

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

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

Marina Kaas, Ute Friedrich and Nikolaus Korber\*

Institut für Anorganische Chemie, Universität Regensburg, Universitätsstrasse 31, 93053 Regensburg, Germany

Correspondence e-mail: nikolaus.korber@chemie.uni-regensburg.de

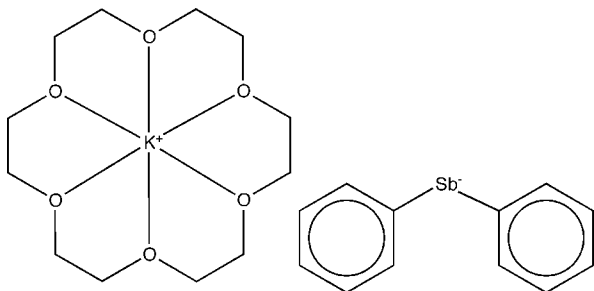
Received 28 May 2014; accepted 6 June 2014

Key indicators: single-crystal X-ray study;  $T = 123$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.023;  $wR$  factor = 0.053; data-to-parameter ratio = 17.6.

Red crystals of the title salt,  $[\text{K}(\text{C}_{12}\text{H}_{24}\text{O}_6)][\text{Sb}(\text{C}_6\text{H}_5)_2]$ , were obtained by the reaction of  $\text{SbPh}_3$ ,  $\text{KSnBi}$  and 18-crown-6 in liquid ammonia. The asymmetric unit contains one half of a  $[\text{K}(\text{18-crown-6})]^+$  cation and one half of an  $\text{SbPh}_2^-$  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  $\pi$ -electrons of the phenyl groups of the  $\text{SbPh}_2^-$  anion [shortest  $\text{K} \cdots \text{C}$  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

 $[\text{K}(\text{C}_{12}\text{H}_{24}\text{O}_6)][\text{Sb}(\text{C}_6\text{H}_5)_2]$   
 $M_r = 579.36$ 

 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<sup>3</sup>  
 $Z = 4$ 

 Mo  $K\alpha$  radiation  
 $\mu = 1.27$  mm<sup>-1</sup>  
 $T = 123$  K  
 $0.47 \times 0.27 \times 0.15$  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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.019$ 

## 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<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.56$  e Å<sup>-3</sup>

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).

UF thanks the Chemical Industry Fund for a scholarship.

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

## References

- Agilent (2012). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.  
 Bourhis, L. J., Dolomanov, O. V., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2011). In preparation.  
 Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.  
 Clark, R. C. & Reid, J. S. (1995). *Acta Cryst.* **A51**, 887–897.  
 Desiraju, G. R. (2007). *Angew. Chem. Int. Ed.* **46**, 8342–8356.  
 Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.  
 Effendy, Grigsby, W.J., Hart, R.D., Raston, C.L., Skelton, B.W. & White, A.H. (1997). *Aust. J. Chem.* **50**, 675–682.  
 Palatinus, L. & Chapuis, G. (2007). *J. Appl. Cryst.* **40**, 786–790.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Ugrinov, A. & Sevov, S. C. (2003). *J. Am. Chem. Soc.* **125**, 14059–14064.

## supporting information

*Acta Cryst.* (2014). E70, m260 [https://doi.org/10.1107/S1600536814013282]

