inorganic compounds

Octarubidium di-μ-sulfato-κ⁴*O*:*O*′-bis-[*cis*-dioxido-*cis*-disulfatotungstate(VI)]

Kenny Ståhl* and Rolf W. Berg

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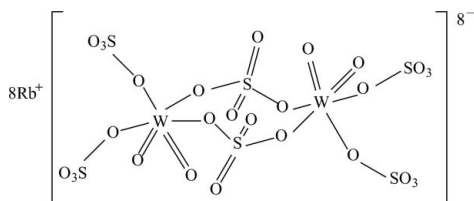
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The title compound, $Rb_8[W_2O_4(SO_4)_6]$, was precipitated from a melt of tungsten(VI) oxide and rubidium sulfate in rubidium disulfate. The unit cell contains two discrete $[\{W^{VI}O_2(SO_4)_2\}_2(\mu\text{-}SO_4)_2]^{8-}$ units connected by Rb–O coordination. The W atom is octahedrally surrounded by two oxide ligands, two terminal sulfate ligands and two bridging sulfate groups. One Rb atom is coordinated by eight O atoms, whereas the three other Rb atoms are coordinated by nine O atoms from sulfate and oxide groups, leading to distorted $[RbO_x]$ polyhedra.

Related literature

For methods used in the synthesis, see: Berg *et al.* (2006); Borup *et al.* (1990); Nørbygaard *et al.* (1998). For the crystal structure of the potassium analog, see: Schäffer & Berg (2005).

Experimental

Crystal data

$Rb_8[W_2O_4(SO_4)_6]$	V = 1452.30 (12) Å³
M_r = 1691.86	Z = 2
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 9.6405 (5) Å	μ = 21.77 mm⁻¹
b = 13.9890 (7) Å	T = 120 K
c = 10.7692 (5) Å	0.45 × 0.20 × 0.05 mm
β = 90.472 (1)°	

Data collection

Bruker SMART APEX diffractometer	Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002) T_{min} = 0.077, T_{max} = 0.59
18895 measured reflections	3360 reflections with $I > 2\sigma(I)$
3496 independent reflections	R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020	200 parameters
$wR(F^2)$ = 0.052	$\Delta\rho_{max}$ = 1.35 e Å⁻³
S = 1.11	$\Delta\rho_{min}$ = -1.51 e Å⁻³
3496 reflections	

Table 1
Selected bond lengths (Å).

W1—O1	1.716 (2)	Rb3—O12^i	3.101 (2)
W1—O2	1.721 (2)	Rb3—O11^{vii}	3.170 (2)
W1—O3	1.960 (2)	Rb3—O12^v	3.173 (3)
W1—O4	2.009 (2)	Rb3—O1	3.220 (2)
W1—O5	2.097 (2)	Rb3—O13^v	3.225 (3)
W1—O6	2.254 (2)	Rb4—O8^{viii}	2.910 (3)
Rb1—O1	2.877 (2)	Rb4—O3^i	2.914 (2)
Rb1—O8	2.931 (2)	Rb4—O10	2.941 (3)
Rb1—O7	2.939 (3)	Rb4—O14	2.964 (2)
Rb1—O9^i	2.955 (3)	Rb4—O13^i	2.981 (3)
Rb1—O6^{ii}	3.009 (2)	Rb4—O12^i	3.103 (3)
Rb1—O11^{iii}	3.010 (2)	Rb4—O6^i	3.221 (2)
Rb1—O2^{iii}	3.084 (2)	Rb4—O5	3.253 (2)
Rb1—O5^{ii}	3.185 (2)	Rb4—O4	3.423 (2)
Rb2—O14^{iv}	2.761 (3)	S1—O11	1.445 (2)
Rb2—O7^v	2.893 (2)	S1—O14	1.449 (2)
Rb2—O9^{vi}	2.903 (3)	S1—O6^{ix}	1.490 (2)
Rb2—O2	2.939 (2)	S1—O4	1.531 (2)
Rb2—O13^{ii}	2.986 (2)	S2—O12	1.451 (3)
Rb2—O8	3.082 (3)	S2—O9	1.452 (3)
Rb2—O10^v	3.106 (3)	S2—O8	1.454 (3)
Rb2—O9	3.337 (3)	S2—O3	1.575 (2)
Rb2—O1	3.374 (3)	S3—O13	1.461 (2)
Rb3—O11	2.776 (2)	S3—O7	1.464 (3)
Rb3—O10^{ii}	2.788 (3)	S3—O10	1.464 (3)
Rb3—O7^v	2.874 (2)	S3—O5	1.528 (2)
Rb3—O14^{vii}	2.935 (2)		

