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# Tetrapotassium *cis*-dioxido-*trans*-bis(sulfato-$\kappa O$)sulfato($\kappa^2 O,O'$)-molybdate(VI)

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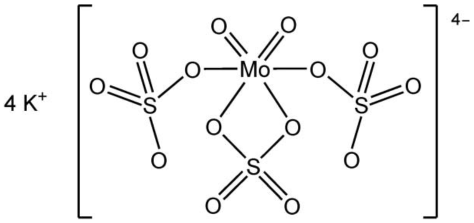
Key indicators: single-crystal X-ray study; $T$ = 120 K; mean $\sigma$(Mo–O) = 0.002 Å; $R$ factor = 0.017; $wR$ factor = 0.042; data-to-parameter ratio = 15.4.

The title compound, $K_4[Mo^{VI}O_2(SO_4)_3]$, was precipitated from a melt of molybdenum(VI) oxide and potassium sulfate in potassium disulfate. The compound contains monomeric $[Mo^{VI}O_2(SO_4)_3]^{4-}$ anions, with the $Mo^{VI}$ atom, both oxide ligands, and the S atom and both ligating O atoms of the bidentate sulfate group lying on a crystallographic mirror plane. One of the potassium cations is nine-coordinate, while the other is eight-coordinate.

## Related literature

For related literature, see: Topsøe & Nielsen (1947); Berg & Thorup (2005); Borup *et al.* (1990); Nørbygaard *et al.* (1998); Nielsen *et al.* (1993); Rasmussen *et al.* (2003); Salles *et al.* (1996); Schäffer & Berg (2005); Tamasi & Cini (2003).



## Experimental

### *Crystal data*

$K_4[MoO_2(SO_4)_3]$
$M_r$ = 572.52
Orthorhombic, *Pnma*
$a$ = 7.5931 (5) Å
$b$ = 17.1276 (11) Å
$c$ = 10.5132 (7) Å
$V$ = 1367.26 (16) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 2.71 mm$^{-1}$
$T$ = 120 (2) K
0.28 × 0.18 × 0.07 mm

### *Data collection*

Bruker SMART APEX CCD diffractometer
Absorption correction: Gaussian (*XPREP*; Bruker, 2002)
$T_{min}$ = 0.512, $T_{max}$ = 0.684
16179 measured reflections
1693 independent reflections
1681 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.018

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.016
$wR(F^2)$ = 0.041
$S$ = 1.11
1693 reflections
110 parameters
$\Delta\rho_{max}$ = 0.59 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.46 e Å$^{-3}$

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

| | | | |
|---|---|---|---|
| Mo1—O1 | 1.6889 (18) | K1—O5$^{i}$ | 2.6408 (12) |
| Mo1—O2 | 1.6883 (18) | K1—O8$^{ii}$ | 3.2305 (14) |
| Mo1—O3 | 2.2665 (17) | K2—O8$^{iii}$ | 2.7005 (14) |
| Mo1—O4 | 2.1837 (16) | K2—O6$^{iv}$ | 3.0239 (13) |
| Mo1—O6 | 2.0365 (12) | | |
| O2—Mo1—O1 | 105.77 (10) | O6—Mo1—O4 | 79.65 (3) |
| O2—Mo1—O6 | 98.04 (4) | O2—Mo1—O3 | 92.29 (8) |
| O1—Mo1—O6 | 94.84 (4) | O1—Mo1—O3 | 161.94 (8) |
| O6—Mo1—O6$^{v}$ | 158.22 (7) | O6—Mo1—O3 | 82.38 (3) |
| O2—Mo1—O4 | 155.71 (8) | O4—Mo1—O3 | 63.43 (6) |
| O1—Mo1—O4 | 98.52 (8) | | |

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2277).

