

# Crystal structures of two solvates of (18-crown-6)-potassium acetate

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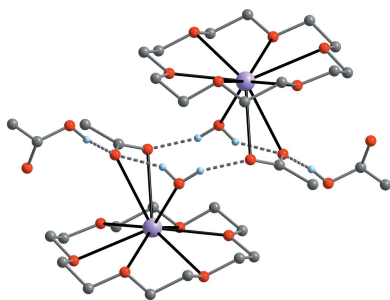
**Keywords:** crystal structure; potassium; crown ether; 18-crown-6; acetate; hydrate; hydro-acetate; hydrogen bridge bonds.**CCDC references:** 1513648; 1513649**Supporting information:** this article has supporting information at journals.iucr.org/e

The crystal and molecular structures of two solvated forms of [K(18c6)]OAc (18c6 = 18-crown-6 = 1,4,7,10,13,16-hexaoxacyclooctadecane and OAc = acetate) were determined by single-crystal X-ray diffraction, namely (acetato- $\kappa^2O,O'$ )(1,4,7,10,13,16-hexaoxacyclooctadecane- $\kappa^6O$ )potassium dihydrate, [K(CH<sub>3</sub>COO)(C<sub>12</sub>H<sub>24</sub>O<sub>6</sub>)]·2H<sub>2</sub>O (**1**) and (acetato- $\kappa^2O,O'$ )aqua(1,4,7,10,13,16-hexaoxacyclooctadecane- $\kappa^6O$ )potassium acetic acid monosolvate [K(CH<sub>3</sub>COO)(C<sub>12</sub>H<sub>24</sub>O<sub>6</sub>)(H<sub>2</sub>O)]·CH<sub>3</sub>COOH (**2**). In both compounds, the acetate anion is bonded to the potassium ion in a chelating fashion and the metal atom is consequently slightly displaced from the O<sub>6</sub> plane of the crown ether. In the crystals, O—H···O hydrogen bonds lead to a polymeric ladder structure in the dihydrate **1**, while the acetic acid hydrate **2** features inversion dimers.

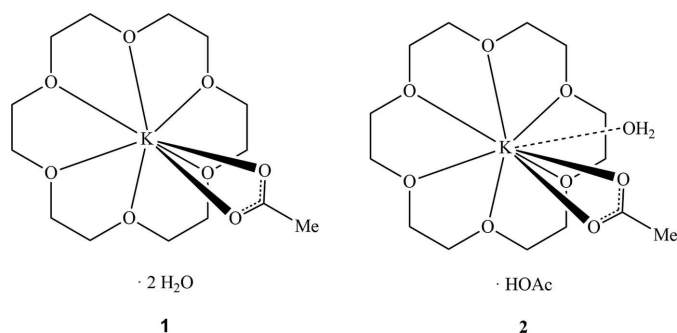
## 1. Chemical context

As a result of the macrocyclic ether 1,4,7,10,13,16-hexaoxacyclooctadecane ('18-crown-6') being a hexadentate ligand that is highly specific for the potassium cation, it is frequently used to manipulate the properties of various potassium compounds. On the one hand, the [K(18c6)]<sup>+</sup> cation (18c6 = 18-crown-6) is a powerful tool to crystallize large anions with the objective to make them accessible for single-crystal structure determination. Thus, the crystal structures of numerous anionic complex compounds have been observed from their [K(18c6)]<sup>+</sup> salts, e.g. [HPMo<sub>12</sub>O<sub>40</sub>]<sup>4-</sup> (Neier *et al.*, 1995) and [HgR<sup>f</sup><sub>2</sub>X]<sup>-</sup> (R<sup>f</sup> = CF<sub>3</sub>, C<sub>6</sub>F<sub>5</sub>, X = Br, I; Schulz *et al.*, 2003) to mention just two examples among many. The same applies to a broad ensemble of unusual non-metal anions such as I<sub>3</sub><sup>-</sup> (Sievert *et al.*, 1996) and the radical species C<sub>2</sub>N<sub>4</sub>S<sub>2</sub><sup>-</sup> (Makarov *et al.*, 2005). Moreover, since the early days of crown-ether chemistry, 18-crown-6 has been used to enhance the solubility of reactive potassium salts in organic media, e.g. KMnO<sub>4</sub> (Doheny & Ganem, 1980). [K(18c6)]OAc (OAc = acetate) has been shown to be useful as an acetylation agent for alkyl halides (Liotta *et al.*, 1974), and over the past few years 'CECILS' (crown ether complex cation ionic liquids) such as [K(18c6)]OAc and [K(18c6)]OH gained in importance as basic catalysts for various organic transformations (e.g. Song *et al.*, 2011; Abaszadeh & Seifi, 2015).

In view of the broad application of 18-crown-6-complexed potassium acetate, [K(18c6)]OAc, it is surprising that the crystal structure of this simple compound has never been determined. In this paper we present the structures of two



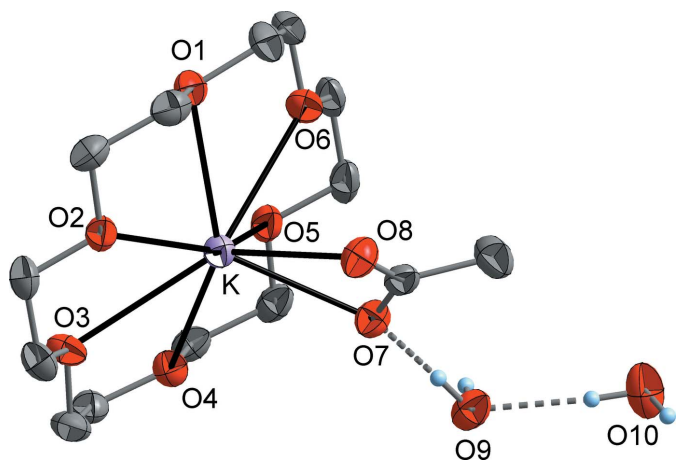
solvated forms thereof, namely the dihydrate,  $[\text{K}(\text{18c6})]\text{OAc}\cdot 2\text{H}_2\text{O}$  (**1**), and the acetic acid hydrate,  $[\text{K}(\text{18c6})]\text{OAc}\cdot\text{HOAc}\cdot\text{H}_2\text{O}$  (**2**).



## 2. Structural commentary

Both title compounds crystallize in the monoclinic space group  $P2_1/c$  with one formula unit of  $[\text{K}(\text{18c6})]\text{OAc}$  and two solvent molecules in the asymmetric unit. In the dihydrate **1** (Fig. 1), the potassium atom is coordinated by the crown ether ligand in a slightly unsymmetrical hexadentate mode with  $\text{K}-\text{O}$  distances (Table 1) ranging from 2.8248 (13) to 2.9684 (11) Å and a median value of 2.922 Å. The acetate counter-ion is attached to potassium in a chelating coordination mode where the  $\text{K}-\text{O}$  distances are significantly different with 2.6992 (11) (K–O7) and 2.8861 (11) Å (K–O8). As a result of the additional coordination of the acetate ion, the potassium ion is slightly displaced from the crown ether  $\text{O}_6$  plane. The two water molecules do not coordinate to the potassium ion and interact *via*  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds (see *Supramolecular features* section).

By contrast, in the acetic acid hydrate **2** (Fig. 2) the coordination number of the potassium atom is raised to nine by a coordinating water molecule and consequently the  $\text{K}-\text{O}$  bonds (Table 2) to the acetate ligand are significantly elongated to 2.9562 (16) (K–O8) and 3.0303 (19) Å (K–O7).



**Figure 1**  
The molecular structure of compound **1**, with displacement ellipsoids drawn at the 50% probability level. C-bound H atoms have been omitted for clarity.