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2002); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP-3* (Farrugia, 1997) and *ATOMS* (Dowty, 2000); software used to prepare material for publication: *SHELXTL*.

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supporting information

Octarubidium di-*µ*-sulfato-*κ*⁴*O*:*O*′-bis[*cis*-dioxido-*cis*-disulfatotungstate(VI)]

Kenny Ståhl and Rolf W. Berg

S1. Comment

Tungsten trioxide is known as a highly inert solid, practically insoluble in acids. It was discovered that WO_3 can be dissolved in considerable amounts in acidic sulfate melts at high temperatures (Schäffer and Berg, 2005 and Berg *et al.*, 2006). When therefore WO_3, Rb_2SO_4 and $Rb_2S_2O_7$ are mixed in varying molar amounts in sealed ampoules and heated for equilibration in a rocking furnace at appr. 600 °C for appr. 1 hr, clear melts are formed ($Rb_2S_2O_7$ is hygroscopic and needs to be handled in a dry box). It has been shown that $[WO_2]^{2+}$ ions formed are solvated by SO_4^{2-} ions. The stoichiometry of the reaction have been determined fairly accurately as 1:1:1, or $2WO_3 + 2M_2SO_4 + 2M_2S_2O_7 \rightarrow M_8[(WO_2)_2(\mu\text{-}SO_4)_2(SO_4)_4]$ for the case of M = K, Schäffer and Berg, 2005. Here we have studied the case of M = Rb and have found closely analogous results for single crystals of the compound, $1WO_3$:$1Rb_2S_2O_7$:$1Rb_2SO_4$. The main difference for M = Rb as compared to M = K is as expected in the M - O coordination: Rb1/K1, CN = 8/8, <M - O> = 2.999 (1)/2.822 (1); Rb2/K2, CN = 9/7, <M - O> = 3.042 (1)/2.825 (1); Rb3/K3, CN = 9/8, <M - O> = 3.029 (1)/2.909 (1); Rb4/K4, CN = 9/9, <M - O> = 3.079 (1)/2.945 (1). As the oxo-sulfatotungstate groups are extended in the b-direction, the major changes in the unit cell axes are observed in the a- and c- directions.

S2. Experimental

The crystals were grown from a melt of 20.6 mol % tungsten trioxide and 19.9 mol % rubidium sulfate in rubidium di-sulfate, using the methods described by Borup *et al.* (1990), Nørbygaard *et al.* (1998) and Berg *et al.* (2006).
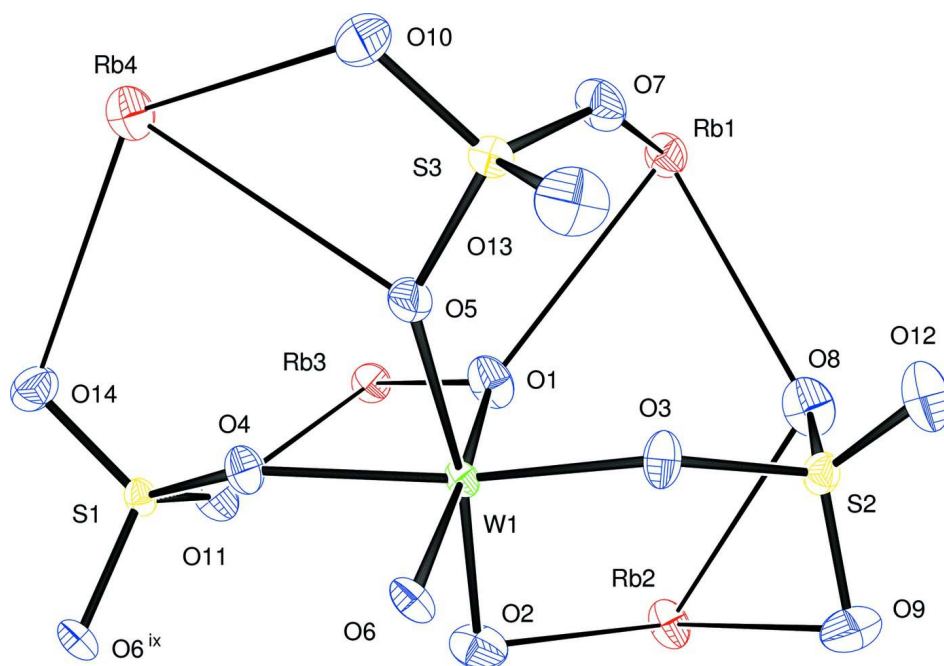
Figure 1

