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(18-Crown-6)potassium(I) diphenylstibate(−1)

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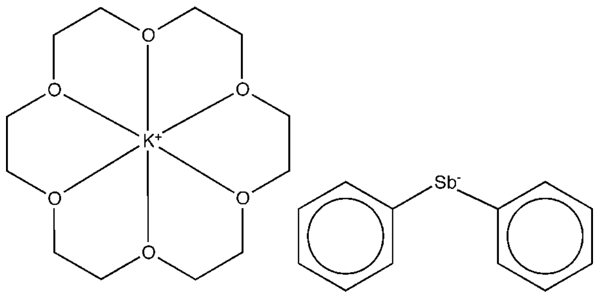
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.003 Å; R factor = 0.023; wR factor = 0.053; data-to-parameter ratio = 17.6.

Red crystals of the title salt, $[K(C_{12}H_{24}O_6)][Sb(C_6H_5)_2]$, were obtained by the reaction of $SbPh_3$, KSnBi and 18-crown-6 in liquid ammonia. The asymmetric unit contains one half of a $[K(18\text{-crown-}6)]^+$ cation and one half of an $SbPh_2^-$ anion, with the central element lying on a twofold axis and a centre of inversion, respectively. In the crystal structure, the sequestered potassium cations show weak interactions with the π-electrons of the phenyl groups of the $SbPh_2^-$ anion [shortest K⋯C distances = 3.190 (2) and 3.441 (2) Å], leading to one-dimensional strands along the crystallographic c axis. These strands are aligned in a pseudo-hexagonal packing perpendicular to the ab plane.

Related literature

For literature focusing on mechanisms of crystallization and intermolecular interactions or diphenylstibide as a nucleophile, see: Desiraju (2007); Ugrinov & Sevov (2003). For a related compound, see Effendy *et al.* (1997).



Experimental

Crystal data

$[K(C_{12}H_{24}O_6)][Sb(C_6H_5)_2]$
M_r = 579.36
Monoclinic, $I2/a$
a = 15.6933 (9) Å
b = 9.2655 (3) Å
c = 19.1321 (10) Å
β = 112.654 (6)°
V = 2567.3 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.27 mm^{-1}
T = 123 K
0.47 × 0.27 × 0.15 mm

Data collection

Agilent SuperNova (Single source at offset, Eos) diffractometer
Absorption correction: analytical [*CrysAlis PRO* (Agilent, 2012), using a multi-faceted crystal model based on expressions derived by Clark & Reid (1995)]
T_{min} = 0.726, T_{max} = 0.863
4194 measured reflections
2592 independent reflections
2297 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.053
S = 1.08
2592 reflections
147 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

Table 1
Selected bond lengths (Å).

Sb1—C1	2.154 (2)	K1—O3	2.8106 (16)
K1—O2	2.7823 (14)	K1—O1	2.7738 (15)

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SUPERFLIP* (Palatinus & Chapuis, 2007); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009; Bourhis *et al.*, 2011).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: FF2129).

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supporting information

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(18-Crown-6)potassium(I) diphenylstibate(−1)

Marina Kaas, Ute Friedrich and Nikolaus Korber

1. Comment

The crystal structure of [K(18-crown-6)]$SbPh_2$ was obtained during the investigations of oxidation processes of SnBi-polyanions in liquid ammonia. The asymmetric unit contains one half of a [K(18-crown-6)]$^+$-cation together with one half of a $SbPh_2^-$ anion. Figure 1 shows these two molecular ions. K—O bond lengths within the complex range from 2.7738 (15) Å to 2.8106 (15) Å. The Sb—C bond lengths of $SbPh_2^-$-anion of 2.154 (2) Å and the previously reported values for $SbPh_3$ of 2.1392 (77) Å - 2.1539 (68) Å (Effendy *et al.*, 1997) are in good agreement. The two phenyl groups of $SbPh_2^-$ exhibit a torsion angle of 28.53 (15)° between C2—C1—Sb1—C1^{ii} and C1—Sb1—C1^{ii}—C2^{ii}. Short K—C distances suggest π-interactions between the alkali metal cation and the phenyl rings. Two carbon atoms of each phenyl ligand are connected to the cation with a hapticity of two and distances of 3.190 (2) Å and 3.441 (2) Å. Due to symmetry, the $SbPh_2^-$-anion acts as a linker molecule between two [K(18-crown-6)]$^+$ complexes that leads to the formation of one dimensional strands along the *c*-axis (Figure 2). The strands are arranged in a hexagonal packing and no further interactions can be found between neighbouring strands (Figure 3).