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# supporting information

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# Tetrapotassium *cis*-dioxido-*trans*-bis(sulfato-*κO*)sulfato(*κ*$^2$*O,O′*)molybdate(VI)

**Susan J. Cline Schäffer and Rolf W. Berg**

## S1. Comment

Efforts to improve the industrial vanadium-based sulfuric acid catalyst (Topsøe & Nielsen, 1947) have led to investigations of potassium disulfate's use as a suitable solvent for the production of new sulfate-containing catalysts. Many of the previously reported sulfato compounds precipitated from melts of potassium disulfate contain polymeric anions (Nørbygaard *et al.*, 1998, Berg & Thorup, 2005), while some contain dimers (Nielsen *et al.*, 1993, Rasmussen *et al.*, 2003, Schäffer & Berg, 2005). Monomers such as that in the title compound (Fig. 1) are less common (Borup *et al.*, 1990).

The coordination sphere of the Mo$^{VI}$ atom contains two oxido ligands, two terminally bound sulfato ligands, and a bidentate sulfato ligand. Because of the bidentate ligand, the coordination sphere angles (see Table 1) in the mirror plane are grossly distorted from octahedral. The O3—Mo1—O4 angle is only 63.43 (6)°, while the O1—Mo1—O2 angle opposite to it is 105.77 (10)°. Deviations of 10° or less are found for the other angles between *cis* O atoms. The Mo=O bond distances are nearly equivalent (1.6883 (18)Å & 1.6889 (18) Å), which is expected since both bonds are *trans* to O atoms in the bidentate sulfato ligand. The Mo—O distance to the terminal sulfato [Mo1—O6] is slightly shorter than those to the bidentate sulfato ligand [Mo1—O3 and Mo1—O4]. The Mo—O distances compare well with previously reported values (Salles *et al.*, 1996, Nørbygaard *et al.*, 1998).

The S—O bond distances of 1.5044 (17)–1.5532 (17)Å involving O bound to Mo are longer than the terminal S—O bond lengths of 1.4497–1.4621 (12) Å, an effect typical for sulfato complexes of many different transition metal centers (Borup *et al.*, 1990, Nielsen *et al.*, 1993, Berg & Thorup, 2005). The sulfato ligands have approximately tetrahedral geometry. The angles vary from 101.14° for O3—S1—O4 to 114.68 (10)° for O5—S1—O5$^{i}$ [symmetry code (i): *x*, 1/2 - *y*, *z*]; both extremes involve the bidentate sulfato ligand. Five of the eight remaining independent O—S—O angles deviate by less than 2° from ideal. The potassium cation, K1, is nine-coordinate, while K2 is eight-coordinate. The K—O distances range from 2.6408 to 3.2305 (14) Å.

## S2. Experimental

Crystals were grown from a melt of 28.0 mol% molybdenum(VI) oxide, 43.9 mol% potassium sulfate, and 28.1 mol% potassium disulfate, using a method described previously (Nørbygaard *et al.*, 1998).

## S3. Refinement

(type here to add refinement details)