**Table 1**  
Selected bond lengths (Å) for **1**.

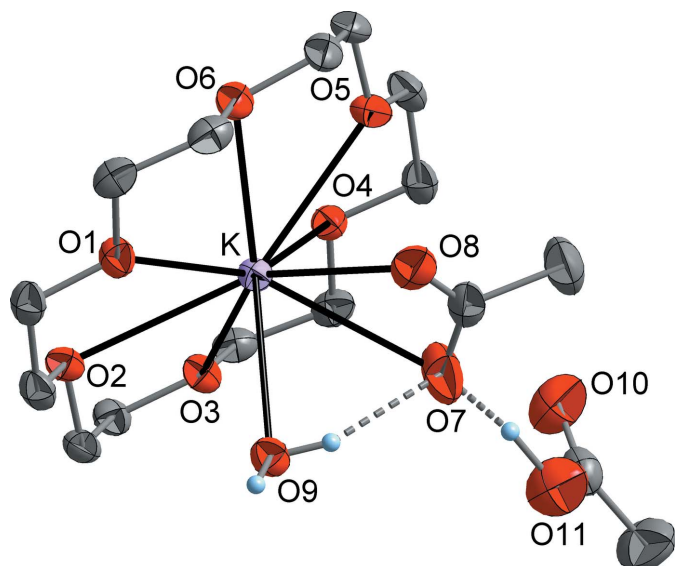
K–O1	2.9684 (11)	K–O5	2.9506 (12)
K–O2	2.8649 (10)	K–O6	2.8248 (13)
K–O3	2.9244 (11)	K–O7	2.6992 (11)
K–O4	2.9380 (11)	K–O8	2.8861 (11)

**Table 2**  
Selected bond lengths (Å) for **2**.

K–O1	2.7861 (12)	K–O6	2.9435 (13)
K–O2	3.0045 (13)	K–O7	3.0303 (19)
K–O3	2.8510 (12)	K–O8	2.9562 (16)
K–O4	3.0337 (13)	K–O9	2.7855 (14)
K–O5	2.9019 (13)		

Moreover, in compound **2** the coordination of the 18c6 ligand is more unsymmetrical than in compound **1** [ $\text{K}-\text{O} = 2.7855$  (14)– $3.0337$  (13) Å], but the average  $\text{K}-\text{O}$  distance is virtually identical at 2.920 Å. In general, the geometry of the  $[\text{K}(\text{18c6})]\text{OAc}$  ion pair is not fundamentally influenced by the additional water ligand. Thus, the angle between the  $\text{KO}_2\text{C}$ (acetate) plane and the  $\text{O}_6$  plane of the 18c6 ligand is similar in both compounds [**1**: 68 (1), **2** 64 (1)°]. The same applies to the displacement of the potassium ion from the  $\text{O}_6$  centroid, which is 0.080 Å in **1** and 0.082 Å in **2**.

The geometry of the  $[\text{K}(\text{18c6})]\text{OAc}$  ion pair in the title compounds fits well with other  $[\text{K}(\text{18c6})]^+$  salts with coordinating anions, *e.g.* the picrate  $[\text{K}(\text{18c6})]\text{O}-\text{C}_6\text{H}_2-2,4,6-(\text{NO}_2)_3$  [ $\text{K}-\text{O} = 2.862$  (4)– $2.989$  (4) Å,  $\text{K}-\text{centroid}(\text{O}_6)$  0.0892 (1) Å; Barnes & Collard, 1988) and the triflate  $[\text{K}(\text{18c6})]\text{OSO}_2\text{CF}_3$  [ $\text{K}-\text{O} = 2.765$  (4)– $2.853$  (4) Å,  $\text{K}-\text{centroid}(\text{O}_6)$  0.043 Å; Mandai *et al.*, 2015). By contrast, in numerous other compounds the potassium ion is situated exactly in the center of the macrocycle and is coordinated symmetrically by the six crown ether O atoms. Since this case resembles the situation in



**Figure 2**  
The molecular structure of compound **2**, with displacement ellipsoids drawn at the 50% probability level. C-bound H atoms have been omitted for clarity.

**Table 3**  
 Hydrogen-bond geometry (Å, °) for (1).

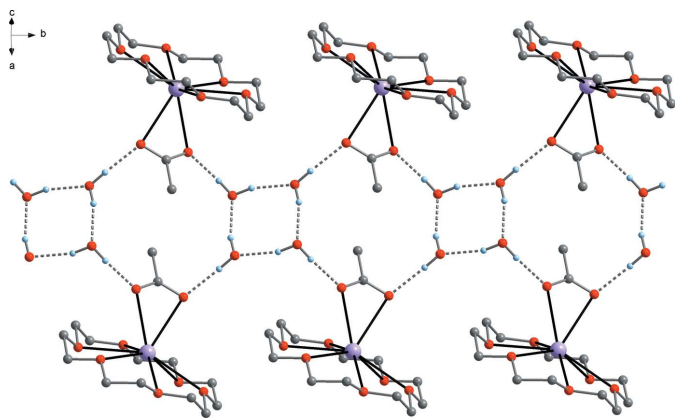
$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O9-H1\cdots O10^i$	0.94 (1)	1.88 (1)	2.790 (3)	165 (4)
$O9-H2\cdots O7^i$	0.93 (1)	1.81 (2)	2.741 (3)	175 (4)
$O10-H3\cdots O8^{ii}$	0.93 (1)	1.88 (2)	2.810 (3)	175 (4)
$O10-H4\cdots O9$	0.93 (1)	1.92 (1)	2.827 (3)	166 (4)

 Symmetry codes: (i)  $-x+1, -y+1, -z$ ; (ii)  $x, y+1, z$ .

isolated  $[K(18c6)]^+$  ions, it has been frequently observed in salts with weakly coordinating anions such as thiocyanate [ $K-O = 2.768(1)$ – $2.836(1)$  Å, median 2.805 Å] and hexafluoridophosphate [ $K-O = 2.791(2)$ – $2.825(5)$  Å, median 2.809 Å], where the anions are weakly attached to the potassium ion symmetrically from both sides of the  $[K(18c6)]^+$  cation (Mandai *et al.*, 2015). Of course, there are also many intermediate cases such as the halidomercurates  $[K(18c6)][Hg(CF_3)_2X]$  ( $X = Br, I$ ; Schulz *et al.*, 2003). Herein the cation–anion interactions are weak in general, but the  $K\cdots X$  interaction is a little stronger than the  $K\cdots F$  interaction on the opposite side of the  $[K(18c6)]^+$  cation and the potassium ion is therefore moved slightly out of the macrocycle [*e.g.*  $X = I$ :  $K-O = 2.768(6)$ – $2.895(6)$  Å,  $K$ -centroid( $O_6$ ) = 0.020 Å].

### 3. Supramolecular features

In both title compounds, both acetate oxygen atoms O7 and O8 are involved in hydrogen bonding (Table 3). In the case of compound **1** (Fig. 3), two hydrogen atoms of adjacent water molecules donate to the acetate ligand with slightly different  $O\cdots O$  distances of 2.741 (3) Å ( $O7\cdots O9$  including H2) and 2.810 (3) Å ( $O8\cdots O10$  including H3). Through the oxygen atom (O9, O10) and the second hydrogen atom (H1, H4) of each water molecule, a cyclic  $(H_2O)_4$  moiety is formed with similar  $O\cdots O$  distances of 2.790 (3) Å ( $O9\cdots O10$  including H1) and 2.827 (3) Å ( $O9\cdots O10'$  including H4'). By this interconnection of  $[K(18c6)(OAc)]$  moieties and water molecules, a double-stranded polymeric structure is formed. Each


**Figure 3**  
 Representation of the polymeric supramolecular structure of compound **1** linked by  $O-H\cdots O$  hydrogen bonds. The double-stranded chain extends along the crystallographic  $b$ -axis direction.