2. Experimental

KSnBi was prepared from the elements in a high temperature synthesis at 723 K in a sealed tube. 88 mg (0.25 mmol) $SbPh_3$, 203 mg (0.27 mmol) KSnBi and 66 mg (0.25 mmol) 18-crown-6 were dissolved in dried liquid ammonia in a baked-out reaction vessel. Liquid ammonia was dried over potassium metal and condensed using a standard Schlenk line. The mixture was stored at 237 K for crystallization. Crystals appeared as red blocks in a brownish red solution after 4 weeks.

3. Refinement

All H-atoms could be located in the difference map but were positioned with idealized geometry, with *U*iso(H) set to 1.2Ueq of the parent atom. Furthermore there were no irregularities such as dislocation.

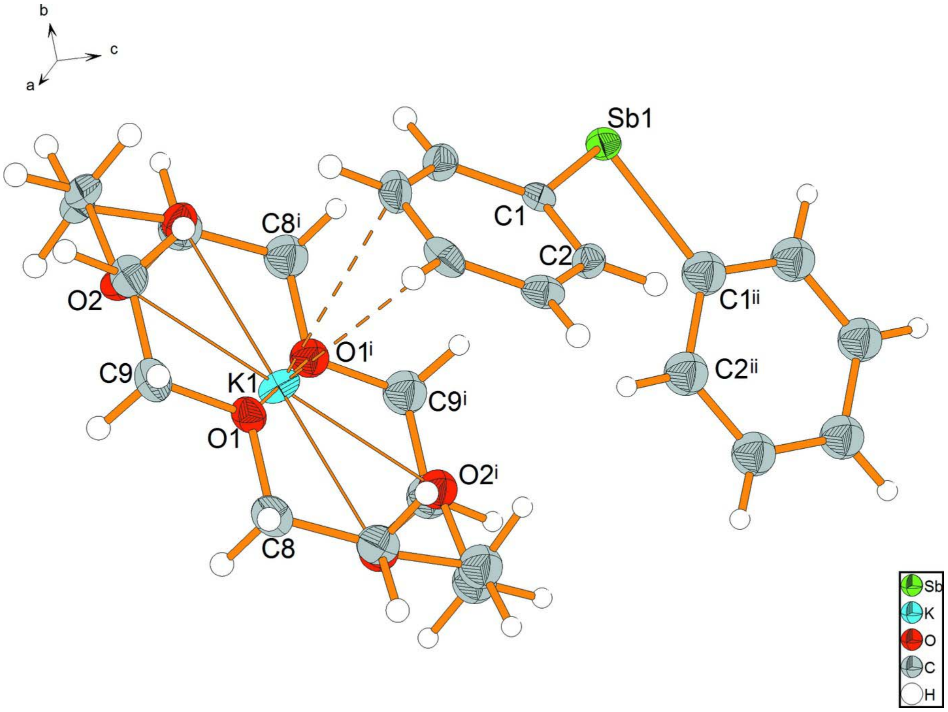

Figure 1

Molecular unit of the title compound with labeling and displacement ellipsoids drawn at the 50% probability level. Symmetry codes: (i) 1.5 - *x*, 1.5 - *y*, 1.5 - *z*, (ii) 1.5 - *x*, *y*, 2 - *z*