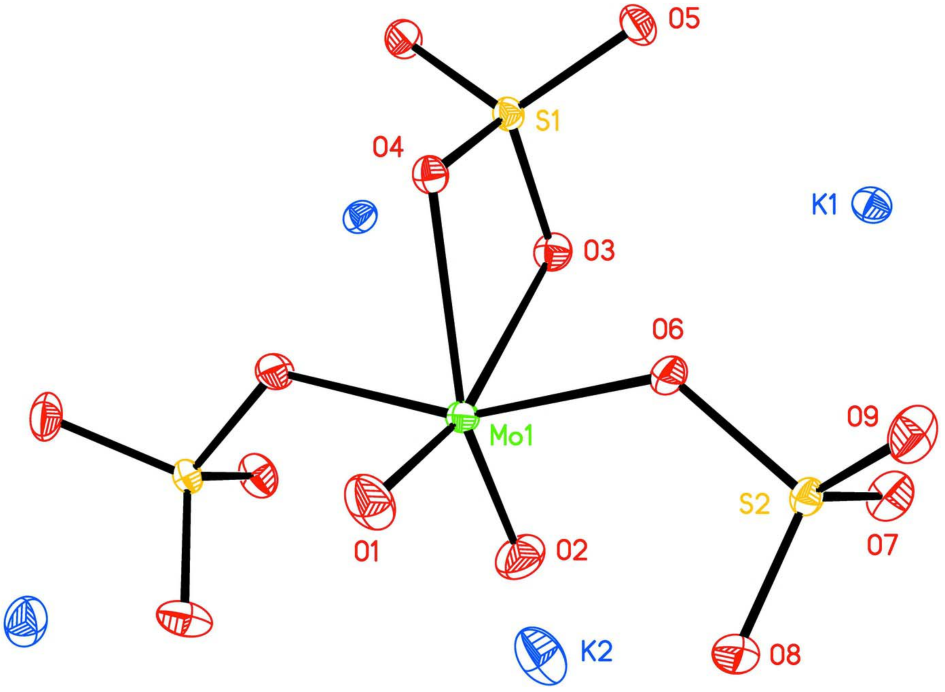

**Figure 1**

The $[Mo^{VI}O_2(SO_4)_3]^{4-}$ anion and four $K^+$ cations, showing displacement ellipsoids at 50% probability.

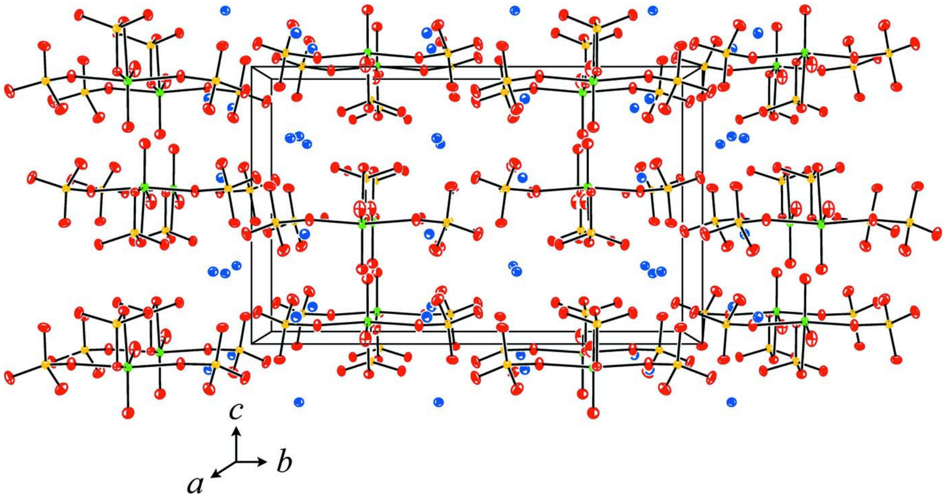

**Figure 2**

The crystal packing, viewed along the *a* axis.

### Tetrapotassium *cis*-dioxido-*trans*-bis(sulfato-\k*O*)sulfato($\kappa^2$*O*,*O′*)molybdate(VI)

*Crystal data*

$K_4[MoO_2(SO_4)_3]$
$M_r = 572.52$
Orthorhombic, *Pnma*
Hall symbol: -P 2ac 2n
$a = 7.5931$ (5) Å
$b = 17.1276$ (11) Å
$c = 10.5132$ (7) Å
$V = 1367.26$ (16) Å$^3$
$Z = 4$
$F(000) = 1112$
$D_x = 2.781$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9999 reflections
$\theta = 2.3–28.0°$
$\mu = 2.71$ mm$^{-1}$
$T = 120$ K
Tabular, colorless
0.28 × 0.18 × 0.08 mm

*Data collection*

Bruker SMART APEX CCD diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: gaussian (*XPREP*; Bruker, 2002)
$T_{\rm min} = 0.512$, $T_{\rm max} = 0.684$
16179 measured reflections
1693 independent reflections
1681 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\rm max} = 28.0°$, $\theta_{\rm min} = 2.3°$
$h = -9 \rightarrow 10$
$k = -22 \rightarrow 22$
$l = -13 \rightarrow 13$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.016$
$wR(F^2) = 0.041$
$S = 1.11$
1693 reflections
110 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
$w = 1/[\sigma^2(F_o^2) + (0.0174P)^2 + 1.7854P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.59$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.46$ e Å$^{-3}$
Extinction correction: *SHELXTL* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00164 (15)

*Special details*

**Experimental**. Five series of frames were filtered for statistical outliers then corrected for absorption using *XPREP* in *SHELXTL* (Sheldrick, 2008) before using *SAINT* & *SADABS* (Bruker, 2002) to sort, merge, and scale the combined data. A series of identical frames was collected twice during the experiment to monitor decay, and none was observed.