The asymmetric unit of (I) showing 50% probability displacement ellipsoids and the atomic numbering. O6[iv] was added to complete the S1 coordination.
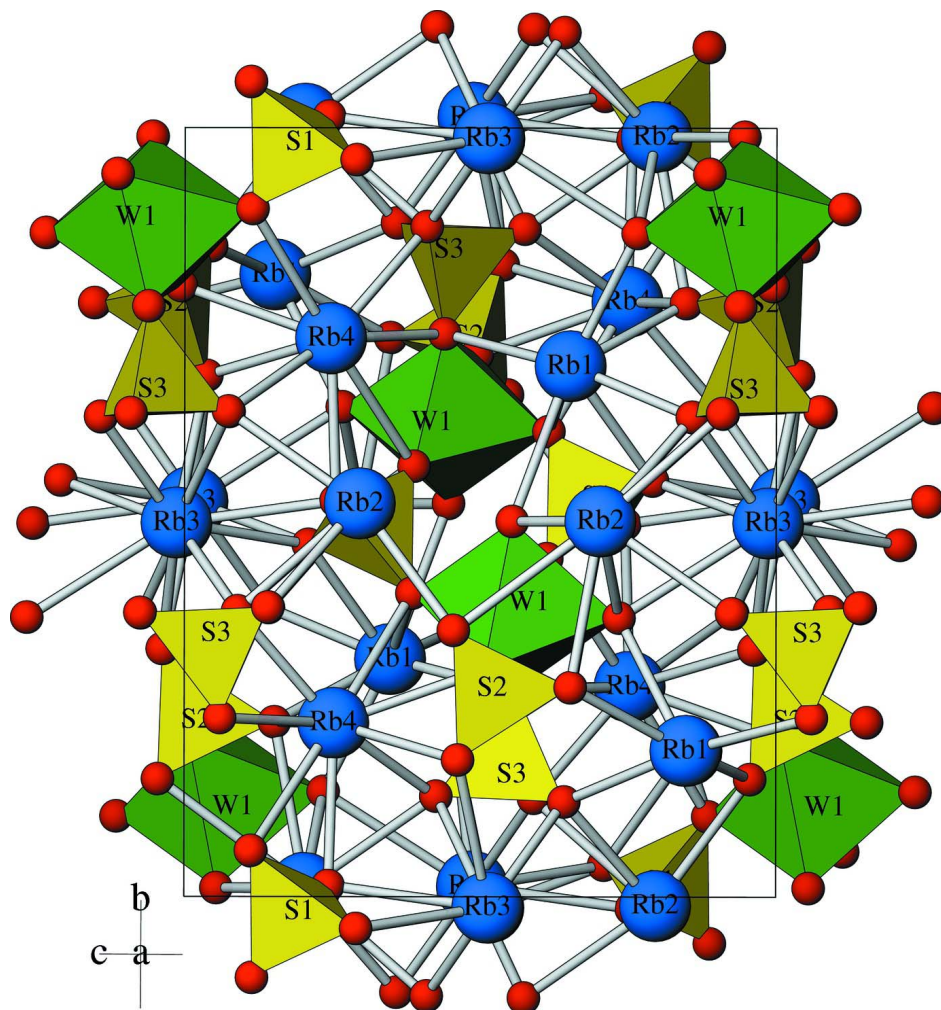
Figure 2

The crystal packing of (I) viewed down the *a* axis. W atoms are in the centers of the octahedra and S atoms are in the centers of the tetrahedra. Rb are shown as large and O as small spheres.