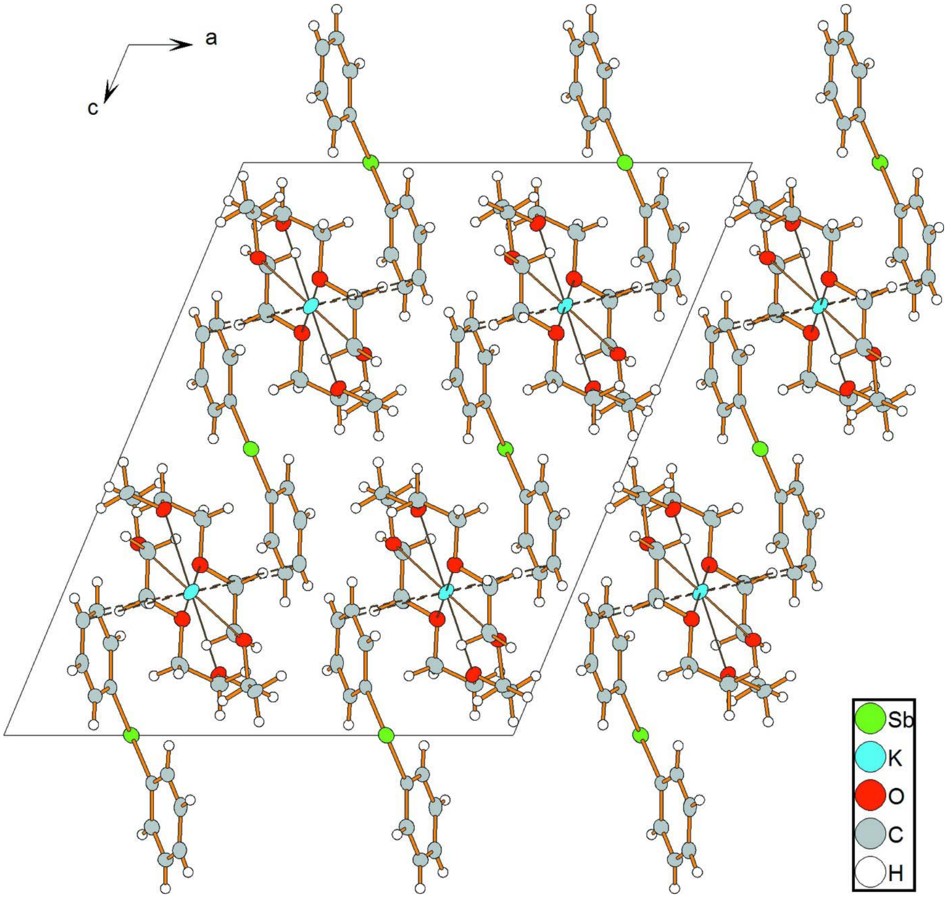

Figure 2

View of the crystal structure showing the one-dimensional strands along the *c*-axis.