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

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

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

| | $x$ | $y$ | $z$ | $U_{\rm iso}$*/$U_{\rm eq}$ |
|---|---|---|---|---|
| K1 | 0.43682 (4) | 0.08865 (2) | −0.24076 (3) | 0.01208 (8) |
| K2 | 0.25655 (5) | 0.10844 (2) | 0.39352 (3) | 0.01644 (9) |
| Mo1 | 0.32312 (3) | 0.2500 | 0.065899 (18) | 0.00961 (7) |
| S1 | 0.62301 (7) | 0.2500 | −0.09790 (5) | 0.00926 (11) |
| S2 | 0.23382 (5) | 0.06574 (2) | 0.06161 (3) | 0.00949 (9) |
| O1 | 0.3105 (3) | 0.2500 | 0.22628 (17) | 0.0209 (4) |
| O2 | 0.1129 (2) | 0.2500 | 0.0135 (2) | 0.0219 (4) |
| O3 | 0.4316 (2) | 0.2500 | −0.13493 (15) | 0.0124 (3) |
| O4 | 0.6095 (2) | 0.2500 | 0.04687 (15) | 0.0102 (3) |
| O5 | 0.70888 (16) | 0.17874 (7) | −0.13903 (11) | 0.0135 (2) |

| | | | | |
|---|---|---|---|---|
| O6 | 0.36968 (15) | 0.13324 (7) | 0.05145 (11) | 0.0134 (2) |
| O7 | 0.16034 (17) | 0.05557 (8) | −0.06586 (11) | 0.0161 (3) |
| O8 | 0.10253 (17) | 0.08813 (8) | 0.15593 (12) | 0.0184 (3) |
| O9 | 0.33322 (16) | −0.00276 (7) | 0.10186 (13) | 0.0176 (3) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| K1 | 0.01082 (17) | 0.01361 (16) | 0.01182 (16) | 0.00149 (12) | −0.00089 (12) | 0.00023 (12) |
| K2 | 0.02176 (19) | 0.01335 (17) | 0.01422 (17) | 0.00508 (14) | 0.00204 (14) | −0.00023 (13) |
| Mo1 | 0.00891 (10) | 0.00789 (10) | 0.01203 (11) | 0.000 | 0.00220 (7) | 0.000 |
| S1 | 0.0105 (2) | 0.0077 (2) | 0.0096 (2) | 0.000 | 0.0013 (2) | 0.000 |
| S2 | 0.01081 (18) | 0.00829 (17) | 0.00938 (18) | −0.00132 (14) | 0.00002 (13) | 0.00071 (13) |
| O1 | 0.0304 (11) | 0.0172 (9) | 0.0150 (9) | 0.000 | 0.0080 (8) | 0.000 |
| O2 | 0.0117 (8) | 0.0178 (9) | 0.0364 (11) | 0.000 | −0.0020 (8) | 0.000 |
| O3 | 0.0119 (8) | 0.0139 (8) | 0.0115 (7) | 0.000 | −0.0019 (6) | 0.000 |
| O4 | 0.0115 (8) | 0.0102 (7) | 0.0089 (7) | 0.000 | 0.0001 (6) | 0.000 |