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$M_r = 1691.86$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 9.6405\ (5)$ Å
$b = 13.9890\ (7)$ Å
$c = 10.7692\ (5)$ Å
$\beta = 90.472\ (1)°$
$V = 1452.30\ (12)$ Å^3
$Z = 2$

$F(000) = 1528$
$D_x = 3.869$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6979 reflections
$\theta = 2.4–28.0°$
$\mu = 21.77$ mm^{-1}
$T = 120$ K
Irregular, colourless
$0.45 × 0.20 × 0.05$ mm

Data collection

Bruker SMART APEX diffractometer	18895 measured reflections
Radiation source: fine-focus sealed tube	3496 independent reflections
Graphite monochromator	3360 reflections with $I > 2\sigma(I)$
ω scan, frame data integration	$R_{int} = 0.036$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)	$\theta_{max} = 28.0°$, $\theta_{min} = 2.4°$
	$h = -12\rightarrow12$
	$k = -18\rightarrow18$
$T_{min} = 0.077$, $T_{max} = 0.59$	$l = -14\rightarrow14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.020$	$w = 1/[\sigma^2(F_o^2) + (0.0287P)^2 + 1.1918P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.052$	
$S = 1.11$	$(\Delta/\sigma)_{max} = 0.001$
3496 reflections	$\Delta\rho_{max} = 1.35$ e Å^{-3}
200 parameters	$\Delta\rho_{min} = -1.51$ e Å^{-3}
0 restraints	Extinction correction: *SHELXTL* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.00175 (10)

Special details

Experimental. Oxford Cryosystem liquid nitrogen cryostream cooler

Geometry. All e.s.d.'s 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.

Refinement. A series of identical frames was collected twice during the experiment to monitor decay. No decay was detected and decay correction was not applied. Systematic conditions suggested the unambiguous space group. The structure was solved by direct methods (Sheldrick, 2008). The space group choice was confirmed by successful convergence of the full-matrix least-squares refinement on F^2 (Sheldrick, 2008). Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ 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. The highest peak in the final difference Fourier map was 0.85Å and the deepest hole was 0.78Å from W1. The final map had no other significant features. A final analysis of variance between observed and calculated structure factors showed no dependence on amplitude or resolution.

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

	x	y	z	U_{iso}*/U_{eq}
W1	0.168190 (12)	0.117132 (8)	0.917586 (11)	0.00604 (6)
Rb1	0.31202 (3)	0.30953 (2)	0.65221 (3)	0.01095 (8)
Rb2	0.50882 (3)	0.01145 (2)	0.79641 (3)	0.01149 (8)
Rb3	0.19055 (3)	0.01388 (2)	0.51347 (3)	0.01057 (8)
Rb4	−0.23085 (3)	0.22589 (2)	0.74755 (3)	0.01173 (8)
S1	−0.07712 (8)	−0.01972 (5)	0.80402 (7)	0.00671 (15)
S2	0.46037 (8)	0.22894 (6)	0.97953 (7)	0.00823 (15)
S3	0.03580 (8)	0.34141 (5)	0.94629 (7)	0.00726 (15)
O1	0.2116 (3)	0.13695 (17)	0.7656 (2)	0.0126 (5)
O2	0.2550 (3)	0.01169 (17)	0.9456 (2)	0.0134 (5)

