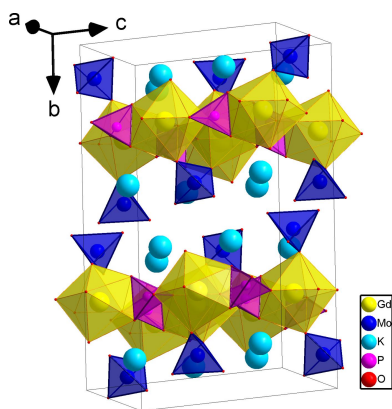
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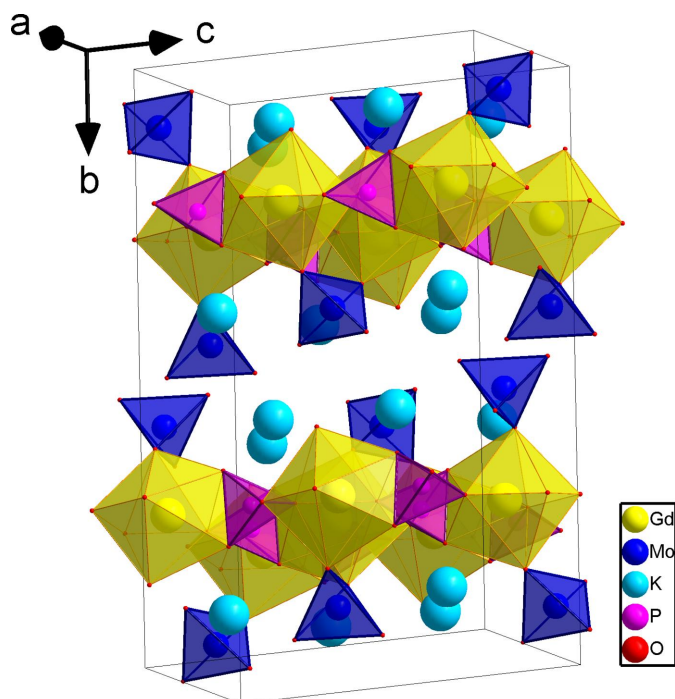
The title compound dipotassium gadolinium(III) phosphate(V) molybdate(VI),  $K_2Gd(PO_4)(MoO_4)$ , was synthesized from a high-temperature melt starting from  $GdF_3$  as a source of gadolinium. Its structure is isotypic with other  $M^I_2M^{III}(M^{VI}O_4)(PO_4)$  compounds, where  $M^I = Na, K$  or  $Cs$ , and  $M^{III} =$  rare-earth cation,  $M^{VI} = Mo$  or  $W$ . The three-dimensional framework is built up from  $[Gd(PO_4)(MoO_4)]$  anionic sheets, which are organized by adhesion of  $[GdPO_4]$  layers and  $[MoO_4]$  tetrahedra stacked above and below these layers. The interstitial space is occupied by K cations having eightfold oxygen coordination. The polyhedron of  $GdO_8$  was estimated to be a triangular dodecahedron by the continuous shape measurement method.

## 1. Chemical context

Layered phosphate(V) molybdates(VI)  $M^I_2M^{III}(M^{VI}O_4)(PO_4)$  comprising an alkali metal and a rare-earth metal  $M^{III}$  such as Sm (Zhao *et al.*, 2009), Eu (Terebilenko *et al.*, 2022), Y (Zhang *et al.*, 2016) or Bi (Grigorjevaite *et al.*, 2020) are considered to be promising luminescent materials (Guo *et al.*, 2019). The initial structural models of this group of compounds,  $Na_2Y(PO_4)(MoO_4)$ , were monoclinic, space group  $C2/c$ , as described by Ben Amara & Dabbabi (1987). Subsequent work determined that the material crystallizes in an orthorhombic system, space group  $Ibca$  (Marsh, 1987). The discovery of  $K_2Bi(PO_4)(MoO_4)$  by Zatovsky *et al.* (2006) opened a new group of luminescent materials that are isostructural to  $Na_2Y(PO_4)(MoO_4)$  and have high color purity and quantum yield (Grigorjevaite & Katelnikovas, 2016).