| O5 | 0.0158 (6) | 0.0095 (5) | 0.0153 (6) | 0.0017 (4) | 0.0044 (5) | −0.0012 (4) |
| O6 | 0.0116 (5) | 0.0088 (5) | 0.0197 (6) | −0.0019 (4) | 0.0022 (5) | −0.0001 (4) |
| O7 | 0.0187 (6) | 0.0186 (6) | 0.0111 (6) | −0.0037 (5) | −0.0034 (4) | 0.0017 (4) |
| O8 | 0.0163 (6) | 0.0201 (6) | 0.0187 (6) | −0.0045 (5) | 0.0072 (5) | −0.0055 (5) |
| O9 | 0.0181 (6) | 0.0116 (6) | 0.0232 (6) | −0.0002 (5) | −0.0046 (5) | 0.0058 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Mo1—O1 | 1.6889 (18) | K2—O8$^{\text{vi}}$ | 2.7005 (14) |
| Mo1—O2 | 1.6883 (18) | K2—O4$^{\text{vii}}$ | 2.7419 (8) |
| Mo1—O3 | 2.2665 (17) | K2—O8 | 2.7799 (14) |
| Mo1—O4 | 2.1837 (16) | K2—O5$^{\text{vii}}$ | 2.8710 (13) |
| Mo1—O6 | 2.0365 (12) | K2—O7$^{\text{viii}}$ | 2.9106 (14) |
| Mo1—O6$^{\text{i}}$ | 2.0365 (12) | K2—O9$^{\text{viii}}$ | 2.9220 (14) |
| S1—O3 | 1.5044 (17) | K2—O1 | 3.0229 (11) |
| S1—O4 | 1.5254 (16) | K2—O6$^{\text{vii}}$ | 3.0239 (13) |
| S1—O5 | 1.4497 (12) | O1—K2$^{\text{i}}$ | 3.0229 (11) |
| S1—O5$^{\text{i}}$ | 1.4497 (12) | O3—K1$^{\text{i}}$ | 2.9794 (7) |
| S2—O6 | 1.5532 (12) | O4—K2$^{\text{ix}}$ | 2.7419 (8) |
| S2—O7 | 1.4621 (12) | O4—K2$^{\text{vi}}$ | 2.7419 (8) |
| S2—O8 | 1.4575 (13) | O5—K1$^{\text{iii}}$ | 2.6408 (12) |
| S2—O9 | 1.4577 (12) | O5—K2$^{\text{vi}}$ | 2.8710 (13) |
| K1—O5$^{\text{ii}}$ | 2.6408 (12) | O6—K2$^{\text{vi}}$ | 3.0239 (13) |
| K1—O7$^{\text{iii}}$ | 2.7083 (12) | O7—K1$^{\text{ii}}$ | 2.7083 (12) |
| K1—O9$^{\text{iv}}$ | 2.7102 (12) | O7—K2$^{\text{v}}$ | 2.9106 (14) |
| K1—O5 | 2.7915 (13) | O8—K2$^{\text{vii}}$ | 2.7005 (14) |
| K1—O7 | 2.8476 (13) | O8—K1$^{\text{viii}}$ | 3.2305 (14) |
| K1—O3 | 2.9794 (7) | O9—K1$^{\text{iv}}$ | 2.7102 (12) |
| K1—O9$^{\text{v}}$ | 3.0176 (13) | O9—K2$^{\text{v}}$ | 2.9220 (13) |
| K1—O6 | 3.2064 (12) | O9—K1$^{\text{viii}}$ | 3.0176 (13) |
| K1—O8$^{\text{v}}$ | 3.2305 (14) | | |