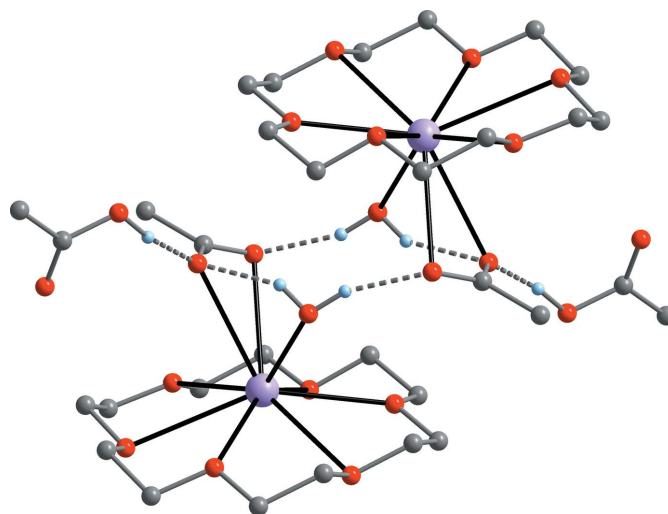
**Table 4**  
 Hydrogen-bond geometry (Å, °) for (2).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O9-H1\cdots O7$	0.94 (1)	1.93 (2)	2.785 (7)	151 (9)
$O9-H2\cdots O8^i$	0.94 (1)	1.89 (2)	2.786 (7)	160 (9)
$O11-H3\cdots O7$	1.08 (5)	1.43 (5)	2.513 (6)	168 (5)

 Symmetry code: (i)  $-x, -y+1, -z+1$ .

chain is characterized by repeating  $O(H)-H\cdots O-C(Me)-O\cdots H-O-H$  units, and through interconnection of the two chains a ladder-like architecture with alternating  $C_2O_8H_4$  14-membered rings and  $O_4H_4$  eight-membered rings is built. The strength of the hydrogen bonds between the water molecules and acetate ions is similar to those observed in other hydrated metal acetates, *e.g.*  $[Zn(OAc)_2(Diap)_2]\cdot H_2O$  [ $Diap = cyclo-C_4H_{10}N_2C=S$ ;  $O\cdots O = 2.773(2)$ – $2.814(1)$  Å; Beheshti *et al.*, 2007] and  $[[Na_2Cu(OAc)_4(H_2O)\cdot H_2O]_n]$  [ $O\cdots O = 2.764(4)$ – $2.944(8)$  Å; Li *et al.*, 2010].

In the acetic acid hydrate **2** (Fig. 4), the acetate ligand accepts two  $O-H\cdots O$  hydrogen bonds (Table 4) from water molecules with very similar  $O\cdots O$  distances, an intramolecular one to the  $K$ -coordinating water molecule [ $O7\cdots O9 = 2.785(7)$  Å, including H1] and an intermolecular one to an adjacent  $[K(18c6)(OAc)(H_2O)]$  moiety [ $O8\cdots O9' = 2.786(7)$  Å, including H2']. In addition, one of the acetate oxygen atoms is attached to the acetic acid molecule with a considerably stronger  $O-H\cdots O$  bond [ $O7\cdots O11 = 2.513(6)$  Å, including H3]. The strength of this hydrogen bond between the acetic acid molecule and acetate ion is comparable with that observed in non-complexed  $KOAc\cdot HOAc$  [ $O\cdots O = 2.476(8)$  Å; Currie, 1972] and in  $NaOAc\cdot HOAc$  [ $O\cdots O = 2.48(1)$  Å; Barrow *et al.*, 1975]. Neither of the oxygen atoms of the acetic acid moiety in compound **2** (O10, O11) are involved in hydrogen bonding and consequently the supramolecular structure is simpler than that of compound **1**.


**Figure 4**  
 Representation of the dimeric supramolecular structure of compound **2** arising from  $O-H\cdots O$  hydrogen bonding.

**Table 5**  
Experimental details.

	1	2
<b>Crystal data</b>		
Chemical formula	[K(C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> )(C <sub>12</sub> H <sub>24</sub> O <sub>6</sub> )]·2H <sub>2</sub> O	[K(C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> )(C <sub>12</sub> H <sub>24</sub> O <sub>6</sub> )(H <sub>2</sub> O)]·C <sub>2</sub> H <sub>4</sub> O <sub>2</sub>
<i>M<sub>r</sub></i>	398.49	440.52
Crystal system, space group	Monoclinic, <i>P</i> <sub>2</sub> <sub>1</sub> / <i>c</i>	Monoclinic, <i>P</i> <sub>2</sub> <sub>1</sub> / <i>c</i>
Temperature (K)	173	200
<i>a</i> , <i>b</i> , <i>c</i> (Å)	11.683 (2), 8.594 (2), 20.083 (4)	11.3233 (1), 8.5450 (1), 23.3869 (3)
$\beta$ (°)	100.59 (3)	99.053 (1)
<i>V</i> (Å <sup>3</sup> )	1982.1 (7)	2234.67 (4)
<i>Z</i>	4	4
Radiation type	Mo <i>K</i> $\alpha$	Mo <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	0.31	0.29
Crystal size (mm)	0.60 × 0.40 × 0.30	0.40 × 0.40 × 0.20
<b>Data collection</b>		
Diffractometer	Bruker SMART CCD	Bruker SMART CCD
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2001)	Multi-scan ( <i>SADABS</i> ; Bruker, 2001)
<i>T</i> <sub>min</sub> – <i>T</i> <sub>max</sub>	0.834, 0.912	0.893, 0.945
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	11876, 4316, 3626	13151, 4846, 3833
<i>R</i> <sub>int</sub>	0.025	0.035
( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.639	0.639
<b>Refinement</b>		
<i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.031, 0.080, 1.04	0.041, 0.104, 1.05
No. of reflections	4316	4846
No. of parameters	244	268
No. of restraints	4	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ , $\Delta\rho_{\min}$ (e Å <sup>-3</sup> )	0.19, -0.25	0.43, -0.31

Computer programs: *SMART* and *SAINT* (Bruker, 2001), *SHELXS97* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015), *DIAMOND* (Brandenburg, 1999) and *pubCIF* (Westrip, 2010).

Namely, through interconnection of two [K(18c6)(OAc)(H<sub>2</sub>O)] moieties by the aforementioned H<sub>2</sub>O···OAc bridges, a dimeric structure with a centrosymmetric C<sub>2</sub>O<sub>6</sub>H<sub>4</sub> ring is present.

#### 4. Database survey

For other structurally characterized salts with the [K(18c6)]<sup>+</sup> cation, see: Neier *et al.* (1995), Sievert *et al.* (1996), Schulz *et al.* (2003), Makarov *et al.* (2005) and Mandai *et al.* (2015). For a review of metal complexes with crown ethers, see: Dalley (1978) and Shono (1994).

For other structurally characterized hydrates and acetic acid solvates of metal acetates, see: Currie (1972), Barrow *et al.* (1975), Beheshti *et al.* (2007) and Li *et al.* (2010).

#### 5. Synthesis and crystallization

Single crystals of the title compounds were obtained by slow evaporation of a solution of commercial available potassium acetate in the presence of an equimolar amount of 18-crown-6 in water (**1**) or in diluted acetic acid (**2**).

#### 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. All H atoms were fixed geome-

trically and refined using a riding model with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C). C–H distances in CH<sub>3</sub> groups were constrained to 0.98 Å and those in CH<sub>2</sub> groups to 0.99 Å. Methyl H atoms were allowed to rotate around the C–C vector (AFIX 137 in *SHELXL*). O–H distances within H<sub>2</sub>O molecules were restrained to 0.96 Å (DFIX restraint in *SHELXL*; the s.u. applied was 0.01 Å), while the coordinates of the HOAc hydrogen atom H3 in compound **2** was refined freely.

#### Acknowledgements

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## supporting information

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## Crystal structures of two solvates of (18-crown-6)potassium acetate

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## Computing details

For both compounds, data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *publCIF* (Westrip, 2010).