In the case of  $Rb_2Bi(PO_4)(MoO_4):Eu^{3+}$  powders, the quantum efficiency has been shown to reach *ca* 100% for the  $Rb_2Bi_{0.5}Eu_{0.5}(PO_4)(MoO_4)$  phosphor (Grigorjevaite & Katelnikovas, 2016). High color purity and emission spectra peculiarities make these compounds attractive for red-component design in near-UV LED-driven solid-state light sources (Zozulia *et al.*, 2023). One of the main disadvantages of these luminescence hosts is the relatively high activator content needed (from 50 to 75%) to reach a high quantum efficiency (Grigorjevaite & Katelnikovas, 2016). Different strategies have been applied to improve the luminescence performance and lower the luminescent dopant content, including rare-earth co-doping (Naidu *et al.*, 2012) and anion modifications (Guo *et al.*, 2019). To tune the luminescence properties of these phosphors, the quest for new representatives of this group of compounds can shed light on the development of new phosphors based on them.

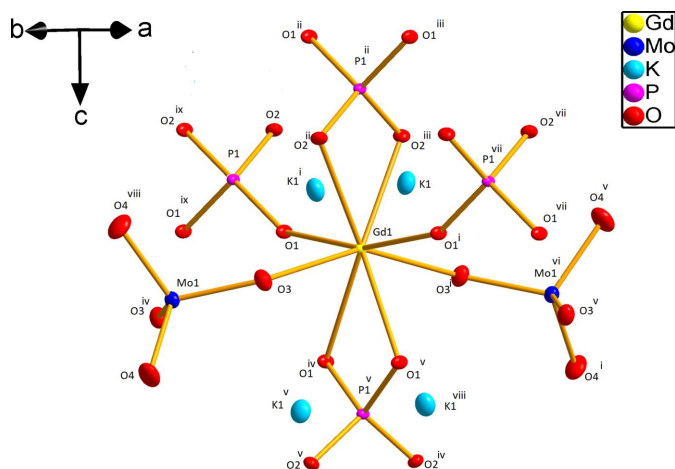




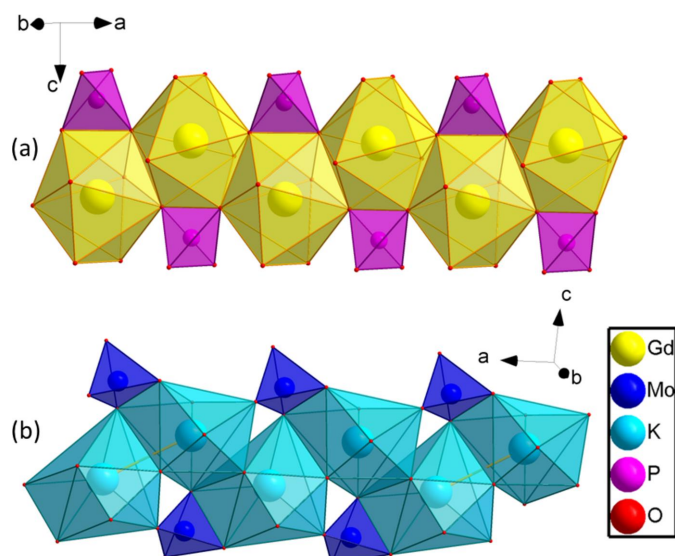
**Figure 1**  
Representation of the unit-cell content of  $\text{K}_2\text{Gd}(\text{PO}_4)(\text{MoO}_4)$ .

## 2. Structural commentary

The three-dimensional framework of the title compound is organized by linking together slightly distorted  $\text{GdO}_8$  dodecahedra with non-condensed phosphate and molybdate tetrahedra (Fig. 1). These moieties are arranged into layers perpendicular to the  $[010]$  direction with each phosphate layer being followed by two molybdate layers. In this packing, the



**Figure 2**  
Representation of the coordination environment of gadolinium atoms in  $\text{K}_2\text{Gd}(\text{PO}_4)(\text{MoO}_4)$ . Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i)  $2 - x, \frac{1}{2} - y, z$ ; (ii)  $\frac{3}{2} - x, \frac{1}{2} - y, \frac{1}{2} - z$ ; (iii)  $\frac{1}{2} + x, y, \frac{1}{2} - z$ ; (iv)  $\frac{3}{2} - x, y, 1 - z$ ; (v)  $\frac{1}{2} + x, \frac{1}{2} - x, 1 - z$ ; (vii)  $1 + x, y, z$ ; (viii)  $\frac{3}{2} - x, y, 1 - z$ ; (ix)  $1 - x, \frac{1}{2} - y, z$ .]