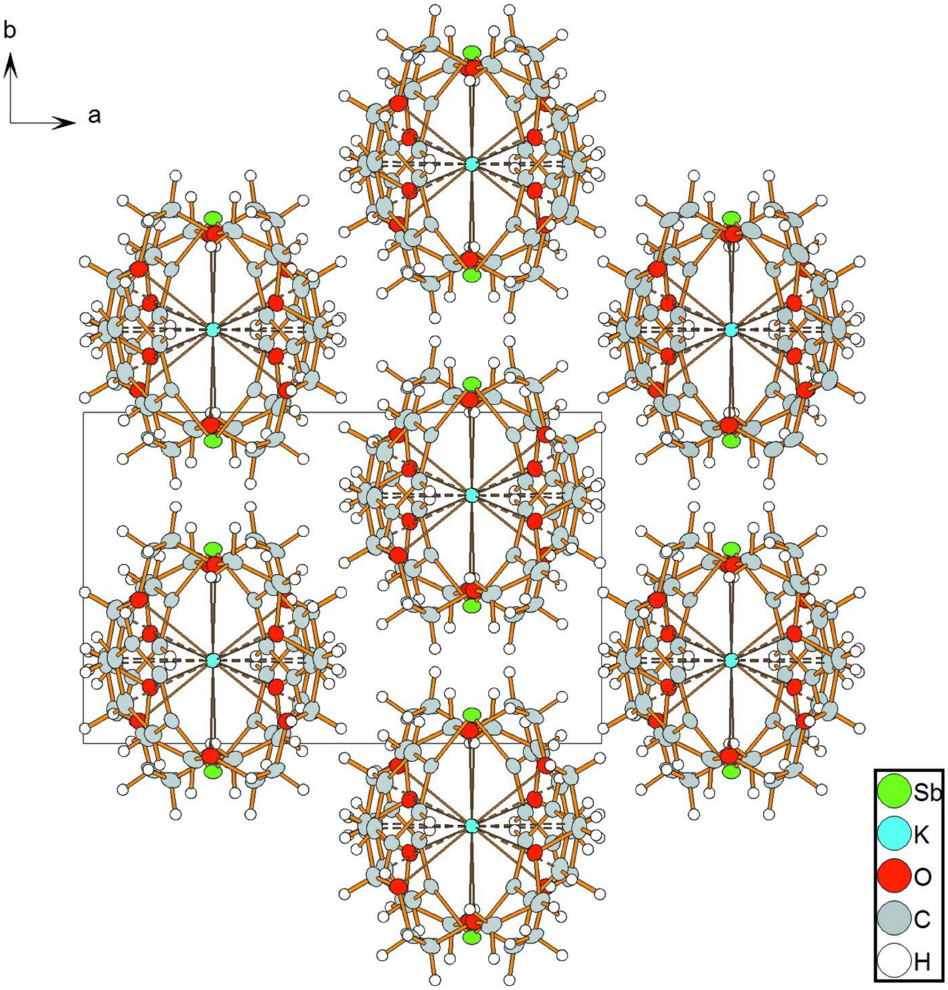

Figure 3

Projection of the crystal structure showing the pseudo-hexagonal arrangement of the strands perpendicular to the *ab*-plane.

(1,4,7,10,13,16-Hexaoxacyclooctadecane)potassium diphenylstibide

Crystal data

$[K(C_{12}H_{24}O_6)][Sb(C_6H_5)_2]$
$M_r = 579.36$
Monoclinic, $I2/a$
$a = 15.6933\ (9)$ Å
$b = 9.2655\ (3)$ Å
$c = 19.1321\ (10)$ Å
$\beta = 112.654\ (6)°$
$V = 2567.3\ (2)$ Å^3
$Z = 4$

$F(000) = 1184$
$D_x = 1.499$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1900 reflections
$\theta = 3.4–28.0°$
$\mu = 1.27$ mm^{-1}
$T = 123$ K
Block, clear reddish red
0.47 × 0.27 × 0.15 mm

Data collection

Agilent SuperNova (Single source at offset, Eos) diffractometer

Graphite monochromator
phi and ω scans

Absorption correction: analytical [*CrysAlis PRO* (Agilent, 2012), using a multi-faceted crystal model based on expressions derived by Clark & Reid (1995)]
$T_{\min} = 0.726$, $T_{\max} = 0.863$
4194 measured reflections
2592 independent reflections
2297 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\max} = 26.4°$, $\theta_{\min} = 3.1°$
$h = -18 \rightarrow 19$
$k = -11 \rightarrow 10$
$l = -15 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.053$
$S = 1.08$
2592 reflections
147 parameters
0 restraints
Primary atom site location: iterative
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.019P)^2 + 0.7904P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.39$ e Å^{-3}
$\Delta\rho_{\min} = -0.56$ e Å^{-3}