(1) (Acetato- $\kappa^2O,O'$ )(1,4,7,10,13,16-hexaoxacyclooctadecane- $\kappa^6O$ )potassium dihydrate

## Crystal data

$[\text{K}(\text{C}_2\text{H}_3\text{O}_2)(\text{C}_{12}\text{H}_{24}\text{O}_6)] \cdot 2\text{H}_2\text{O}$

$M_r = 398.49$

Monoclinic,  $P2_1/c$

$a = 11.683$  (2) Å

$b = 8.594$  (2) Å

$c = 20.083$  (4) Å

$\beta = 100.59$  (3)°

$V = 1982.1$  (7) Å<sup>3</sup>

$Z = 4$

$F(000) = 856$

$D_x = 1.335$  Mg m<sup>-3</sup>

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

Cell parameters from 100 reflections

$\theta = 2.0$ – $27.5$ °

$\mu = 0.31$  mm<sup>-1</sup>

$T = 173$  K

Block, colorless

$0.60 \times 0.40 \times 0.30$  mm

## Data collection

Bruker SMART CCD  
diffractometer

Radiation source: fine-focus sealed tube

$\omega$  scans

Absorption correction: multi-scan  
(SADABS; Bruker, 2001)

$T_{\min} = 0.834$ ,  $T_{\max} = 0.912$

11876 measured reflections

4316 independent reflections

3626 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 27.0$ °,  $\theta_{\min} = 1.8$ °

$h = -14$ → $14$

$k = -10$ → $10$

$l = -25$ → $12$

## Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.031$

$wR(F^2) = 0.080$

$S = 1.04$

4316 reflections

244 parameters

4 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: mixed

H atoms treated by a mixture of independent  
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0364P)^2 + 0.3159P]$

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

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

$\Delta\rho_{\max} = 0.19$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.25$  e Å<sup>-3</sup>

Extinction correction: SHELXL2016  
(Sheldrick, 2015),

$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0102 (9)



*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>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.19215 (12)	-0.24843 (16)	0.26697 (7)	0.0406 (3)
H1A	0.221033	-0.313862	0.307113	0.049*
H1B	0.230318	-0.283302	0.229444	0.049*
C2	0.06322 (12)	-0.26499 (16)	0.24703 (7)	0.0382 (3)
H2A	0.041712	-0.376538	0.244126	0.046*
H2B	0.024397	-0.215362	0.281429	0.046*
C3	-0.09719 (11)	-0.19603 (16)	0.16312 (7)	0.0374 (3)
H3A	-0.133676	-0.141690	0.197352	0.045*
H3B	-0.124628	-0.305238	0.160118	0.045*
C4	-0.13129 (12)	-0.11823 (15)	0.09584 (7)	0.0391 (3)
H4A	-0.091094	-0.168296	0.062154	0.047*
H4B	-0.216334	-0.127743	0.079787	0.047*
C5	-0.14346 (12)	0.13078 (17)	0.04410 (7)	0.0403 (3)
H5A	-0.228583	0.115280	0.030922	0.048*
H5B	-0.106306	0.096893	0.006017	0.048*
C6	-0.11728 (11)	0.29849 (16)	0.05959 (7)	0.0377 (3)
H6A	-0.151622	0.363527	0.020283	0.045*
H6B	-0.151457	0.330953	0.099006	0.045*
C7	0.03764 (12)	0.47494 (15)	0.09298 (7)	0.0370 (3)
H7A	0.004496	0.504928	0.133100	0.044*
H7B	0.006123	0.546253	0.055350	0.044*
C8	0.16749 (12)	0.48729 (16)	0.10895 (7)	0.0396 (3)
H8A	0.201647	0.438366	0.072478	0.048*
H8B	0.191122	0.598056	0.112413	0.048*
C9	0.33226 (12)	0.41468 (17)	0.18955 (7)	0.0422 (3)
H9A	0.359744	0.523840	0.191439	0.051*
H9B	0.367361	0.358512	0.155230	0.051*
C10	0.36735 (11)	0.33922 (18)	0.25737 (7)	0.0430 (3)
H10A	0.452159	0.351775	0.273446	0.052*
H10B	0.326178	0.389241	0.290618	0.052*
C11	0.36879 (11)	0.09996 (19)	0.31478 (7)	0.0452 (4)
H11A	0.323873	0.143600	0.347687	0.054*
H11B	0.452733	0.114220	0.333181	0.054*
C12	0.34212 (12)	-0.0685 (2)	0.30425 (8)	0.0483 (4)
H12A	0.384333	-0.110725	0.269761	0.058*
H12B	0.368262	-0.125858	0.347112	0.058*
C14	0.37145 (11)	0.02001 (15)	0.09102 (6)	0.0323 (3)
C15	0.50091 (13)	0.0405 (2)	0.09116 (9)	0.0514 (4)
H15A	0.535582	-0.060718	0.084288	0.062*

H15B	0.538963	0.084059	0.134728	0.062*
H15C	0.511530	0.111363	0.054547	0.062*
O1	0.21942 (8)	-0.08938 (11)	0.28225 (5)	0.0363 (2)
O2	0.02642 (7)	-0.19209 (10)	0.18282 (4)	0.0337 (2)
O3	-0.09923 (8)	0.04182 (10)	0.10328 (4)	0.0358 (2)
O4	0.00621 (8)	0.31881 (10)	0.07425 (4)	0.0353 (2)
O5	0.20816 (8)	0.41003 (11)	0.17174 (5)	0.0371 (2)
O6	0.33857 (8)	0.17840 (12)	0.25161 (5)	0.0421 (2)
O7	0.30476 (8)	0.13088 (11)	0.06867 (5)	0.0371 (2)
O8	0.33793 (9)	-0.10438 (11)	0.11438 (5)	0.0411 (2)
O9	0.63538 (9)	0.64950 (12)	0.01773 (5)	0.0439 (2)
H1	0.6336 (18)	0.5550 (15)	-0.0055 (9)	0.073 (6)*
H2	0.6560 (16)	0.7200 (18)	-0.0134 (8)	0.068 (6)*
O10	0.40196 (10)	0.64828 (13)	0.03746 (7)	0.0561 (3)
H3	0.3823 (17)	0.7270 (19)	0.0651 (9)	0.078 (6)*
H4	0.4786 (9)	0.666 (2)	0.0337 (10)	0.072 (6)*
K	0.15130 (2)	0.07687 (3)	0.15141 (2)	0.03251 (9)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0477 (8)	0.0403 (7)	0.0343 (7)	0.0144 (6)	0.0086 (6)	0.0010 (5)
C2	0.0502 (8)	0.0332 (7)	0.0334 (7)	0.0008 (6)	0.0135 (6)	0.0018 (5)
C3	0.0289 (6)	0.0326 (7)	0.0528 (8)	-0.0046 (5)	0.0130 (5)	-0.0003 (6)
C4	0.0320 (6)	0.0335 (7)	0.0490 (8)	-0.0077 (5)	0.0002 (6)	-0.0065 (6)
C5	0.0383 (7)	0.0467 (8)	0.0315 (7)	-0.0040 (6)	-0.0054 (5)	0.0027 (6)
C6	0.0340 (7)	0.0431 (7)	0.0344 (7)	0.0025 (6)	0.0022 (5)	0.0097 (6)
C7	0.0498 (8)	0.0300 (6)	0.0330 (7)	-0.0010 (6)	0.0122 (6)	-0.0020 (5)
C8	0.0503 (8)	0.0352 (7)	0.0367 (7)	-0.0104 (6)	0.0169 (6)	-0.0043 (6)
C9	0.0336 (7)	0.0474 (8)	0.0496 (8)	-0.0148 (6)	0.0180 (6)	-0.0163 (6)
C10	0.0264 (6)	0.0584 (9)	0.0453 (8)	-0.0128 (6)	0.0094 (5)	-0.0213 (7)
C11	0.0249 (6)	0.0722 (10)	0.0360 (7)	0.0049 (6)	-0.0008 (5)	-0.0059 (7)
C12	0.0284 (7)	0.0660 (10)	0.0488 (8)	0.0141 (7)	0.0029 (6)	0.0021 (7)
C14	0.0370 (6)	0.0346 (7)	0.0254 (6)	-0.0023 (5)	0.0057 (5)	-0.0034 (5)
C15	0.0366 (7)	0.0540 (9)	0.0601 (10)	-0.0016 (7)	-0.0002 (6)	0.0185 (7)
O1	0.0293 (4)	0.0430 (5)	0.0365 (5)	0.0083 (4)	0.0056 (4)	0.0000 (4)
O2	0.0297 (4)	0.0368 (5)	0.0358 (5)	-0.0009 (4)	0.0090 (3)	0.0042 (4)
O3	0.0361 (5)	0.0321 (5)	0.0345 (5)	-0.0052 (4)	-0.0056 (4)	-0.0002 (4)
O4	0.0352 (5)	0.0319 (5)	0.0384 (5)	-0.0017 (4)	0.0063 (4)	-0.0023 (4)
O5	0.0333 (5)	0.0428 (5)	0.0369 (5)	-0.0102 (4)	0.0111 (4)	-0.0038 (4)
O6	0.0367 (5)	0.0542 (6)	0.0341 (5)	-0.0079 (4)	0.0035 (4)	-0.0097 (4)
O7	0.0401 (5)	0.0349 (5)	0.0380 (5)	0.0031 (4)	0.0111 (4)	0.0016 (4)
O8	0.0480 (6)	0.0338 (5)	0.0443 (5)	0.0002 (4)	0.0162 (4)	0.0032 (4)
O9	0.0538 (6)	0.0397 (6)	0.0411 (5)	-0.0111 (5)	0.0161 (5)	-0.0043 (4)
O10	0.0452 (6)	0.0414 (6)	0.0854 (9)	-0.0081 (5)	0.0213 (6)	-0.0203 (6)
K	0.03047 (15)	0.03681 (16)	0.03057 (15)	-0.00472 (11)	0.00648 (10)	-0.00161 (10)