**Figure 3**  
Zigzag chains build up from (a)  $\text{GdO}_8$  and (b)  $\text{KO}_8$  polyhedra

gadolinium and potassium cations are eightfold coordinated by oxygen (Fig. 2) and ordered into zigzag chains (Fig. 3).

Each Gd cation is surrounded by two molybdate tetrahedra and four phosphate tetrahedra; two of the phosphate groups are coordinated in a bidentate manner (Fig. 2). The Gd—O bond lengths lie in the range 2.314 (3)–2.453 (3) Å. Among the Gd—O bond lengths, those corresponding to the bidentately coordinated phosphate groups are the longest [2.427 (2) and 2.453 (2) Å]. The chains built up from  $\text{GdO}_8$  polyhedra are interlinked by phosphate moieties into  $[\text{GdPO}_4]$  layers propagating in the  $ac$  plane. The nearest Gd···Gd distance within a zigzag chain is 3.9332 (2) Å.  $[\text{Gd}(\text{PO}_4)(\text{MoO}_4)]$  nets are formed by adhesion of  $[\text{GdPO}_4]$  layers and  $\text{MoO}_4$  tetrahedra above and below these layers (Fig. 1).

Both the phosphate and molybdate tetrahedra have an almost regular geometry with typical bond lengths. The central atoms of the  $\text{GdO}_8$ ,  $\text{MoO}_4$  and  $\text{PO}_4$  polyhedra are located on a twofold axis. The potassium cation resides inside the interlayer space having eightfold coordination, as has been found for other potassium-based representatives of this family (Zatovsky *et al.*, 2006). Importantly, there is a difference in the nearest oxygen coordination of sodium- and potassium-based frameworks. In case of  $\text{Na}_2\text{Y}(\text{PO}_4)(\text{WO}_4)$ , the  $\text{NaO}_6$  sodium environment is described as an effective 3 + 3 coordination indicating a relatively large void between two successive  $[\text{Y}(\text{PO}_4)(\text{WO}_4)]$  layers (Daub *et al.*, 2012).

## 3. Coordination environment calculations

The distortions of the coordination environment of gadolinium, potassium, phosphorus and molybdenum have been calculated by the continuous shape measurement method with the *Shape 2.1* program (Llunell *et al.*, 2013). The shape measurements in this work are taken from normalized coordination polyhedra (Alvarez, 2021). There are two types of polyhedra within the structure studied: two are tetrahedral,

**Table 1**  
Experimental details.

Crystal data	
Chemical formula	K <sub>2</sub> Gd(PO <sub>4</sub> )(MoO <sub>4</sub> )
<i>M<sub>r</sub></i>	490.36
Crystal system, space group	Orthorhombic, <i>Ibca</i>
Temperature (K)	200
<i>a</i> , <i>b</i> , <i>c</i> (Å)	6.9527 (2), 19.7112 (6), 12.2466 (3)
<i>V</i> (Å <sup>3</sup> )	1678.35 (8)
<i>Z</i>	8
Radiation type	Mo <i>K</i> α
<i>μ</i> (mm <sup>-1</sup> )	10.52
Crystal size (mm)	0.10 × 0.08 × 0.02
Data collection	
Diffractometer	XtaLAB Synergy, Dualflex, HyPix
Absorption correction	Gaussian ( <i>CrysAlis PRO</i> ; Rigaku OD, 2020)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.422, 1.000
No. of measured, independent and observed [ <i>I</i> > 2σ( <i>I</i> )] reflections	6547, 1079, 999
<i>R<sub>int</sub></i>	0.026
(sin θ/λ) <sub>max</sub> (Å <sup>-1</sup> )	0.707
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.017, 0.045, 1.13
No. of reflections	1079
No. of parameters	61
Δρ <sub>max</sub> , Δρ <sub>min</sub> (e Å <sup>-3</sup> )	1.53, -0.64