Special details

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
Sb1	0.7500	1.08580 (2)	1.0000	0.02163 (8)
K1	0.7500	0.7500	0.7500	0.02190 (15)
O2	0.62864 (10)	0.83061 (17)	0.60590 (8)	0.0237 (3)
O3	0.74551 (10)	1.03820 (17)	0.70288 (8)	0.0241 (4)
O1	0.60734 (10)	0.56706 (17)	0.66609 (8)	0.0225 (3)
C1	0.67008 (14)	0.9316 (2)	0.91614 (11)	0.0191 (5)
C10	0.54293 (15)	0.7554 (3)	0.57688 (12)	0.0278 (5)
H10A	0.5106	0.7774	0.5236	0.033*
H10B	0.5045	0.7855	0.6036	0.033*
C4	0.56109 (15)	0.7424 (3)	0.79930 (12)	0.0279 (5)
H4	0.5263	0.6798	0.7609	0.033*
C11	0.61898 (16)	0.9803 (3)	0.58922 (12)	0.0292 (5)
H11A	0.5781	1.0236	0.6106	0.035*
H11B	0.5924	0.9947	0.5348	0.035*
C3	0.59436 (14)	0.6990 (3)	0.87465 (12)	0.0249 (5)
H3	0.5813	0.6068	0.8870	0.030*
C7	0.66870 (16)	0.3883 (3)	0.76152 (13)	0.0286 (5)
H7A	0.6253	0.4205	0.7831	0.034*
H7B	0.6779	0.2853	0.7701	0.034*
C2	0.64697 (14)	0.7928 (3)	0.93162 (12)	0.0215 (5)
H2	0.6675	0.7622	0.9817	0.026*
C5	0.58069 (15)	0.8799 (3)	0.78264 (12)	0.0283 (6)
H5	0.5577	0.9111	0.7326	0.034*

C9	0.56103 (15)	0.5973 (3)	0.58705 (12)	0.0256 (5)
H9A	0.5032	0.5446	0.5668	0.031*
H9B	0.5991	0.5670	0.5600	0.031*
C8	0.63067 (16)	0.4186 (3)	0.67829 (13)	0.0267 (5)
H8A	0.6763	0.3946	0.6574	0.032*
H8B	0.5762	0.3600	0.6530	0.032*
C12	0.71193 (16)	1.0495 (3)	0.62224 (12)	0.0276 (5)
H12A	0.7541	1.0016	0.6037	0.033*
H12B	0.7075	1.1502	0.6074	0.033*
C6	0.63440 (15)	0.9725 (3)	0.83964 (12)	0.0248 (5)
H6	0.6471	1.0645	0.8267	0.030*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sb1	0.02653 (12)	0.01591 (12)	0.02407 (12)	0.000	0.01153 (9)	0.000
K1	0.0173 (3)	0.0167 (3)	0.0253 (3)	−0.0019 (3)	0.0011 (3)	0.0031 (3)
O2	0.0198 (8)	0.0233 (9)	0.0239 (8)	0.0032 (7)	0.0040 (6)	0.0018 (7)
O3	0.0268 (8)	0.0216 (9)	0.0252 (8)	−0.0006 (7)	0.0114 (7)	0.0043 (7)
O1	0.0253 (8)	0.0221 (9)	0.0194 (7)	−0.0013 (7)	0.0078 (6)	−0.0053 (6)
C1	0.0175 (10)	0.0202 (12)	0.0218 (10)	0.0044 (9)	0.0098 (9)	−0.0001 (9)
C10	0.0185 (11)	0.0411 (16)	0.0187 (11)	0.0017 (11)	0.0016 (9)	−0.0024 (11)
C4	0.0201 (11)	0.0368 (15)	0.0276 (12)	0.0002 (11)	0.0102 (9)	−0.0106 (11)
C11	0.0305 (13)	0.0315 (14)	0.0248 (12)	0.0122 (12)	0.0098 (10)	0.0096 (11)
C3	0.0192 (11)	0.0213 (12)	0.0367 (13)	−0.0001 (10)	0.0134 (10)	−0.0017 (10)
C7	0.0311 (13)	0.0200 (13)	0.0400 (14)	−0.0053 (11)	0.0195 (11)	0.0003 (11)
C2	0.0200 (11)	0.0240 (12)	0.0213 (11)	0.0019 (10)	0.0088 (9)	0.0032 (9)
C5	0.0230 (12)	0.0424 (16)	0.0192 (11)	0.0038 (12)	0.0080 (9)	0.0020 (11)
C9	0.0221 (11)	0.0327 (14)	0.0200 (11)	−0.0061 (11)	0.0058 (9)	−0.0080 (10)
C8	0.0308 (12)	0.0194 (12)	0.0338 (12)	−0.0096 (11)	0.0167 (10)	−0.0076 (10)
C12	0.0356 (13)	0.0236 (13)	0.0286 (12)	0.0084 (11)	0.0178 (10)	0.0090 (10)
C6	0.0245 (12)	0.0264 (13)	0.0251 (11)	0.0020 (11)	0.0114 (9)	0.0041 (10)