*Geometric parameters (Å, °)*

C1—O1	1.4241 (17)	C9—H9B	0.9900
C1—C2	1.493 (2)	C10—O6	1.4220 (18)
C1—H1A	0.9900	C10—H10A	0.9900
C1—H1B	0.9900	C10—H10B	0.9900
C2—O2	1.4271 (15)	C11—O6	1.4227 (17)
C2—H2A	0.9900	C11—C12	1.488 (2)
C2—H2B	0.9900	C11—H11A	0.9900
C3—O2	1.4259 (15)	C11—H11B	0.9900
C3—C4	1.495 (2)	C12—O1	1.4316 (16)
C3—H3A	0.9900	C12—H12A	0.9900
C3—H3B	0.9900	C12—H12B	0.9900
C4—O3	1.4261 (16)	C14—O8	1.2584 (16)
C4—H4A	0.9900	C14—O7	1.2601 (16)
C4—H4B	0.9900	C14—C15	1.5222 (19)
C5—O3	1.4277 (16)	C14—K	3.0775 (14)
C5—C6	1.494 (2)	C15—H15A	0.9800
C5—H5A	0.9900	C15—H15B	0.9800
C5—H5B	0.9900	C15—H15C	0.9800
C6—O4	1.4292 (15)	K—O1	2.9684 (11)
C6—H6A	0.9900	K—O2	2.8649 (10)
C6—H6B	0.9900	K—O3	2.9244 (11)
C7—O4	1.4234 (16)	K—O4	2.9380 (11)
C7—C8	1.4956 (19)	K—O5	2.9506 (12)
C7—H7A	0.9900	K—O6	2.8248 (13)
C7—H7B	0.9900	K—O7	2.6992 (11)
C8—O5	1.4270 (17)	K—O8	2.8861 (11)
C8—H8A	0.9900	O9—H1	0.935 (9)
C8—H8B	0.9900	O9—H2	0.933 (9)
C9—O5	1.4287 (16)	O10—H3	0.930 (9)
C9—C10	1.496 (2)	O10—H4	0.925 (9)
C9—H9A	0.9900		
O1—C1—C2	108.91 (11)	O1—C12—H12A	109.7
O1—C1—H1A	109.9	C11—C12—H12A	109.7
C2—C1—H1A	109.9	O1—C12—H12B	109.7
O1—C1—H1B	109.9	C11—C12—H12B	109.7
C2—C1—H1B	109.9	H12A—C12—H12B	108.2
H1A—C1—H1B	108.3	O8—C14—O7	124.10 (12)
O2—C2—C1	108.79 (11)	O8—C14—C15	118.35 (12)
O2—C2—H2A	109.9	O7—C14—C15	117.53 (12)
C1—C2—H2A	109.9	O8—C14—K	69.40 (7)
O2—C2—H2B	109.9	O7—C14—K	60.89 (7)
C1—C2—H2B	109.9	C15—C14—K	152.01 (10)
H2A—C2—H2B	108.3	C14—C15—H15A	109.5
O2—C3—C4	109.16 (10)	C14—C15—H15B	109.5
O2—C3—H3A	109.8	H15A—C15—H15B	109.5

C4—C3—H3A	109.8	C14—C15—H15C	109.5
O2—C3—H3B	109.8	H15A—C15—H15C	109.5
C4—C3—H3B	109.8	H15B—C15—H15C	109.5
H3A—C3—H3B	108.3	C1—O1—C12	110.99 (10)
O3—C4—C3	108.40 (11)	C1—O1—K	104.96 (7)
O3—C4—H4A	110.0	C12—O1—K	107.66 (8)
C3—C4—H4A	110.0	C3—O2—C2	111.21 (10)
O3—C4—H4B	110.0	C3—O2—K	119.27 (7)
C3—C4—H4B	110.0	C2—O2—K	118.17 (7)
H4A—C4—H4B	108.4	C4—O3—C5	112.42 (10)
O3—C5—C6	108.32 (10)	C4—O3—K	111.20 (7)
O3—C5—H5A	110.0	C5—O3—K	114.02 (8)
C6—C5—H5A	110.0	C7—O4—C6	111.79 (10)
O3—C5—H5B	110.0	C7—O4—K	115.59 (7)
C6—C5—H5B	110.0	C6—O4—K	119.00 (7)
H5A—C5—H5B	108.4	C8—O5—C9	111.47 (10)
O4—C6—C5	108.72 (11)	C8—O5—K	107.42 (7)
O4—C6—H6A	109.9	C9—O5—K	104.72 (8)
C5—C6—H6A	109.9	C10—O6—C11	111.82 (11)
O4—C6—H6B	109.9	C10—O6—K	119.92 (8)
C5—C6—H6B	109.9	C11—O6—K	121.45 (8)
H6A—C6—H6B	108.3	C14—O7—K	95.04 (7)
O4—C7—C8	109.02 (11)	C14—O8—K	86.51 (8)
O4—C7—H7A	109.9	H1—O9—H2	102.4 (17)
C8—C7—H7A	109.9	H3—O10—H4	106.0 (17)
O4—C7—H7B	109.9	O7—K—O6	82.55 (3)
C8—C7—H7B	109.9	O7—K—O2	134.19 (3)
H7A—C7—H7B	108.3	O6—K—O2	116.84 (3)
O5—C8—C7	108.44 (10)	O7—K—O8	46.80 (3)
O5—C8—H8A	110.0	O6—K—O8	80.06 (3)
C7—C8—H8A	110.0	O2—K—O8	93.48 (3)
O5—C8—H8B	110.0	O7—K—O3	123.50 (3)
C7—C8—H8B	110.0	O6—K—O3	149.82 (3)
H8A—C8—H8B	108.4	O2—K—O3	58.02 (3)
O5—C9—C10	108.15 (11)	O8—K—O3	128.09 (3)
O5—C9—H9A	110.1	O7—K—O4	86.43 (3)
C10—C9—H9A	110.1	O6—K—O4	116.96 (3)
O5—C9—H9B	110.1	O2—K—O4	114.79 (3)
C10—C9—H9B	110.1	O8—K—O4	129.49 (3)
H9A—C9—H9B	108.4	O3—K—O4	56.85 (3)
O6—C10—C9	109.03 (11)	O7—K—O5	76.12 (3)
O6—C10—H10A	109.9	O6—K—O5	58.43 (3)
C9—C10—H10A	109.9	O2—K—O5	149.68 (3)
O6—C10—H10B	109.9	O8—K—O5	113.44 (3)
C9—C10—H10B	109.9	O3—K—O5	109.39 (3)
H10A—C10—H10B	108.3	O4—K—O5	58.64 (3)
O6—C11—C12	108.85 (11)	O7—K—O1	121.82 (3)
O6—C11—H11A	109.9	O6—K—O1	58.09 (3)