Computer programs: *CrysAlis PRO* (Rigaku OD, 2020), *SHELXT2018/2* (Sheldrick, 2015a), *SHELXL2018/3* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

namely, MoO<sub>4</sub> and PO<sub>4</sub> and two are eightfold coordinated, KO<sub>8</sub> and GdO<sub>8</sub>. The shape measurements of a set of atoms with respect to a reference shape (*e.g.*, the tetrahedron, abbreviated T-4 by IUPAC) calibrates the overall distance of the atoms to the vertices of the tetrahedral shape in the same position. Thus, a zero-shape measurement for a set of atoms indicates that the polyhedron has exactly the reference shape, expressed as *S*(T-4) = 0.00 for an ideal tetrahedron. Increasing values of the shape measurement will be found for more distorted polyhedra, in other words, these values are essentially spatial distance minima of the central atom from a minimization polyhedral fitting procedure. For the title compound, the MoO<sub>4</sub> tetrahedron has minor distortions, as indicated by the value of *S* of 0.053. In contrast, the PO<sub>4</sub> tetrahedron reveals more severe deviations, having *C*<sub>2</sub> site symmetry with a calculated value of *S* = 0.238.

In case of GdO<sub>8</sub>, the lowest value of *S* of 2.725 was obtained for a triangular dodecahedron (TDD-8) (Casanova *et al.* 2005) and KO<sub>8</sub> is best described as a biaugmented trigonal prism, as indicated by the value of *S* of 3.999. Thus, the GdO<sub>8</sub> polyhedron in K<sub>2</sub>Bi(PO<sub>4</sub>)(MoO<sub>4</sub>) is found to be a triangular dodecahedron (TDD-8), as has also been observed for K<sub>2</sub>Eu(PO<sub>4</sub>)(WO<sub>4</sub>) (Terebilenko *et al.*, 2022).

#### 4. Synthesis and crystallization

Single crystals of the title compound were grown from molten salts 7K<sub>2</sub>Mo<sub>2</sub>O<sub>7</sub>–3K<sub>4</sub>P<sub>2</sub>O<sub>7</sub> containing 5% mol of GdF<sub>3</sub>. A mixture of K<sub>2</sub>Mo<sub>2</sub>O<sub>7</sub> and K<sub>4</sub>P<sub>2</sub>O<sub>7</sub> was heated in a platinum

crucible up to 1273 K. After melting, 5% mol of GdF<sub>3</sub> was added to the initial molten salts under stirring. The mixture was then held at this temperature for 2 h and cooled down to room temperature at a rate of 50 K h<sup>-1</sup>. The solidified melt was leached out with warm water to dissolve the superfluous flux. The final product consisted of colourless plates. The yield was 64% by Gd.

#### 5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

#### Acknowledgements

The authors are grateful to Sergiu G. Shova from the "Petru Poni" Institute of Macromolecular Chemistry for the diffraction data collection.

#### Funding information

Funding for this research was provided by: National Research Foundation of Ukraine (grant No. 2022.01/0168).

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## supporting information

*Acta Cryst.* (2024). E80, 117-119 [https://doi.org/10.1107/S2056989023011106]

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### Computing details

#### Dipotassium gadolinium(III) phosphate(V) molybdate(VI)

##### Crystal data

$K_2Gd(PO_4)(MoO_4)$

$M_r = 490.36$

Orthorhombic, *Ibca*

$a = 6.9527$  (2) Å

$b = 19.7112$  (6) Å

$c = 12.2466$  (3) Å

$V = 1678.35$  (8) Å<sup>3</sup>

$Z = 8$

$F(000) = 1784$

$D_x = 3.881$  Mg m<sup>-3</sup>

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

Cell parameters from 4624 reflections

$\theta = 3.3$ – $30.0^\circ$

$\mu = 10.52$  mm<sup>-1</sup>

$T = 200$  K

Plate, clear light colourless

$0.10 \times 0.08 \times 0.02$  mm

##### Data collection

XtaLAB Synergy, Dualflex, HyPix  
diffractometer

Radiation source: micro-focus sealed X-ray  
tube, PhotonJet (Mo) X-ray Source

Mirror monochromator

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

$\omega$  scans

Absorption correction: gaussian  
(CrysAlisPro; Rigaku OD, 2020)