Geometric parameters (Å, º)

Sb1—C1	2.154 (2)	C4—C5	1.376 (3)
K1—O2	2.7823 (14)	C11—H11A	0.9700
K1—O3	2.8106 (16)	C11—H11B	0.9700
K1—O1	2.7738 (15)	C11—C12	1.493 (3)
K1—C4	3.441 (2)	C3—H3	0.9300
K1—C5	3.190 (2)	C3—C2	1.390 (3)
O2—C10	1.424 (3)	C7—H7A	0.9700
O2—C11	1.418 (3)	C7—H7B	0.9700
O3—C7^{i}	1.426 (3)	C7—C8	1.496 (3)
O3—C12	1.429 (2)	C2—H2	0.9300
O1—C9	1.431 (2)	C5—H5	0.9300
O1—C8	1.419 (3)	C5—C6	1.389 (3)
C1—C2	1.399 (3)	C9—H9A	0.9700

C1—C6	1.403 (3)	C9—H9B	0.9700
C10—H10A	0.9700	C8—H8A	0.9700
C10—H10B	0.9700	C8—H8B	0.9700
C10—C9	1.490 (3)	C12—H12A	0.9700
C4—H4	0.9300	C12—H12B	0.9700
C4—C3	1.390 (3)	C6—H6	0.9300