C12—C11—H11A	109.9	O2—K—O1	58.75 (3)
O6—C11—H11B	109.9	O8—K—O1	82.87 (3)
C12—C11—H11B	109.9	O3—K—O1	109.41 (4)
H11A—C11—H11B	108.3	O4—K—O1	147.39 (3)
O1—C12—C11	109.71 (11)	O5—K—O1	109.13 (3)
O1—C1—C2—O2	69.56 (13)	C6—C5—O3—K	-58.71 (12)
O2—C3—C4—O3	-64.30 (14)	C8—C7—O4—C6	178.85 (10)
O3—C5—C6—O4	63.29 (14)	C8—C7—O4—K	38.46 (12)
O4—C7—C8—O5	-71.79 (13)	C5—C6—O4—C7	-176.94 (10)
O5—C9—C10—O6	66.16 (13)	C5—C6—O4—K	-38.05 (12)
O6—C11—C12—O1	-63.66 (15)	C7—C8—O5—C9	178.87 (10)
C2—C1—O1—C12	177.74 (11)	C7—C8—O5—K	64.69 (11)
C2—C1—O1—K	-66.22 (11)	C10—C9—O5—C8	177.00 (11)
C11—C12—O1—C1	175.73 (11)	C10—C9—O5—K	-67.16 (10)
C11—C12—O1—K	61.37 (12)	C9—C10—O6—C11	-179.41 (10)
C4—C3—O2—C2	179.89 (10)	C9—C10—O6—K	-27.92 (13)
C4—C3—O2—K	36.94 (13)	C12—C11—O6—C10	-177.04 (11)
C1—C2—O2—C3	-176.43 (11)	C12—C11—O6—K	31.96 (14)
C1—C2—O2—K	-33.03 (13)	O8—C14—O7—K	30.29 (13)
C3—C4—O3—C5	-171.43 (11)	C15—C14—O7—K	-148.16 (11)
C3—C4—O3—K	59.34 (12)	O7—C14—O8—K	-28.09 (12)
C6—C5—O3—C4	173.54 (11)	C15—C14—O8—K	150.35 (11)

## Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O9—H1...O10 <sup>i</sup>	0.94 (1)	1.88 (1)	2.790 (3)	165 (4)
O9—H2...O7 <sup>i</sup>	0.93 (1)	1.81 (2)	2.741 (3)	175 (4)
O10—H3...O8 <sup>ii</sup>	0.93 (1)	1.88 (2)	2.810 (3)	175 (4)
O10—H4...O9	0.93 (1)	1.92 (1)	2.827 (3)	166 (4)

Symmetry codes: (i)  $-x+1, -y+1, -z$ ; (ii)  $x, y+1, z$ .(2) (Acetato- $\kappa^2O, O'$ )aqua(1,4,7,10,13,16-hexaoxacyclooctadecane- $\kappa^6O$ )potassium acetic acid monosolvate

## Crystal data

[K(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)(C<sub>12</sub>H<sub>24</sub>O<sub>6</sub>)(H<sub>2</sub>O)]·C<sub>2</sub>H<sub>4</sub>O<sub>2</sub> $M_r = 440.52$ Monoclinic,  $P2_1/c$  $a = 11.3233$  (1) Å $b = 8.5450$  (1) Å $c = 23.3869$  (3) Å $\beta = 99.053$  (1)° $V = 2234.67$  (4) Å<sup>3</sup> $Z = 4$  $F(000) = 944$  $D_x = 1.309$  Mg m<sup>-3</sup>Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 100 reflections

 $\theta = 2.0$ – $27.5^\circ$  $\mu = 0.29$  mm<sup>-1</sup> $T = 200$  K

Block, colorless

 $0.40 \times 0.40 \times 0.20$  mm

*Data collection*

Bruker SMART CCD  
diffractometer  
Radiation source: fine-focus sealed tube  
 $\omega$  scans  
Absorption correction: multi-scan  
(SADABS; Bruker, 2001)  
 $T_{\min} = 0.893$ ,  $T_{\max} = 0.945$   
13151 measured reflections

4846 independent reflections  
3833 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.035$   
 $\theta_{\max} = 27.0^\circ$ ,  $\theta_{\min} = 1.8^\circ$   
 $h = -14 \rightarrow 14$   
 $k = -10 \rightarrow 10$   
 $l = -29 \rightarrow 19$

*Refinement*

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.104$   
 $S = 1.05$   
4846 reflections  
268 parameters  
2 restraints  
Primary atom site location: structure-invariant  
direct methods  
Secondary atom site location: difference Fourier  
map

Hydrogen site location: mixed  
H atoms treated by a mixture of independent  
and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0411P)^2 + 0.8233P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$   
Extinction correction: SHELXL2016  
(Sheldrick, 2015),  
 $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$   
Extinction coefficient: 0.0064 (8)

*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>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	-0.26514 (15)	0.3641 (2)	0.59639 (8)	0.0344 (4)
H1A	-0.295602	0.316810	0.629859	0.041*
H1B	-0.325899	0.348656	0.561493	0.041*
C2	-0.24318 (16)	0.5356 (2)	0.60680 (8)	0.0339 (4)
H2A	-0.211614	0.582388	0.573534	0.041*
H2B	-0.319274	0.588713	0.610600	0.041*
C3	-0.12935 (17)	0.7194 (2)	0.66672 (8)	0.0326 (4)
H3A	-0.202139	0.780529	0.670253	0.039*
H3B	-0.095777	0.759557	0.632950	0.039*
C4	-0.03993 (17)	0.7378 (2)	0.72029 (8)	0.0322 (4)
H4A	-0.027326	0.850197	0.729470	0.039*
H4B	-0.069596	0.686669	0.753275	0.039*
C5	0.15966 (17)	0.6830 (2)	0.76087 (8)	0.0318 (4)
H5A	0.136112	0.624324	0.793796	0.038*
H5B	0.169625	0.794519	0.772089	0.038*
C6	0.27450 (17)	0.6196 (2)	0.74649 (9)	0.0347 (4)
H6A	0.292850	0.668968	0.710610	0.042*
H6B	0.340818	0.642839	0.778267	0.042*