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

**Marina Kaas, Ute Friedrich and Nikolaus Korber**

**S1. Comment**

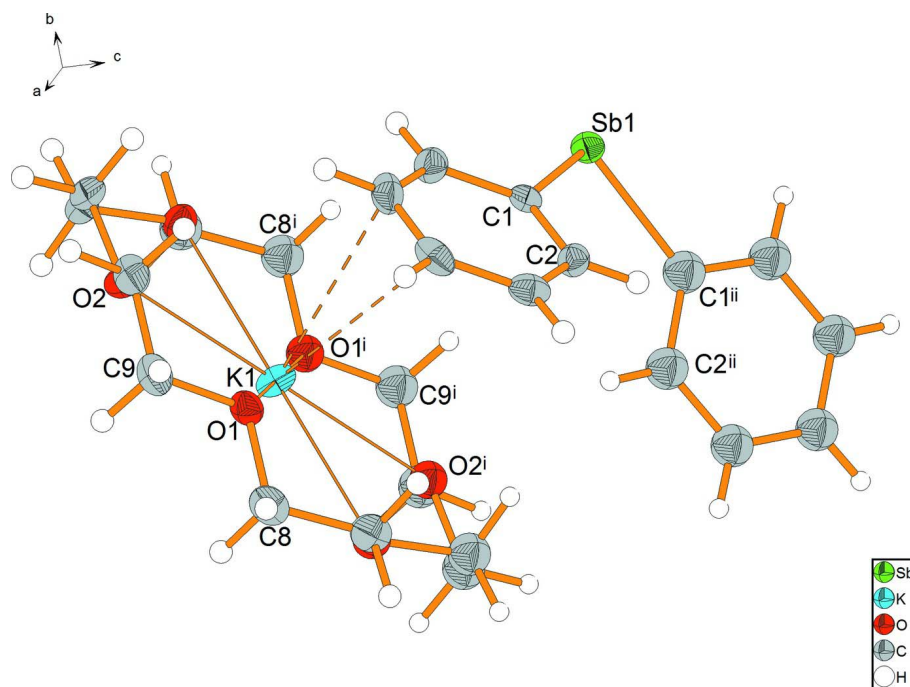
The crystal structure of [K(18-crown-6)]SbPh<sub>2</sub> was obtained during the investigations of oxidation processes of SnBi-polyanions in liquid ammonia. The asymmetric unit contains one half of a [K(18-crown-6)]<sup>+</sup>-cation together with one half of a SbPh<sub>2</sub><sup>-</sup> anion. Figure 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<sub>2</sub><sup>-</sup>-anion of 2.154 (2) Å and the previously reported values for SbPh<sub>3</sub> of 2.1392 (77) Å - 2.1539 (68) Å (Effendy *et al.*, 1997) are in good agreement. The two phenyl groups of SbPh<sub>2</sub><sup>-</sup> exhibit a torsion angle of 28.53 (15)° between C2—C1—Sb1—C1<sup>ii</sup> and C1—Sb1—C1<sup>ii</sup>—C2<sup>ii</sup>. Short K—C distances suggest  $\pi$ -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<sub>2</sub><sup>-</sup>-anion acts as a linker molecule between two [K(18-crown-6)]<sup>+</sup> 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).

**S2. 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<sub>3</sub>, 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.

**S3. Refinement**

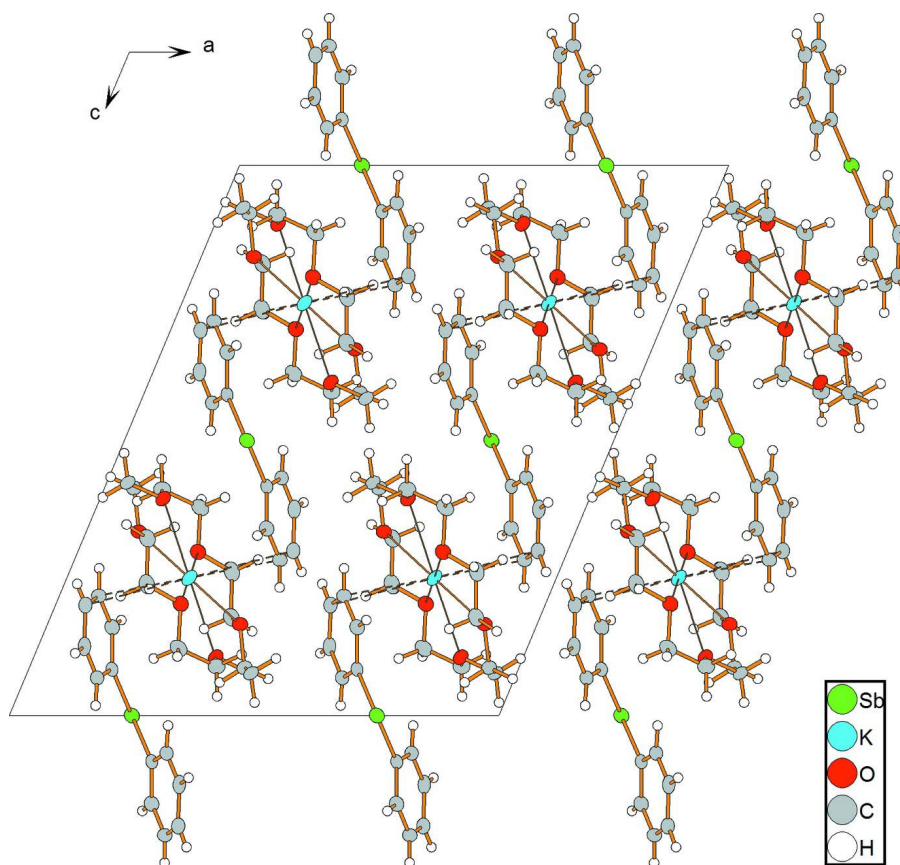
All H-atoms could be located in the difference map but were positioned with idealized geometry, with *U*<sub>iso</sub>(H) set to 1.2U<sub>eq</sub> of the parent atom. Furthermore there were no irregularities such as dislocation.