C1—Sb1—C1^{ii}	96.89 (12)	O2—C10—H10A	109.9
O2—K1—O2^{i}	180.00 (6)	O2—C10—H10B	109.9
O2^{i}—K1—O3^{i}	60.27 (4)	O2—C10—C9	109.03 (18)
O2^{i}—K1—O3	119.73 (4)	H10A—C10—H10B	108.3
O2—K1—O3^{i}	119.73 (4)	C9—C10—H10A	109.9
O2—K1—O3	60.27 (4)	C9—C10—H10B	109.9
O2—K1—C4	86.78 (5)	K1—C4—H4	95.9
O2—K1—C4^{i}	93.22 (5)	C3—C4—K1	105.73 (13)
O2^{i}—K1—C4	93.22 (5)	C3—C4—H4	120.7
O2^{i}—K1—C4^{i}	86.78 (5)	C5—C4—K1	67.95 (13)
O2—K1—C5	77.81 (5)	C5—C4—H4	120.7
O2—K1—C5^{i}	102.19 (5)	C5—C4—C3	118.7 (2)
O2^{i}—K1—C5^{i}	77.81 (5)	O2—C11—H11A	109.9
O2^{i}—K1—C5	102.19 (5)	O2—C11—H11B	109.9
O3—K1—O3^{i}	180.0	O2—C11—C12	108.82 (18)
O3—K1—C4^{i}	79.08 (5)	H11A—C11—H11B	108.3
O3^{i}—K1—C4^{i}	100.92 (5)	C12—C11—H11A	109.9
O3—K1—C4	100.92 (5)	C12—C11—H11B	109.9
O3^{i}—K1—C4	79.08 (5)	C4—C3—H3	119.8
O3—K1—C5^{i}	102.44 (6)	C2—C3—C4	120.3 (2)
O3^{i}—K1—C5^{i}	77.56 (6)	C2—C3—H3	119.8
O3^{i}—K1—C5	102.44 (6)	O3^{i}—C7—H7A	109.8
O3—K1—C5	77.56 (6)	O3^{i}—C7—H7B	109.8
O1—K1—O2	59.91 (4)	O3^{i}—C7—C8	109.56 (18)
O1—K1—O2^{i}	120.09 (5)	H7A—C7—H7B	108.2
O1^{i}—K1—O2^{i}	59.91 (5)	C8—C7—H7A	109.8
O1^{i}—K1—O2	120.09 (5)	C8—C7—H7B	109.8
O1^{i}—K1—O3	61.31 (4)	C1—C2—H2	118.9
O1—K1—O3^{i}	61.31 (4)	C3—C2—C1	122.2 (2)
O1^{i}—K1—O3^{i}	118.69 (4)	C3—C2—H2	118.9
O1—K1—O3	118.69 (4)	K1—C5—H5	86.5
O1^{i}—K1—O1	180.0	C4—C5—K1	88.49 (14)
O1^{i}—K1—C4	116.26 (5)	C4—C5—H5	119.6
O1—K1—C4^{i}	116.26 (5)	C4—C5—C6	120.8 (2)
O1—K1—C4	63.74 (5)	C6—C5—K1	95.02 (14)
O1^{i}—K1—C4^{i}	63.74 (5)	C6—C5—H5	119.6
O1—K1—C5	78.25 (5)	O1—C9—C10	108.91 (18)
O1^{i}—K1—C5^{i}	78.25 (5)	O1—C9—H9A	109.9
O1^{i}—K1—C5	101.75 (5)	O1—C9—H9B	109.9
O1—K1—C5^{i}	101.75 (5)	C10—C9—H9A	109.9
C4—K1—C4^{i}	180.00 (10)	C10—C9—H9B	109.9

C5—K1—C4	23.57 (6)	H9A—C9—H9B	108.3
C5^{i}—K1—C4^{i}	23.57 (6)	O1—C8—C7	109.22 (18)
C5—K1—C4^{i}	156.43 (6)	O1—C8—H8A	109.8
C5^{i}—K1—C4	156.43 (6)	O1—C8—H8B	109.8
C5^{i}—K1—C5	180.0	C7—C8—H8A	109.8
C10—O2—K1	115.99 (12)	C7—C8—H8B	109.8
C11—O2—K1	117.29 (12)	H8A—C8—H8B	108.3
C11—O2—C10	112.83 (17)	O3—C12—C11	108.62 (18)
C7^{i}—O3—K1	113.46 (12)	O3—C12—H12A	110.0
C7^{i}—O3—C12	111.61 (16)	O3—C12—H12B	110.0
C12—O3—K1	111.65 (13)	C11—C12—H12A	110.0
C9—O1—K1	117.40 (12)	C11—C12—H12B	110.0
C8—O1—K1	113.42 (12)	H12A—C12—H12B	108.3
C8—O1—C9	110.95 (17)	C1—C6—H6	119.0
C2—C1—Sb1	125.20 (15)	C5—C6—C1	122.0 (2)
C2—C1—C6	115.9 (2)	C5—C6—H6	119.0
C6—C1—Sb1	118.81 (17)		

Symmetry codes: (i) $-x+3/2, -y+3/2, -z+3/2$; (ii) $-x+3/2, y, -z+2$.