C7	0.36809 (16)	0.3840 (2)	0.72475 (9)	0.0357 (4)
H7A	0.436684	0.406551	0.755438	0.043*
H7B	0.386265	0.426119	0.687675	0.043*
C8	0.34709 (17)	0.2111 (2)	0.71987 (9)	0.0374 (5)
H8A	0.421645	0.157251	0.713968	0.045*
H8B	0.322839	0.170756	0.755975	0.045*
C9	0.22477 (18)	0.0196 (2)	0.66723 (9)	0.0373 (5)
H9A	0.188685	-0.013663	0.701176	0.045*
H9B	0.297603	-0.043805	0.666202	0.045*
C10	0.13805 (17)	-0.0062 (2)	0.61311 (9)	0.0370 (5)
H10A	0.170412	0.037599	0.579565	0.044*
H10B	0.124689	-0.119710	0.606545	0.044*
C11	-0.05449 (18)	0.0615 (2)	0.56565 (8)	0.0347 (4)
H11A	-0.065285	-0.048774	0.552956	0.042*
H11B	-0.022018	0.120701	0.535208	0.042*
C12	-0.17179 (17)	0.1291 (2)	0.57418 (8)	0.0341 (4)
H12A	-0.230412	0.117291	0.538289	0.041*
H12B	-0.202776	0.072861	0.605745	0.041*
C13	0.26513 (17)	0.4042 (3)	0.54918 (9)	0.0395 (5)
C14	0.3933 (2)	0.3558 (4)	0.54951 (12)	0.0628 (7)
H14A	0.441672	0.448409	0.544505	0.075*
H14B	0.397490	0.282164	0.517759	0.075*
H14C	0.424067	0.305288	0.586489	0.075*
C15	0.46601 (19)	0.7951 (3)	0.60242 (11)	0.0458 (5)
C16	0.5436 (3)	0.9332 (3)	0.59606 (15)	0.0734 (8)
H16A	0.588137	0.915124	0.563968	0.088*
H16B	0.599944	0.948428	0.631968	0.088*
H16C	0.493720	1.026816	0.588064	0.088*
O1	-0.15612 (11)	0.29088 (15)	0.58855 (6)	0.0329 (3)
O2	-0.15927 (11)	0.55729 (14)	0.65841 (5)	0.0299 (3)
O3	0.06996 (11)	0.66769 (15)	0.71136 (5)	0.0298 (3)
O4	0.26219 (11)	0.45464 (14)	0.73867 (6)	0.0311 (3)
O5	0.25553 (11)	0.18135 (14)	0.67223 (5)	0.0322 (3)
O6	0.02708 (11)	0.06945 (14)	0.61872 (5)	0.0319 (3)
O7	0.24709 (14)	0.5417 (2)	0.56419 (9)	0.0654 (5)
O8	0.18335 (13)	0.30957 (19)	0.53554 (6)	0.0459 (4)
O9	0.01027 (12)	0.64125 (16)	0.55199 (6)	0.0339 (3)
H2	-0.0421 (19)	0.648 (3)	0.5168 (7)	0.066 (8)*
H1	0.0852 (12)	0.616 (3)	0.5426 (10)	0.054 (7)*
O10	0.47534 (17)	0.7214 (2)	0.64660 (8)	0.0707 (5)
O11	0.39035 (17)	0.7625 (3)	0.55675 (8)	0.0718 (6)
H3	0.337 (4)	0.662 (6)	0.564 (2)	0.165 (18)*
K	0.06834 (3)	0.40782 (4)	0.63478 (2)	0.02625 (12)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0225 (9)	0.0459 (12)	0.0334 (10)	-0.0006 (8)	-0.0003 (7)	0.0009 (8)

C2	0.0258 (9)	0.0432 (11)	0.0312 (10)	0.0080 (8)	-0.0002 (7)	0.0040 (8)
C3	0.0370 (10)	0.0278 (10)	0.0342 (10)	0.0096 (8)	0.0097 (8)	0.0035 (8)
C4	0.0393 (10)	0.0265 (9)	0.0328 (10)	0.0053 (8)	0.0114 (8)	-0.0041 (8)
C5	0.0433 (11)	0.0251 (9)	0.0255 (9)	-0.0039 (8)	0.0006 (8)	-0.0026 (7)
C6	0.0352 (10)	0.0311 (10)	0.0357 (10)	-0.0073 (8)	-0.0013 (8)	0.0003 (8)
C7	0.0228 (9)	0.0441 (12)	0.0385 (11)	0.0011 (8)	-0.0003 (8)	-0.0001 (9)
C8	0.0290 (10)	0.0416 (11)	0.0399 (11)	0.0117 (8)	0.0002 (8)	0.0031 (9)
C9	0.0380 (11)	0.0239 (10)	0.0516 (12)	0.0078 (8)	0.0118 (9)	0.0052 (8)
C10	0.0394 (11)	0.0233 (9)	0.0515 (12)	0.0009 (8)	0.0170 (9)	-0.0083 (8)
C11	0.0495 (12)	0.0283 (10)	0.0270 (9)	-0.0070 (8)	0.0083 (8)	-0.0059 (7)
C12	0.0380 (10)	0.0328 (10)	0.0295 (9)	-0.0111 (8)	-0.0010 (8)	-0.0044 (8)
C13	0.0294 (10)	0.0581 (14)	0.0307 (10)	0.0042 (10)	0.0040 (8)	0.0103 (9)
C14	0.0367 (12)	0.083 (2)	0.0697 (17)	0.0152 (12)	0.0132 (12)	0.0046 (15)
C15	0.0327 (11)	0.0464 (13)	0.0581 (14)	0.0032 (9)	0.0066 (10)	-0.0108 (11)
C16	0.0665 (18)	0.0566 (17)	0.094 (2)	-0.0209 (14)	0.0030 (16)	0.0084 (15)
O1	0.0250 (6)	0.0326 (7)	0.0400 (7)	-0.0027 (5)	0.0022 (5)	-0.0051 (6)
O2	0.0319 (7)	0.0295 (7)	0.0273 (6)	0.0038 (5)	0.0014 (5)	0.0018 (5)
O3	0.0332 (7)	0.0316 (7)	0.0241 (6)	0.0051 (5)	0.0027 (5)	-0.0048 (5)
O4	0.0275 (6)	0.0265 (7)	0.0391 (7)	-0.0013 (5)	0.0046 (5)	0.0015 (5)
O5	0.0347 (7)	0.0250 (7)	0.0352 (7)	0.0033 (5)	0.0002 (6)	0.0024 (5)
O6	0.0352 (7)	0.0286 (7)	0.0329 (7)	0.0006 (5)	0.0085 (6)	-0.0048 (5)
O7	0.0289 (8)	0.0635 (12)	0.1036 (15)	-0.0045 (8)	0.0099 (9)	-0.0225 (11)
O8	0.0396 (8)	0.0536 (9)	0.0422 (8)	-0.0021 (7)	-0.0002 (7)	0.0117 (7)
O9	0.0345 (7)	0.0357 (7)	0.0314 (7)	0.0034 (6)	0.0043 (6)	0.0001 (6)
O10	0.0710 (12)	0.0823 (14)	0.0568 (11)	-0.0218 (10)	0.0037 (9)	0.0083 (10)
O11	0.0637 (12)	0.0804 (14)	0.0629 (12)	-0.0156 (10)	-0.0156 (10)	0.0053 (10)
K	0.02579 (19)	0.0254 (2)	0.0272 (2)	0.00122 (15)	0.00280 (14)	0.00003 (15)

*Geometric parameters (Å, °)*

C1—O1	1.421 (2)	C10—H10B	0.9900
C1—C2	1.500 (3)	C11—O6	1.427 (2)
C1—H1A	0.9900	C11—C12	1.490 (3)
C1—H1B	0.9900	C11—H11A	0.9900
C2—O2	1.426 (2)	C11—H11B	0.9900
C2—H2A	0.9900	C12—O1	1.427 (2)
C2—H2B	0.9900	C12—H12A	0.9900
C3—O2	1.432 (2)	C12—H12B	0.9900
C3—C4	1.490 (3)	C13—O8	1.233 (3)
C3—H3A	0.9900	C13—O7	1.252 (3)
C3—H3B	0.9900	C13—C14	1.508 (3)
C4—O3	1.426 (2)	C13—K	3.221 (2)
C4—H4A	0.9900	C14—H14A	0.9800
C4—H4B	0.9900	C14—H14B	0.9800
C5—O3	1.421 (2)	C14—H14C	0.9800
C5—C6	1.495 (3)	C15—O10	1.201 (3)
C5—H5A	0.9900	C15—O11	1.290 (3)
C5—H5B	0.9900	C15—C16	1.493 (3)



