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Byproduct identification: crystal structure of potassium 2-ethoxy-2-oxoacetate

Shannon E. Creegan,[a] James A. Ridenour,[b] Robert J. Donnelly[c] and Andrew T. Kerr[a]*

[a]US Naval Research Laboratory, Center for Biomolecular Science and Engineering, 4555 Overlook Ave., SW Washington, DC 20375, USA, [b]US Naval Research Laboratory, Materials Chemistry and Dynamics Branch, 4555 Overlook Ave., SW Washington, DC 20375, USA, and [c]Department of Chemistry, Washington College, 300 Washington Ave., Chestertown, MD 21620, USA. *Correspondence e-mail: andrew.t.kerr2.civ@us.navy.mil

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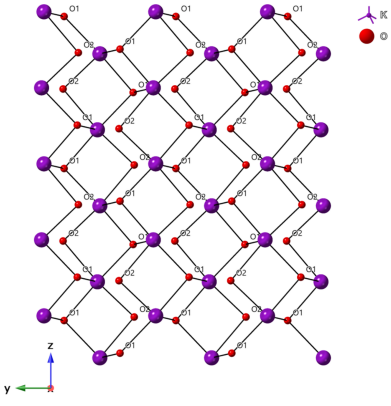
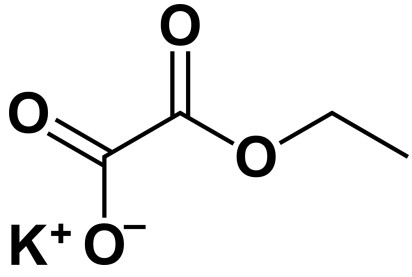
Potassium 2-ethoxy-2-oxoacetate, $K^+ \cdot C_4H_5O_4^-$, was identified as a byproduct in the first step of synthesizing the shelf-stable salt potassium nitroacetonitrile (KNAN). A single crystal of the title salt was obtained by recrystallization of the ethanol filtrate resulting from the purification of the intended product salt, potassium ethyl nitrocyanoacetate. The asymmetric unit of $K^+ \cdot C_4H_5O_4^-$, contains a single potassium cation and a 2-ethoxy-2-oxoacetate anion, the ethyl group of which is disordered over two sets of sites in a 0.792 (13):0.208 (13) ratio. The packing is dictated primarily by K—O electrostatic interactions and comprises eight bonded oxygen atoms per cation, with distances ranging from 2.706 (2) to 3.053 (4) Å.

1. Chemical context

Voinkov *et al.* (2016) reported an alternative synthesis method for the α-nitronitrile compound, nitroacetonitrile, $C_2H_2N_2O_2$ (NAN). This synthesis was developed to bypass the greatest hazard of NAN synthesis, spontaneous explosion (Thomas, 2009), while improving the yield and purity of the NAN produced. The final product of the reaction detailed by Voinkov *et al.* (2016) is the potassium salt of nitroacetonitrile (KNAN). Two key features of KNAN are its shelf-stability and that, as a salt, it is ready for immediate use in contrast to neutral NAN, which is unstable, requires careful storage, and needs to be deprotonated prior to use. The shelf-stable KNAN is produced in a two-step synthesis from ethyl (hydroxyimino)cyanoacetate. During the first step of this process, ethyl (hydroxyimino)cyanoacetate is oxidized with potassium permanganate in the presence of potassium hydroxide to form the salt potassium ethyl nitrocyanoacetate in 63% yield (Fig. 1). It was in executing this first step that we identified the salt potassium 2-ethoxy-2-oxoacetate, the crystal structure of which is reported here.





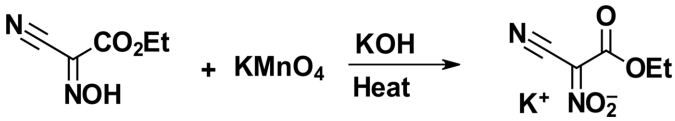


Figure 1
Synthesis scheme for the potassium salt of ethyl nitrocyanoacetate.