**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.

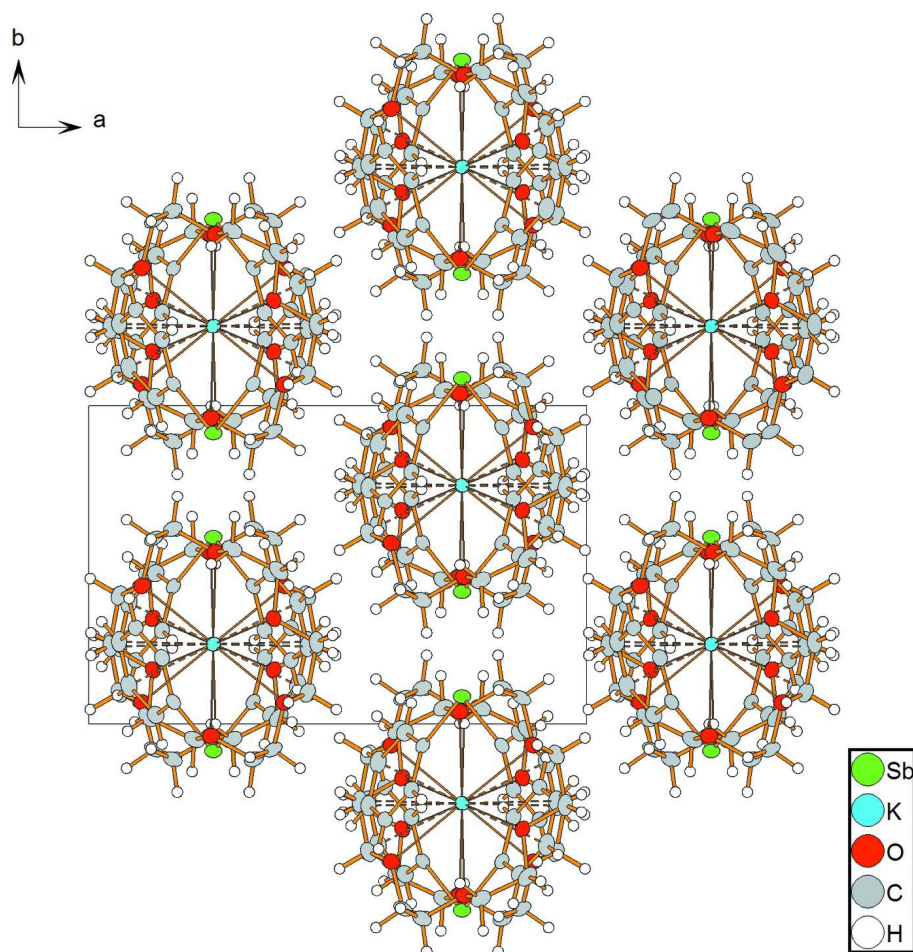


Figure 3

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

$[\text{K}(\text{C}_{12}\text{H}_{24}\text{O}_6)][\text{Sb}(\text{C}_6\text{H}_5)_2]$

$M_r = 579.36$

Monoclinic, *I2/a*

$a = 15.6933 (9) \text{ \AA}$

$b = 9.2655 (3) \text{ \AA}$

$c = 19.1321 (10) \text{ \AA}$

$\beta = 112.654 (6)^\circ$

$V = 2567.3 (2) \text{ \AA}^3$

$Z = 4$

$F(000) = 1184$

$D_x = 1.499 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1900 reflections

$\theta = 3.4\text{--}28.0^\circ$

$\mu = 1.27 \text{ mm}^{-1}$

$T = 123 \text{ K}$

Block, clear reddish red

$0.47 \times 0.27 \times 0.15 \text{ mm}$

#### Data collection

Agilent SuperNova (Single source at offset,  
Eos)

diffractometer

Graphite monochromator

phi and  $\omega$  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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.019$

$\theta_{\text{max}} = 26.4^\circ$ ,  $\theta_{\text{min}} = 3.1^\circ$   
 $h = -18 \rightarrow 19$   
 $k = -11 \rightarrow 10$   
 $l = -15 \rightarrow 23$

*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  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.39 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.56 \text{ e } \text{\AA}^{-3}$

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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, °)*