O3	0.3015 (2)	0.20560 (17)	0.9939 (2)	0.0105 (5)
O4	−0.0203 (2)	0.06004 (16)	0.8874 (2)	0.0095 (4)
O5	0.0314 (2)	0.23235 (16)	0.9395 (2)	0.0113 (5)
O6	0.0953 (2)	0.10525 (15)	1.1153 (2)	0.0086 (4)
O7	0.1394 (3)	0.37617 (16)	0.8585 (2)	0.0123 (5)
O8	0.4896 (3)	0.22782 (18)	0.8473 (2)	0.0146 (5)
O9	0.5342 (3)	0.1533 (2)	1.0446 (3)	0.0221 (6)
O10	−0.1042 (3)	0.37142 (17)	0.9097 (2)	0.0143 (5)
O11	0.0230 (3)	−0.04158 (17)	0.7090 (2)	0.0118 (5)
O12	0.4761 (3)	0.32204 (18)	1.0374 (2)	0.0160 (5)
O13	0.0695 (3)	0.36869 (18)	1.0740 (2)	0.0168 (5)
O14	−0.2088 (3)	0.01462 (17)	0.7559 (2)	0.0121 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
W1	0.00608 (8)	0.00633 (8)	0.00575 (8)	−0.00051 (4)	0.00225 (5)	−0.00028 (4)
Rb1	0.01021 (15)	0.01196 (15)	0.01072 (15)	−0.00113 (11)	0.00177 (11)	0.00109 (11)
Rb2	0.01089 (16)	0.01125 (15)	0.01242 (16)	−0.00146 (11)	0.00470 (11)	−0.00220 (11)
Rb3	0.01010 (15)	0.01395 (15)	0.00769 (15)	−0.00045 (11)	0.00251 (11)	0.00227 (10)
Rb4	0.01259 (16)	0.01381 (15)	0.00883 (15)	−0.00152 (12)	0.00221 (11)	−0.00042 (11)
S1	0.0069 (3)	0.0080 (3)	0.0052 (3)	−0.0015 (3)	0.0008 (3)	0.0002 (3)
S2	0.0065 (3)	0.0102 (4)	0.0080 (4)	−0.0011 (3)	0.0004 (3)	0.0005 (3)
S3	0.0086 (4)	0.0070 (3)	0.0062 (3)	0.0008 (3)	0.0016 (3)	−0.0007 (3)
O1	0.0157 (12)	0.0143 (11)	0.0077 (11)	−0.0046 (10)	0.0025 (9)	−0.0001 (9)
O2	0.0144 (12)	0.0098 (11)	0.0161 (12)	0.0047 (9)	0.0057 (9)	0.0011 (9)
O3	0.0071 (11)	0.0141 (11)	0.0104 (11)	−0.0017 (9)	0.0015 (8)	−0.0033 (9)
O4	0.0099 (11)	0.0093 (10)	0.0094 (11)	−0.0024 (9)	0.0007 (8)	−0.0019 (8)
O5	0.0088 (11)	0.0081 (10)	0.0170 (12)	0.0002 (9)	0.0006 (9)	0.0009 (9)
O6	0.0110 (11)	0.0068 (10)	0.0080 (11)	−0.0025 (8)	0.0025 (9)	0.0006 (8)
O7	0.0123 (12)	0.0127 (12)	0.0120 (12)	−0.0024 (9)	0.0043 (9)	0.0030 (8)
O8	0.0156 (12)	0.0178 (12)	0.0105 (12)	−0.0028 (10)	0.0057 (9)	−0.0012 (9)
O9	0.0171 (13)	0.0202 (14)	0.0288 (15)	0.0015 (11)	−0.0074 (11)	0.0102 (11)
O10	0.0099 (12)	0.0123 (11)	0.0207 (14)	0.0039 (9)	0.0018 (10)	−0.0009 (9)
O11	0.0142 (12)	0.0130 (11)	0.0084 (11)	−0.0017 (9)	0.0049 (8)	−0.0008 (9)
O12	0.0153 (12)	0.0157 (12)	0.0170 (13)	−0.0054 (10)	0.0020 (10)	−0.0047 (10)
O13	0.0250 (15)	0.0173 (12)	0.0079 (12)	0.0004 (11)	−0.0014 (10)	−0.0039 (9)
O14	0.0110 (12)	0.0151 (12)	0.0102 (11)	0.0011 (9)	−0.0017 (9)	0.0014 (9)

Geometric parameters (Å, º)