C6—O4	1.425 (2)	C16—H16A	0.9800
C6—H6A	0.9900	C16—H16B	0.9800
C6—H6B	0.9900	C16—H16C	0.9800
C7—O4	1.425 (2)	K—O1	2.7861 (12)
C7—C8	1.498 (3)	K—O2	3.0045 (13)
C7—H7A	0.9900	K—O3	2.8510 (12)
C7—H7B	0.9900	K—O4	3.0337 (13)
C8—O5	1.420 (2)	K—O5	2.9019 (13)
C8—H8A	0.9900	K—O6	2.9435 (13)
C8—H8B	0.9900	K—O7	3.0303 (19)
C9—O5	1.426 (2)	K—O8	2.9562 (16)
C9—C10	1.491 (3)	K—O9	2.7855 (14)
C9—H9A	0.9900	O9—H2	0.937 (10)
C9—H9B	0.9900	O9—H1	0.935 (10)
C10—O6	1.437 (2)	O11—H3	1.08 (5)
C10—H10A	0.9900		
O1—C1—C2	109.02 (15)	C13—C14—H14A	109.5
O1—C1—H1A	109.9	C13—C14—H14B	109.5
C2—C1—H1A	109.9	H14A—C14—H14B	109.5
O1—C1—H1B	109.9	C13—C14—H14C	109.5
C2—C1—H1B	109.9	H14A—C14—H14C	109.5
H1A—C1—H1B	108.3	H14B—C14—H14C	109.5
O2—C2—C1	109.61 (15)	O10—C15—O11	123.8 (2)
O2—C2—H2A	109.7	O10—C15—C16	121.8 (2)
C1—C2—H2A	109.7	O11—C15—C16	114.4 (2)
O2—C2—H2B	109.7	C15—C16—H16A	109.5
C1—C2—H2B	109.7	C15—C16—H16B	109.5
H2A—C2—H2B	108.2	H16A—C16—H16B	109.5
O2—C3—C4	109.30 (14)	C15—C16—H16C	109.5
O2—C3—H3A	109.8	H16A—C16—H16C	109.5
C4—C3—H3A	109.8	H16B—C16—H16C	109.5
O2—C3—H3B	109.8	C1—O1—C12	112.24 (14)
C4—C3—H3B	109.8	C1—O1—K	123.36 (10)
H3A—C3—H3B	108.3	C12—O1—K	120.72 (10)
O3—C4—C3	109.03 (14)	C2—O2—C3	110.62 (14)
O3—C4—H4A	109.9	C2—O2—K	105.41 (10)
C3—C4—H4A	109.9	C3—O2—K	104.17 (10)
O3—C4—H4B	109.9	C5—O3—C4	111.74 (13)
C3—C4—H4B	109.9	C5—O3—K	121.11 (10)
H4A—C4—H4B	108.3	C4—O3—K	119.97 (10)
O3—C5—C6	108.58 (14)	C7—O4—C6	112.30 (14)
O3—C5—H5A	110.0	C7—O4—K	106.86 (10)
C6—C5—H5A	110.0	C6—O4—K	106.08 (10)
O3—C5—H5B	110.0	C8—O5—C9	112.12 (14)
C6—C5—H5B	110.0	C8—O5—K	121.71 (10)
H5A—C5—H5B	108.4	C9—O5—K	117.67 (10)
O4—C6—C5	108.44 (15)	C11—O6—C10	110.78 (14)

O4—C6—H6A	110.0	C11—O6—K	103.03 (10)
C5—C6—H6A	110.0	C10—O6—K	109.26 (10)
O4—C6—H6B	110.0	C13—O7—K	87.18 (13)
C5—C6—H6B	110.0	C13—O7—H3	121.1 (19)
H6A—C6—H6B	108.4	K—O7—H3	143.3 (19)
O4—C7—C8	107.82 (15)	C13—O8—K	90.94 (13)
O4—C7—H7A	110.1	K—O9—H2	134.4 (17)
C8—C7—H7A	110.1	K—O9—H1	82.7 (15)
O4—C7—H7B	110.1	H2—O9—H1	106 (2)
C8—C7—H7B	110.1	C15—O11—H3	111 (3)
H7A—C7—H7B	108.5	O9—K—O1	83.45 (4)
O5—C8—C7	108.89 (15)	O9—K—O3	81.74 (4)
O5—C8—H8A	109.9	O1—K—O3	116.00 (4)
C7—C8—H8A	109.9	O9—K—O5	140.23 (4)
O5—C8—H8B	109.9	O1—K—O5	117.15 (4)
C7—C8—H8B	109.9	O3—K—O5	113.43 (4)
H8A—C8—H8B	108.3	O9—K—O6	126.82 (4)
O5—C9—C10	109.42 (15)	O1—K—O6	58.76 (4)
O5—C9—H9A	109.8	O3—K—O6	146.14 (4)
C10—C9—H9A	109.8	O5—K—O6	58.48 (4)
O5—C9—H9B	109.8	O9—K—O8	75.08 (4)
C10—C9—H9B	109.8	O1—K—O8	94.65 (4)
H9A—C9—H9B	108.2	O3—K—O8	138.97 (4)
O6—C10—C9	108.94 (15)	O5—K—O8	69.91 (4)
O6—C10—H10A	109.9	O6—K—O8	72.63 (4)
C9—C10—H10A	109.9	O9—K—O2	73.02 (4)
O6—C10—H10B	109.9	O1—K—O2	57.81 (4)
C9—C10—H10B	109.9	O3—K—O2	58.28 (3)
H10A—C10—H10B	108.3	O5—K—O2	146.61 (4)
O6—C11—C12	109.49 (14)	O6—K—O2	108.51 (4)
O6—C11—H11A	109.8	O8—K—O2	139.62 (4)
C12—C11—H11A	109.8	O9—K—O7	57.03 (5)
O6—C11—H11B	109.8	O1—K—O7	124.80 (5)
C12—C11—H11B	109.8	O3—K—O7	96.28 (4)
H11A—C11—H11B	108.2	O5—K—O7	84.11 (4)
O1—C12—C11	109.04 (15)	O6—K—O7	114.05 (4)
O1—C12—H12A	109.9	O8—K—O7	42.69 (5)
C11—C12—H12A	109.9	O2—K—O7	127.27 (4)
O1—C12—H12B	109.9	O9—K—O4	121.61 (4)
C11—C12—H12B	109.9	O1—K—O4	149.21 (4)
H12A—C12—H12B	108.3	O3—K—O4	57.44 (3)
O8—C13—O7	122.60 (19)	O5—K—O4	56.23 (3)
O8—C13—C14	120.3 (2)	O6—K—O4	108.18 (4)
O7—C13—C14	117.1 (2)	O8—K—O4	108.28 (4)
O8—C13—K	66.57 (11)	O2—K—O4	109.28 (4)
O7—C13—K	69.97 (12)	O7—K—O4	85.72 (4)
C14—C13—K	139.36 (15)		

O1—C1—C2—O2	-61.89 (19)	C3—C4—O3—K	-29.99 (18)
O2—C3—C4—O3	67.56 (18)	C8—C7—O4—C6	177.77 (15)
O3—C5—C6—O4	-67.82 (18)	C8—C7—O4—K	-66.31 (15)
O4—C7—C8—O5	65.00 (19)	C5—C6—O4—C7	-179.99 (15)
O5—C9—C10—O6	-67.21 (19)	C5—C6—O4—K	63.62 (15)
O6—C11—C12—O1	63.41 (19)	C7—C8—O5—C9	-175.98 (15)
C2—C1—O1—C12	-175.70 (15)	C7—C8—O5—K	-28.8 (2)
C2—C1—O1—K	25.89 (19)	C10—C9—O5—C8	-174.05 (15)
C11—C12—O1—C1	178.99 (15)	C10—C9—O5—K	37.31 (18)
C11—C12—O1—K	-21.96 (18)	C12—C11—O6—C10	175.38 (15)
C1—C2—O2—C3	175.23 (15)	C12—C11—O6—K	-67.88 (14)
C1—C2—O2—K	63.21 (15)	C9—C10—O6—C11	174.01 (15)
C4—C3—O2—C2	-178.92 (14)	C9—C10—O6—K	61.18 (16)
C4—C3—O2—K	-66.11 (14)	O8—C13—O7—K	42.6 (2)
C6—C5—O3—C4	-175.13 (15)	C14—C13—O7—K	-136.11 (18)
C6—C5—O3—K	34.54 (18)	O7—C13—O8—K	-43.9 (2)
C3—C4—O3—C5	179.29 (14)	C14—C13—O8—K	134.79 (19)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O9—H1...O7	0.94 (1)	1.93 (2)	2.785 (7)	151 (9)
O9—H2...O8 <sup>i</sup>	0.94 (1)	1.89 (2)	2.786 (7)	160 (9)
O11—H3...O7	1.08 (5)	1.43 (5)	2.513 (6)	168 (5)

Symmetry code: (i)  $-x, -y+1, -z+1$ .