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 <sup>i</sup>	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 <sup>ii</sup>	96.89 (12)	O2—C10—H10A	109.9
O2—K1—O2 <sup>i</sup>	180.00 (6)	O2—C10—H10B	109.9
O2 <sup>i</sup> —K1—O3 <sup>i</sup>	60.27 (4)	O2—C10—C9	109.03 (18)
O2 <sup>i</sup> —K1—O3	119.73 (4)	H10A—C10—H10B	108.3
O2—K1—O3 <sup>i</sup>	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 <sup>i</sup>	93.22 (5)	C3—C4—K1	105.73 (13)
O2 <sup>i</sup> —K1—C4	93.22 (5)	C3—C4—H4	120.7
O2 <sup>i</sup> —K1—C4 <sup>i</sup>	86.78 (5)	C5—C4—K1	67.95 (13)
O2—K1—C5	77.81 (5)	C5—C4—H4	120.7
O2—K1—C5 <sup>i</sup>	102.19 (5)	C5—C4—C3	118.7 (2)
O2 <sup>i</sup> —K1—C5 <sup>i</sup>	77.81 (5)	O2—C11—H11A	109.9
O2 <sup>i</sup> —K1—C5	102.19 (5)	O2—C11—H11B	109.9
O3—K1—O3 <sup>i</sup>	180.0	O2—C11—C12	108.82 (18)
O3—K1—C4 <sup>i</sup>	79.08 (5)	H11A—C11—H11B	108.3
O3 <sup>i</sup> —K1—C4 <sup>i</sup>	100.92 (5)	C12—C11—H11A	109.9
O3—K1—C4	100.92 (5)	C12—C11—H11B	109.9
O3 <sup>i</sup> —K1—C4	79.08 (5)	C4—C3—H3	119.8
O3—K1—C5 <sup>i</sup>	102.44 (6)	C2—C3—C4	120.3 (2)
O3 <sup>i</sup> —K1—C5 <sup>i</sup>	77.56 (6)	C2—C3—H3	119.8
O3 <sup>i</sup> —K1—C5	102.44 (6)	O3 <sup>i</sup> —C7—H7A	109.8
O3—K1—C5	77.56 (6)	O3 <sup>i</sup> —C7—H7B	109.8
O1—K1—O2	59.91 (4)	O3 <sup>i</sup> —C7—C8	109.56 (18)
O1—K1—O2 <sup>i</sup>	120.09 (5)	H7A—C7—H7B	108.2
O1 <sup>i</sup> —K1—O2 <sup>i</sup>	59.91 (5)	C8—C7—H7A	109.8
O1 <sup>i</sup> —K1—O2	120.09 (5)	C8—C7—H7B	109.8
O1 <sup>i</sup> —K1—O3	61.31 (4)	C1—C2—H2	118.9
O1—K1—O3 <sup>i</sup>	61.31 (4)	C3—C2—C1	122.2 (2)
O1 <sup>i</sup> —K1—O3 <sup>i</sup>	118.69 (4)	C3—C2—H2	118.9
O1—K1—O3	118.69 (4)	K1—C5—H5	86.5
O1 <sup>i</sup> —K1—O1	180.0	C4—C5—K1	88.49 (14)
O1 <sup>i</sup> —K1—C4	116.26 (5)	C4—C5—H5	119.6
O1—K1—C4 <sup>i</sup>	116.26 (5)	C4—C5—C6	120.8 (2)
O1—K1—C4	63.74 (5)	C6—C5—K1	95.02 (14)
O1 <sup>i</sup> —K1—C4 <sup>i</sup>	63.74 (5)	C6—C5—H5	119.6
O1—K1—C5	78.25 (5)	O1—C9—C10	108.91 (18)
O1 <sup>i</sup> —K1—C5 <sup>i</sup>	78.25 (5)	O1—C9—H9A	109.9
O1 <sup>i</sup> —K1—C5	101.75 (5)	O1—C9—H9B	109.9
O1—K1—C5 <sup>i</sup>	101.75 (5)	C10—C9—H9A	109.9
C4—K1—C4 <sup>i</sup>	180.00 (10)	C10—C9—H9B	109.9
C5—K1—C4	23.57 (6)	H9A—C9—H9B	108.3
C5 <sup>i</sup> —K1—C4 <sup>i</sup>	23.57 (6)	O1—C8—C7	109.22 (18)
C5—K1—C4 <sup>i</sup>	156.43 (6)	O1—C8—H8A	109.8



---

C5 <sup>i</sup> —K1—C4	156.43 (6)	O1—C8—H8B	109.8
C5 <sup>i</sup> —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 <sup>i</sup> —O3—K1	113.46 (12)	O3—C12—H12A	110.0
C7 <sup>i</sup> —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)  $-x+3/2, y, -z+2$ .