$T_{\min} = 0.422$ ,  $T_{\max} = 1.000$

6547 measured reflections

1079 independent reflections

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

$R_{\text{int}} = 0.026$

$\theta_{\max} = 30.2^\circ$ ,  $\theta_{\min} = 3.3^\circ$

$h = -8 \rightarrow 8$

$k = -26 \rightarrow 26$

$l = -16 \rightarrow 16$

##### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.017$

$wR(F^2) = 0.045$

$S = 1.13$

1079 reflections

61 parameters

0 restraints

Primary atom site location: dual

Secondary atom site location: difference Fourier

map

$w = 1/[\sigma^2(F_o^2) + (0.0204P)^2 + 6.0211P]$

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

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

$\Delta\rho_{\max} = 1.53$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.64$  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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Gd1	1.000000	0.250000	0.42488 (2)	0.00554 (8)
Mo1	0.750000	0.41682 (2)	0.500000	0.00954 (10)
K1	0.71711 (11)	0.09429 (4)	0.32974 (5)	0.01672 (16)
P1	0.500000	0.250000	0.32047 (8)	0.0060 (2)
O1	0.6709 (3)	0.24105 (10)	0.40045 (17)	0.0095 (4)
O2	0.4787 (3)	0.18814 (11)	0.24608 (17)	0.0094 (4)
O3	0.9564 (3)	0.36581 (11)	0.47067 (18)	0.0139 (4)
O4	0.8056 (4)	0.46677 (12)	0.61376 (19)	0.0204 (5)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Gd1	0.00341 (13)	0.00828 (12)	0.00493 (11)	−0.00007 (6)	0.000	0.000
Mo1	0.0102 (2)	0.00749 (16)	0.01089 (17)	0.000	0.00090 (13)	0.000
K1	0.0158 (4)	0.0130 (3)	0.0213 (3)	0.0014 (3)	−0.0007 (3)	0.0027 (2)
P1	0.0038 (6)	0.0093 (5)	0.0047 (5)	0.0000 (3)	0.000	0.000
O1	0.0037 (11)	0.0172 (10)	0.0076 (9)	0.0001 (8)	0.0006 (8)	0.0003 (8)
O2	0.0101 (11)	0.0110 (10)	0.0070 (9)	−0.0009 (8)	−0.0016 (7)	−0.0012 (8)
O3	0.0129 (11)	0.0111 (10)	0.0178 (11)	−0.0004 (9)	0.0034 (9)	−0.0019 (9)
O4	0.0209 (13)	0.0177 (11)	0.0226 (12)	0.0014 (10)	−0.0022 (10)	−0.0108 (10)

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

Gd1—O1	2.314 (2)	K1—O1	3.037 (2)
Gd1—O1 <sup>i</sup>	2.314 (2)	K1—O2	2.687 (2)
Gd1—O1 <sup>ii</sup>	2.453 (2)	K1—O2 <sup>iv</sup>	2.755 (2)
Gd1—O1 <sup>iii</sup>	2.453 (2)	K1—O3 <sup>i</sup>	2.958 (2)
Gd1—O2 <sup>iv</sup>	2.427 (2)	K1—O3 <sup>vi</sup>	3.143 (2)
Gd1—O2 <sup>v</sup>	2.427 (2)	K1—O4 <sup>vii</sup>	2.970 (3)
Gd1—O3	2.370 (2)	K1—O4 <sup>viii</sup>	2.679 (2)
Gd1—O3 <sup>i</sup>	2.370 (2)	K1—O4 <sup>vi</sup>	3.180 (3)
Mo1—O3 <sup>ii</sup>	1.788 (2)	P1—O1	1.550 (2)
Mo1—O3	1.788 (2)	P1—O1 <sup>ix</sup>	1.550 (2)
Mo1—O4	1.749 (2)	P1—O2 <sup>ix</sup>	1.529 (2)
Mo1—O4 <sup>ii</sup>	1.749 (2)	P1—O2	1.529 (2)
O1—Gd1—O1 <sup>iii</sup>	126.66 (6)	O4 <sup>ii</sup> —Mo1—O4	111.50 (16)
O1 <sup>i</sup> —Gd1—O1 <sup>iii</sup>	68.18 (8)	O1—K1—O3 <sup>vi</sup>	58.59 (6)
O1—Gd1—O1 <sup>i</sup>	165.14 (10)	O1—K1—O4 <sup>vi</sup>	101.73 (6)
O1 <sup>iii</sup> —Gd1—O1 <sup>ii</sup>	58.64 (10)	O2—K1—O2 <sup>iv</sup>	79.43 (6)
O1 <sup>i</sup> —Gd1—O1 <sup>ii</sup>	126.66 (6)	O2 <sup>iv</sup> —K1—O3 <sup>i</sup>	60.76 (6)
O1—Gd1—O1 <sup>ii</sup>	68.18 (8)	O2 <sup>iv</sup> —K1—O3 <sup>vi</sup>	118.34 (6)
O1 <sup>i</sup> —Gd1—O2 <sup>v</sup>	77.86 (7)	O2—K1—O3 <sup>vi</sup>	76.61 (6)
O1—Gd1—O2 <sup>iv</sup>	77.86 (7)	O2—K1—O3 <sup>i</sup>	120.86 (7)
O1 <sup>i</sup> —Gd1—O2 <sup>iv</sup>	89.27 (7)	O2—K1—O4 <sup>vi</sup>	77.78 (6)