| | | | |
|---|---|---|---|
| O5$^{ii}$—K1—O7$^{iii}$ | 100.00 (4) | O1—Mo1—O6$^{i}$ | 94.84 (4) |
| O5$^{ii}$—K1—O9$^{iv}$ | 175.76 (4) | O6—Mo1—O6$^{i}$ | 158.22 (7) |
| O7$^{iii}$—K1—O9$^{iv}$ | 83.52 (4) | O2—Mo1—O4 | 155.71 (8) |
| O5$^{ii}$—K1—O5 | 110.20 (4) | O1—Mo1—O4 | 98.52 (8) |
| O7$^{iii}$—K1—O5 | 86.53 (4) | O6—Mo1—O4 | 79.65 (3) |
| O9$^{iv}$—K1—O5 | 67.47 (4) | O6$^{i}$—Mo1—O4 | 79.65 (3) |
| O5$^{ii}$—K1—O7 | 86.68 (4) | O2—Mo1—O3 | 92.29 (8) |
| O7$^{iii}$—K1—O7 | 154.84 (5) | O1—Mo1—O3 | 161.94 (8) |
| O9$^{iv}$—K1—O7 | 91.09 (4) | O6—Mo1—O3 | 82.38 (3) |
| O5—K1—O7 | 114.09 (4) | O6$^{i}$—Mo1—O3 | 82.38 (3) |
| O5$^{ii}$—K1—O3 | 68.16 (4) | O4—Mo1—O3 | 63.43 (6) |
| O7$^{iii}$—K1—O3 | 118.86 (4) | O3—S1—O4 | 101.14 (9) |
| O9$^{iv}$—K1—O3 | 108.11 (4) | O5—S1—O5$^{i}$ | 114.68 (10) |
| O5—K1—O3 | 49.76 (4) | O5—S1—O3 | 110.93 (6) |
| O7—K1—O3 | 86.20 (4) | O5$^{i}$—S1—O3 | 110.93 (6) |
| O5$^{ii}$—K1—O9$^{v}$ | 64.98 (4) | O5—S1—O4 | 109.14 (6) |
| O7$^{iii}$—K1—O9$^{v}$ | 84.97 (4) | O5$^{i}$—S1—O4 | 109.14 (6) |
| O9$^{iv}$—K1—O9$^{v}$ | 117.94 (5) | O8—S2—O9 | 111.62 (8) |
| O5—K1—O9$^{v}$ | 169.23 (4) | O8—S2—O7 | 113.20 (8) |
| O7—K1—O9$^{v}$ | 75.89 (4) | O9—S2—O7 | 111.58 (8) |
| O3—K1—O9$^{v}$ | 130.39 (4) | O8—S2—O6 | 107.77 (7) |
| O5$^{ii}$—K1—O6 | 102.42 (4) | O9—S2—O6 | 105.97 (7) |
| O7$^{iii}$—K1—O6 | 150.31 (4) | O7—S2—O6 | 106.20 (7) |
| O9$^{iv}$—K1—O6 | 73.47 (4) | Mo1—O1—K2$^{i}$ | 126.04 (3) |
| O5—K1—O6 | 67.60 (3) | Mo1—O1—K2 | 126.04 (3) |
| O7—K1—O6 | 46.49 (3) | K2$^{i}$—O1—K2 | 106.66 (6) |
| O3—K1—O6 | 54.49 (4) | S1—O3—Mo1 | 96.32 (8) |
| O9$^{v}$—K1—O6 | 122.24 (3) | S1—O3—K1 | 94.81 (4) |
| O5$^{ii}$—K1—O8$^{v}$ | 109.03 (4) | Mo1—O3—K1 | 110.65 (3) |
| O7$^{iii}$—K1—O8$^{v}$ | 67.02 (4) | S1—O3—K1$^{i}$ | 94.81 (4) |
| O9$^{iv}$—K1—O8$^{v}$ | 74.46 (4) | Mo1—O3—K1$^{i}$ | 110.65 (3) |
| O5—K1—O8$^{v}$ | 135.67 (3) | K1—O3—K1$^{i}$ | 136.12 (6) |
| O7—K1—O8$^{v}$ | 87.85 (4) | S1—O4—Mo1 | 99.11 (8) |
| O3—K1—O8$^{v}$ | 173.55 (4) | S1—O4—K2$^{ix}$ | 101.58 (4) |
| O9$^{v}$—K1—O8$^{v}$ | 45.24 (3) | Mo1—O4—K2$^{ix}$ | 112.62 (4) |
| O6—K1—O8$^{v}$ | 122.05 (3) | S1—O4—K2$^{vi}$ | 101.58 (4) |
| O8$^{vi}$—K2—O4$^{vii}$ | 123.65 (4) | Mo1—O4—K2$^{vi}$ | 112.62 (4) |
| O8$^{vi}$—K2—O8 | 102.72 (3) | K2$^{ix}$—O4—K2$^{vi}$ | 124.32 (6) |
| O4$^{vii}$—K2—O8 | 98.32 (4) | S1—O5—K1$^{iii}$ | 158.35 (7) |
| O8$^{vi}$—K2—O5$^{vii}$ | 110.47 (4) | S1—O5—K1 | 104.29 (6) |
| O4$^{vii}$—K2—O5$^{vii}$ | 51.12 (4) | K1$^{iii}$—O5—K1 | 88.78 (4) |
| O8—K2—O5$^{vii}$ | 143.83 (4) | S1—O5—K2$^{vi}$ | 98.14 (6) |
| O8$^{vi}$—K2—O7$^{viii}$ | 72.12 (4) | K1$^{iii}$—O5—K2$^{vi}$ | 95.88 (4) |
| O4$^{vii}$—K2—O7$^{viii}$ | 155.26 (4) | K1—O5—K2$^{vi}$ | 101.88 (4) |
| O8—K2—O7$^{viii}$ | 95.87 (4) | S2—O6—Mo1 | 127.63 (7) |
| O5$^{vii}$—K2—O7$^{viii}$ | 107.47 (3) | S2—O6—K2$^{vi}$ | 121.83 (6) |
| O8$^{vi}$—K2—O9$^{viii}$ | 106.95 (4) | Mo1—O6—K2$^{vi}$ | 107.00 (5) |