W1—O1	1.716 (2)	Rb3—O12[i]	3.101 (2)
W1—O2	1.721 (2)	Rb3—O11[vii]	3.170 (2)
W1—O3	1.960 (2)	Rb3—O12[v]	3.173 (3)
W1—O4	2.009 (2)	Rb3—O1	3.220 (2)
W1—O5	2.097 (2)	Rb3—O13[v]	3.225 (3)
W1—O6	2.254 (2)	Rb4—O8[viii]	2.910 (3)
Rb1—O1	2.877 (2)	Rb4—O3[i]	2.914 (2)

Rb1—O8	2.931 (2)	Rb4—O10	2.941 (3)
Rb1—O7	2.939 (3)	Rb4—O14	2.964 (2)
Rb1—O9[i]	2.955 (3)	Rb4—O13[i]	2.981 (3)
Rb1—O6[ii]	3.009 (2)	Rb4—O12[i]	3.103 (3)
Rb1—O11[iii]	3.010 (2)	Rb4—O6[i]	3.221 (2)
Rb1—O2[iii]	3.084 (2)	Rb4—O5	3.253 (2)
Rb1—O5[ii]	3.185 (2)	Rb4—O4	3.423 (2)
Rb2—O14[iv]	2.761 (3)	S1—O11	1.445 (2)
Rb2—O7[v]	2.893 (2)	S1—O14	1.449 (2)
Rb2—O9[vi]	2.903 (3)	S1—O6[ix]	1.490 (2)
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Rb2—O13[ii]	2.986 (2)	S2—O12	1.451 (3)
Rb2—O8	3.082 (3)	S2—O9	1.452 (3)
Rb2—O10[v]	3.106 (3)	S2—O8	1.454 (3)
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Rb3—O11	2.776 (2)	S3—O7	1.464 (3)
Rb3—O10[ii]	2.788 (3)	S3—O10	1.464 (3)
Rb3—O7[v]	2.874 (2)	S3—O5	1.528 (2)
Rb3—O14[vii]	2.935 (2)		
O1—W1—O2	100.59 (12)	O7[v]—Rb3—O12[i]	146.54 (7)
O1—W1—O3	97.67 (11)	O14[vii]—Rb3—O12[i]	103.19 (7)
O2—W1—O3	98.71 (11)	O11—Rb3—O11[vii]	103.26 (6)
O1—W1—O4	97.90 (10)	O10[ii]—Rb3—O11[vii]	94.90 (7)
O2—W1—O4	97.20 (11)	O7[v]—Rb3—O11[vii]	143.97 (6)
O3—W1—O4	155.24 (9)	O14[vii]—Rb3—O11[vii]	46.63 (6)
O1—W1—O5	98.15 (11)	O12[i]—Rb3—O11[vii]	62.82 (6)
O2—W1—O5	160.75 (11)	O11—Rb3—O12[v]	66.26 (7)
O3—W1—O5	83.02 (9)	O10[ii]—Rb3—O12[v]	141.04 (7)
O4—W1—O5	75.81 (9)	O7[v]—Rb3—O12[v]	78.58 (6)
O1—W1—O6	173.62 (10)	O14[vii]—Rb3—O12[v]	75.47 (7)
O2—W1—O6	85.75 (10)	O12[i]—Rb3—O12[v]	107.66 (6)
O3—W1—O6	81.90 (9)	O11[vii]—Rb3—O12[v]	69.40 (6)
O4—W1—O6	80.50 (9)	O11—Rb3—O1	62.79 (7)
O5—W1—O6	75.48 (9)	O10[ii]—Rb3—O1	89.42 (7)
O1—Rb1—O8	64.22 (7)	O7[v]—Rb3—O1	85.54 (6)
O1—Rb1—O7	75.57 (7)	O14[vii]—Rb3—O1	154.44 (7)
O8—Rb1—O7	84.99 (7)	O12[i]—Rb3—O1	64.64 (6)
O1—Rb1—O9[i]	90.40 (8)	O11[vii]—Rb3—O1	127.46 (6)
O8—Rb1—O9[i]	150.64 (8)	O12[v]—Rb3—O1	128.76 (6)
O7—Rb1—O9[i]	73.96 (7)	O11—Rb3—O13[v]	117.97 (7)
O1—Rb1—O6[ii]	134.22 (7)	O10[ii]—Rb3—O13[v]	74.48 (7)
O8—Rb1—O6[ii]	73.97 (7)	O7[v]—Rb3—O13[v]	46.39 (7)
O7—Rb1—O6[ii]	119.62 (6)	O14[vii]—Rb3—O13[v]	64.96 (6)
O9[i]—Rb1—O6[ii]	134.35 (7)	O12[i]—Rb3—O13[v]	166.10 (7)
O1—Rb1—O11[iii]	123.25 (6)	O11[vii]—Rb3—O13[v]	108.61 (6)
O8—Rb1—O11[iii]	67.10 (7)	O12[v]—Rb3—O13[v]	77.31 (7)