O1—Gd1—O2 <sup>v</sup>	89.27 (7)	O2 <sup>iv</sup> —K1—O4 <sup>vi</sup>	157.11 (7)
O1—Gd1—O3	88.71 (8)	O2 <sup>iv</sup> —K1—O4 <sup>vii</sup>	80.51 (7)
O1 <sup>i</sup> —Gd1—O3	94.81 (8)	O2—K1—O4 <sup>vii</sup>	93.86 (7)
O1 <sup>i</sup> —Gd1—O3 <sup>i</sup>	88.71 (8)	O3 <sup>i</sup> —K1—O1	70.21 (6)
O1—Gd1—O3 <sup>i</sup>	94.81 (8)	O3 <sup>i</sup> —K1—O3 <sup>vi</sup>	85.55 (7)
O2 <sup>v</sup> —Gd1—O1 <sup>ii</sup>	144.86 (7)	O3 <sup>vi</sup> —K1—O4 <sup>vi</sup>	53.60 (6)
O2 <sup>v</sup> —Gd1—O1 <sup>iii</sup>	133.34 (7)	O3 <sup>i</sup> —K1—O4 <sup>vii</sup>	117.92 (7)
O2 <sup>iv</sup> —Gd1—O1 <sup>iii</sup>	144.86 (7)	O3 <sup>i</sup> —K1—O4 <sup>vi</sup>	131.61 (7)
O2 <sup>iv</sup> —Gd1—O1 <sup>ii</sup>	133.34 (7)	O4 <sup>vii</sup> —K1—O1	131.43 (6)
O2 <sup>iv</sup> —Gd1—O2 <sup>v</sup>	60.80 (10)	O4 <sup>viii</sup> —K1—O1	147.62 (7)
O3 <sup>i</sup> —Gd1—O1 <sup>ii</sup>	77.67 (7)	O4 <sup>viii</sup> —K1—O2 <sup>iv</sup>	124.43 (7)
O3—Gd1—O1 <sup>iii</sup>	77.67 (7)	O4 <sup>viii</sup> —K1—O2	152.41 (7)
O3 <sup>i</sup> —Gd1—O1 <sup>iii</sup>	78.52 (7)	O4 <sup>viii</sup> —K1—O3 <sup>vi</sup>	99.58 (7)
O3—Gd1—O1 <sup>ii</sup>	78.52 (7)	O4 <sup>viii</sup> —K1—O3 <sup>i</sup>	85.55 (7)
O3—Gd1—O2 <sup>v</sup>	74.22 (7)	O4 <sup>vii</sup> —K1—O3 <sup>vi</sup>	155.97 (7)
O3 <sup>i</sup> —Gd1—O2 <sup>iv</sup>	74.22 (7)	O4 <sup>vii</sup> —K1—O4 <sup>vi</sup>	103.12 (7)
O3 <sup>i</sup> —Gd1—O2 <sup>v</sup>	132.85 (7)	O4 <sup>viii</sup> —K1—O4 <sup>vii</sup>	78.60 (7)
O3—Gd1—O2 <sup>iv</sup>	132.85 (7)	O4 <sup>viii</sup> —K1—O4 <sup>vi</sup>	78.20 (5)
O3 <sup>i</sup> —Gd1—O3	152.63 (11)	O1 <sup>ix</sup> —P1—O1	101.63 (17)
O3—Mo1—O3 <sup>ii</sup>	111.59 (14)	O2—P1—O1	111.11 (11)
O4—Mo1—O3	107.39 (11)	O2 <sup>ix</sup> —P1—O1 <sup>ix</sup>	111.11 (11)
O4—Mo1—O3 <sup>ii</sup>	109.51 (11)	O2—P1—O1 <sup>ix</sup>	113.12 (11)
O4 <sup>ii</sup> —Mo1—O3	109.50 (11)	O2 <sup>ix</sup> —P1—O1	113.12 (11)
O4 <sup>ii</sup> —Mo1—O3 <sup>ii</sup>	107.39 (11)	O2 <sup>ix</sup> —P1—O2	106.87 (17)