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| O4$^{vii}$—K2—O9$^{viii}$ | 106.35 (4) | S2—O6—K1 | 89.66 (5) |
| O8—K2—O9$^{viii}$ | 119.86 (4) | Mo1—O6—K1 | 109.45 (5) |
| O5$^{vii}$—K2—O9$^{viii}$ | 63.67 (3) | K2$^{vi}$—O6—K1 | 89.74 (3) |
| O7$^{viii}$—K2—O9$^{viii}$ | 48.91 (3) | S2—O7—K1$^{ii}$ | 154.47 (8) |
| O8$^{vi}$—K2—O1 | 81.93 (5) | S2—O7—K1 | 106.67 (6) |
| O4$^{vii}$—K2—O1 | 58.59 (4) | K1$^{ii}$—O7—K1 | 86.31 (3) |
| O8—K2—O1 | 68.58 (4) | S2—O7—K2$^{v}$ | 99.59 (6) |
| O5$^{vii}$—K2—O1 | 101.75 (4) | K1$^{ii}$—O7—K2$^{v}$ | 103.16 (4) |
| O7$^{viii}$—K2—O1 | 146.11 (4) | K1—O7—K2$^{v}$ | 86.41 (4) |
| O9$^{viii}$—K2—O1 | 164.67 (4) | S2—O8—K2$^{vii}$ | 124.63 (7) |
| O8$^{vi}$—K2—O6$^{vii}$ | 179.32 (4) | S2—O8—K2 | 110.89 (7) |
| O4$^{vii}$—K2—O6$^{vii}$ | 55.70 (4) | K2$^{vii}$—O8—K2 | 124.48 (5) |
| O8—K2—O6$^{vii}$ | 77.34 (4) | S2—O8—K1$^{viii}$ | 92.60 (6) |
| O5$^{vii}$—K2—O6$^{vii}$ | 69.31 (3) | K2$^{vii}$—O8—K1$^{viii}$ | 95.48 (4) |
| O7$^{viii}$—K2—O6$^{vii}$ | 108.55 (3) | K2—O8—K1$^{viii}$ | 81.61 (4) |
| O9$^{viii}$—K2—O6$^{vii}$ | 73.56 (3) | S2—O9—K1$^{iv}$ | 157.94 (8) |
| O1—K2—O6$^{vii}$ | 97.48 (4) | S2—O9—K2$^{v}$ | 99.22 (6) |
| O2—Mo1—O1 | 105.77 (10) | K1$^{iv}$—O9—K2$^{v}$ | 102.59 (4) |
| O2—Mo1—O6 | 98.04 (4) | S2—O9—K1$^{viii}$ | 101.52 (6) |
| O1—Mo1—O6 | 94.84 (4) | K1$^{iv}$—O9—K1$^{viii}$ | 82.98 (3) |
| O2—Mo1—O6$^{i}$ | 98.04 (4) | K2$^{v}$—O9—K1$^{viii}$ | 87.16 (3) |

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