O7—Rb1—O11ⁱⁱⁱ	72.87 (7)	O1—Rb3—O13^v	122.86 (6)
O9ⁱ—Rb1—O11ⁱⁱⁱ	123.13 (7)	O8^{viii}—Rb4—O3ⁱ	116.73 (7)
O6ⁱⁱ—Rb1—O11ⁱⁱⁱ	46.76 (6)	O8^{viii}—Rb4—O10	98.95 (7)
O1—Rb1—O2ⁱⁱⁱ	147.59 (7)	O3ⁱ—Rb4—O10	106.38 (7)
O8—Rb1—O2ⁱⁱⁱ	136.20 (7)	O8^{viii}—Rb4—O14	93.69 (7)
O7—Rb1—O2ⁱⁱⁱ	81.33 (6)	O3ⁱ—Rb4—O14	110.37 (7)
O9ⁱ—Rb1—O2ⁱⁱⁱ	61.21 (7)	O10—Rb4—O14	130.00 (7)
O6ⁱⁱ—Rb1—O2ⁱⁱⁱ	77.25 (6)	O8^{viii}—Rb4—O13ⁱ	68.94 (7)
O11ⁱⁱⁱ—Rb1—O2ⁱⁱⁱ	69.12 (6)	O3ⁱ—Rb4—O13ⁱ	68.39 (7)
O1—Rb1—O5ⁱⁱ	112.24 (7)	O10—Rb4—O13ⁱ	160.41 (7)
O8—Rb1—O5ⁱⁱ	93.25 (6)	O14—Rb4—O13ⁱ	67.81 (7)
O7—Rb1—O5ⁱⁱ	170.30 (6)	O8^{viii}—Rb4—O12ⁱ	150.99 (7)
O9ⁱ—Rb1—O5ⁱⁱ	110.80 (7)	O3ⁱ—Rb4—O12ⁱ	46.59 (6)
O6ⁱⁱ—Rb1—O5ⁱⁱ	50.90 (6)	O10—Rb4—O12ⁱ	108.45 (7)
O11ⁱⁱⁱ—Rb1—O5ⁱⁱ	97.65 (6)	O14—Rb4—O12ⁱ	76.14 (7)
O2ⁱⁱⁱ—Rb1—O5ⁱⁱ	93.51 (6)	O13ⁱ—Rb4—O12ⁱ	82.09 (7)
O14^{iv}—Rb2—O7^v	113.73 (7)	O8^{viii}—Rb4—O6ⁱ	71.11 (7)
O14^{iv}—Rb2—O9^{vi}	104.59 (7)	O3ⁱ—Rb4—O6ⁱ	53.40 (6)
O7^v—Rb2—O9^{vi}	75.43 (7)	O10—Rb4—O6ⁱ	88.10 (6)
O14^{iv}—Rb2—O2	155.92 (7)	O14—Rb4—O6ⁱ	141.38 (6)
O7^v—Rb2—O2	84.64 (7)	O13ⁱ—Rb4—O6ⁱ	73.59 (6)
O9^{vi}—Rb2—O2	63.56 (7)	O12ⁱ—Rb4—O6ⁱ	99.87 (6)
O14^{iv}—Rb2—O13ⁱⁱ	70.38 (7)	O8^{viii}—Rb4—O5	118.84 (7)
O7^v—Rb2—O13ⁱⁱ	90.22 (7)	O3ⁱ—Rb4—O5	119.85 (6)
O9^{vi}—Rb2—O13ⁱⁱ	161.61 (8)	O10—Rb4—O5	44.51 (6)
O2—Rb2—O13ⁱⁱ	127.42 (7)	O14—Rb4—O5	87.33 (6)
O14^{iv}—Rb2—O8	94.19 (7)	O13ⁱ—Rb4—O5	154.80 (6)
O7^v—Rb2—O8	135.64 (7)	O12ⁱ—Rb4—O5	88.19 (6)
O9^{vi}—Rb2—O8	131.74 (8)	O6ⁱ—Rb4—O5	131.24 (6)
O2—Rb2—O8	81.38 (7)	O8^{viii}—Rb4—O4	113.04 (6)
O13ⁱⁱ—Rb2—O8	66.65 (7)	O3ⁱ—Rb4—O4	124.58 (6)
O14^{iv}—Rb2—O10^v	66.37 (7)	O10—Rb4—O4	88.06 (6)
O7^v—Rb2—O10^v	47.37 (7)	O14—Rb4—O4	43.02 (6)
O9^{vi}—Rb2—O10^v	88.01 (8)	O13ⁱ—Rb4—O4	110.63 (6)
O2—Rb2—O10^v	130.12 (7)	O12ⁱ—Rb4—O4	77.99 (6)
O13ⁱⁱ—Rb2—O10^v	73.72 (7)	O6ⁱ—Rb4—O4	174.76 (6)
O8—Rb2—O10^v	139.93 (7)	O5—Rb4—O4	44.32 (5)
O14^{iv}—Rb2—O9	92.92 (7)	O11—S1—O14	113.92 (14)
O7^v—Rb2—O9	152.12 (7)	O11—S1—O6^{ix}	108.97 (14)
O9^{vi}—Rb2—O9	90.56 (7)	O14—S1—O6^{ix}	111.57 (14)
O2—Rb2—O9	67.50 (7)	O11—S1—O4	109.38 (14)
O13ⁱⁱ—Rb2—O9	107.16 (7)	O14—S1—O4	106.03 (14)
O8—Rb2—O9	43.73 (6)	O6^{ix}—S1—O4	106.66 (13)
O10^v—Rb2—O9	158.07 (7)	O12—S2—O9	113.40 (16)
O14^{iv}—Rb2—O1	144.45 (6)	O12—S2—O8	114.19 (15)
O7^v—Rb2—O1	82.45 (6)	O9—S2—O8	111.50 (16)
O9^{vi}—Rb2—O1	110.23 (7)	O12—S2—O3	104.03 (14)
O2—Rb2—O1	48.93 (6)	O9—S2—O3	105.97 (15)