O1 <sup>ix</sup> —P1—O1—Gd1	−156.6 (3)	O2 <sup>ix</sup> —P1—O2—K1	133.88 (11)
O1 <sup>ix</sup> —P1—O1—Gd1 <sup>ii</sup>	−0.001 (1)	O2 <sup>ix</sup> —P1—O2—K1 <sup>x</sup>	−106.9 (2)
O1 <sup>ix</sup> —P1—O1—K1	112.05 (8)	O2 <sup>ix</sup> —P1—O2—P1 <sup>ix</sup>	0 (100)
O1 <sup>ix</sup> —P1—O1—P1 <sup>ix</sup>	0 (100)	O3 <sup>ii</sup> —Mo1—O3—Gd1	15.95 (10)
O1—P1—O2—Gd1 <sup>v</sup>	−123.85 (10)	O3 <sup>ii</sup> —Mo1—O3—K1 <sup>iii</sup>	−114.91 (9)
O1 <sup>ix</sup> —P1—O2—Gd1 <sup>v</sup>	122.59 (11)	O3 <sup>ii</sup> —Mo1—O3—K1 <sup>i</sup>	152.13 (15)
O1 <sup>ix</sup> —P1—O2—K1 <sup>x</sup>	15.7 (2)	O3 <sup>ii</sup> —Mo1—O4—K1 <sup>xi</sup>	32.18 (14)
O1—P1—O2—K1 <sup>x</sup>	129.30 (18)	O3—Mo1—O4—K1 <sup>xii</sup>	143.40 (15)
O1 <sup>ix</sup> —P1—O2—K1	−103.53 (11)	O3 <sup>ii</sup> —Mo1—O4—K1 <sup>xii</sup>	−95.27 (17)
O1—P1—O2—K1	10.03 (14)	O3—Mo1—O4—K1 <sup>xi</sup>	−89.15 (12)
O1—P1—O2—P1 <sup>ix</sup>	0 (78)	O3 <sup>ii</sup> —Mo1—O4—K1 <sup>iii</sup>	116.29 (10)
O1 <sup>ix</sup> —P1—O2—P1 <sup>ix</sup>	0 (100)	O3—Mo1—O4—K1 <sup>iii</sup>	−5.05 (11)
O2—P1—O1—Gd1 <sup>ii</sup>	−120.61 (11)	O4 <sup>ii</sup> —Mo1—O3—Gd1	−102.81 (16)
O2 <sup>ix</sup> —P1—O1—Gd1	−37.5 (3)	O4—Mo1—O3—Gd1	135.96 (16)
O2 <sup>ix</sup> —P1—O1—Gd1 <sup>ii</sup>	119.18 (11)	O4—Mo1—O3—K1 <sup>i</sup>	−87.85 (14)
O2—P1—O1—Gd1	82.7 (2)	O4 <sup>ii</sup> —Mo1—O3—K1 <sup>iii</sup>	126.33 (10)
O2 <sup>ix</sup> —P1—O1—K1	−128.76 (10)	O4 <sup>ii</sup> —Mo1—O3—K1 <sup>i</sup>	33.37 (16)
O2—P1—O1—K1	−8.56 (12)	O4—Mo1—O3—K1 <sup>iii</sup>	5.11 (11)
O2—P1—O1—P1 <sup>ix</sup>	0 (100)	O4 <sup>ii</sup> —Mo1—O4—K1 <sup>xi</sup>	150.89 (14)

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O2 <sup>ix</sup> —P1—O1—P1 <sup>ix</sup>	0 (23)	O4 <sup>ii</sup> —Mo1—O4—K1 <sup>iii</sup>	-125.01 (10)
O2 <sup>ix</sup> —P1—O2—Gd1 <sup>v</sup>	0.000 (1)	O4 <sup>ii</sup> —Mo1—O4—K1 <sup>xii</sup>	23.44 (10)

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