2. Structural commentary

The asymmetric unit of the title compound contains a single potassium cation and an anion, the ethyl fragment of which is disordered in a 0.792 (13):0.208 (13) ratio (Fig. 2). All bond lengths are in expected ranges when compared to similar potassium oxalate salt structures. The dihedral angle between the carboxy groups was determined to be 15.2 (6)°. The observed packing interactions (Fig. 3) of this polymeric salt structure are primarily electrostatic interactions between the potassium ion and the oxygen atoms of the 2-ethoxy-2-oxoacetate anion. These distances between potassium and oxygen range from 2.706 (2) to 3.053 (4) Å in length (see Table 1 for all K—O bonds).

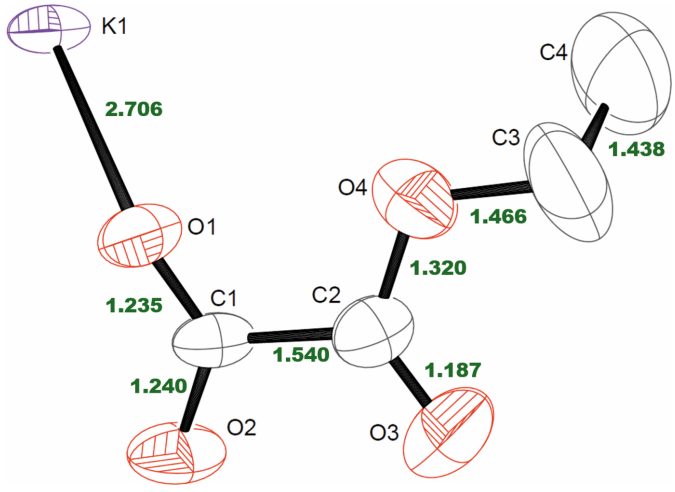


Figure 2
The asymmetric unit showing the potassium cation and the disordered 2-ethoxy-2-oxoacetate anion. Displacement ellipsoids are drawn at the 50% probability level; bonds lengths are detailed in green.

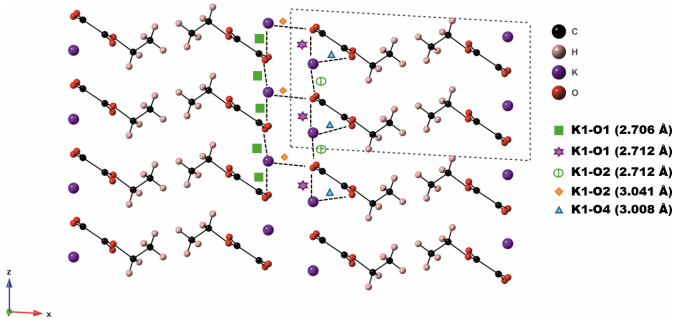


Figure 3
Ball-and-stick representation of the packing of 2-ethoxy-2-oxoacetate molecules and potassium cations viewed down [010]. Several K—O distances are noted with black dotted lines.

Table 1
Selected bond lengths (Å).

K1—O1	2.706 (2)	K1—O1^{iv}	2.855 (3)
K1—O2^{i}	2.712 (2)	K1—O4^{ii}	3.008 (3)
K1—O1^{ii}	2.741 (2)	K1—O2^{iv}	3.041 (3)
K1—O2^{iii}	2.828 (3)	K1—O3^{iii}	3.053 (4)

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

Taking into account K—O distances of less than 3.0 Å, the oxygen atoms arrange in form of a distorted square pyramid around K^+, as represented by a purple polyhedron in Fig. 4. The extended structure reveals the formation of potassium-oxygen chains propagating parallel to [001] (Figs. 4, 5). Within a distance of 3.05 Å, atom O1 binds in a μ_3-mode to three potassium ions while O2 interacts with two cations (Fig. 6). In contrast, both O3 and O4 only interact with one potassium ion each, and at longer distances (Table 1).