O13$^{\text{ii}}$—Rb2—O1	78.50 (7)	O8—S2—O3	106.88 (13)
O8—Rb2—O1	56.93 (6)	O13—S3—O7	111.90 (16)
O10$^{\text{v}}$—Rb2—O1	120.93 (6)	O13—S3—O10	112.06 (16)
O9—Rb2—O1	79.98 (6)	O7—S3—O10	111.32 (15)
O11—Rb3—O10$^{\text{ii}}$	152.16 (7)	O13—S3—O5	108.18 (14)
O11—Rb3—O7$^{\text{v}}$	77.41 (7)	O7—S3—O5	108.64 (14)
O10$^{\text{ii}}$—Rb3—O7$^{\text{v}}$	100.02 (7)	O10—S3—O5	104.36 (14)
O11—Rb3—O14$^{\text{vii}}$	138.78 (7)	S2—O3—W1	136.45 (14)
O10$^{\text{ii}}$—Rb3—O14$^{\text{vii}}$	68.51 (7)	S1—O4—W1	134.71 (14)
O7$^{\text{v}}$—Rb3—O14$^{\text{vii}}$	110.14 (7)	S3—O5—W1	139.07 (14)
O11—Rb3—O12$^{\text{i}}$	75.60 (7)	S1$^{\text{ix}}$—O6—W1	130.47 (13)
O10$^{\text{ii}}$—Rb3—O12$^{\text{i}}$	94.76 (7)		

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