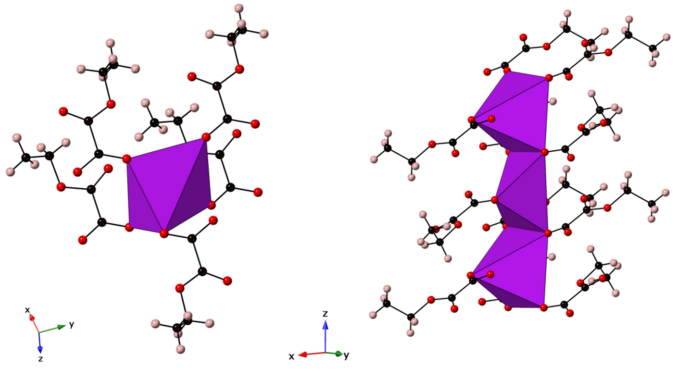


Figure 4
Ball-and-stick representation showing the distorted square-pyramidal coordination environment around the potassium cation as a purple polyhedron.

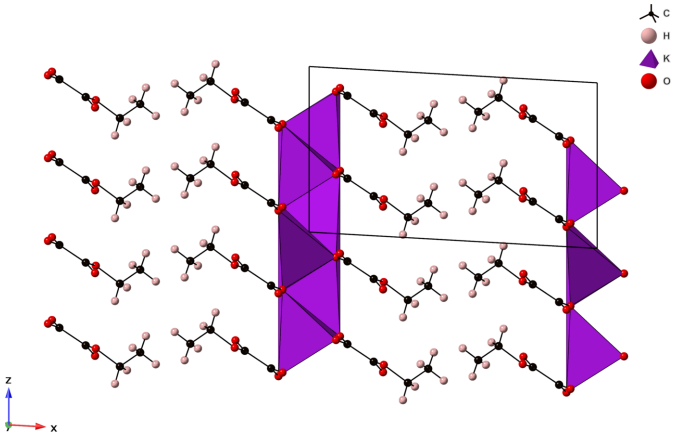


Figure 5
Ball and stick model of the extended structure of the title salt, showing the formation of potassium–oxygen chains propagating along [001] in polyhedral representation.

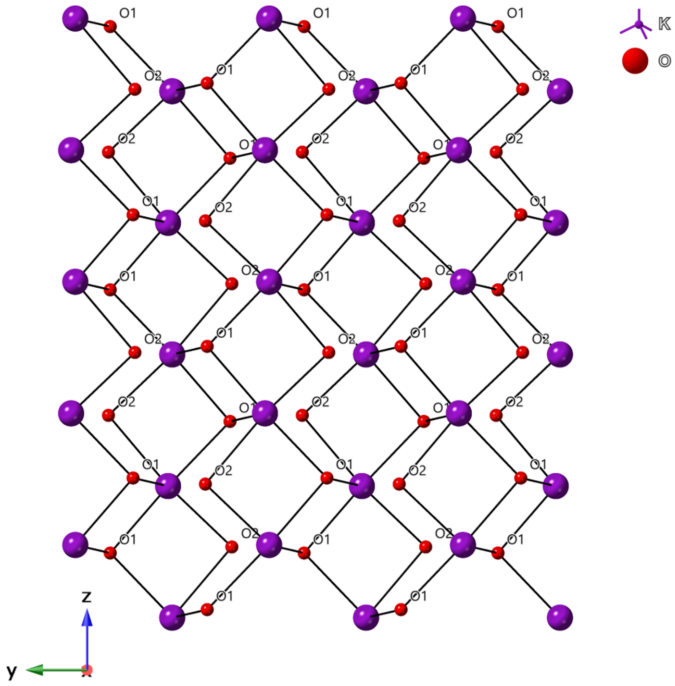


Figure 6
Interactions of O1 and O2 with potassium cations.

3. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.46; Groom *et al.*, 2016) yielded entries containing either 2-ethoxy-2-oxoacetate bound to a metal or containing oxalate moieties bound to potassium as either a backbone structure or a metal coordinating ligand (Fig. 7). The most similar structures found were $K_{0.53}(NH_4)_{0.47}(H_2C_2O_4)$-$((HC_2O_4)H_2O)_2$ (refcode ZAXLOB; Hamdouni *et al.*, 2011) and $[Cu(EtOOC–COO)_2(Hpz)_4]$ (MUVFIT; Garau *et al.*, 2010). The ZAXLOB structure contains both oxalic acid and the hydrogenoxalate anion bound to potassium ions with K—O interaction distances from 2.900 to 2.920 Å. MUVFIT contains two 2-ethoxy-2-oxoacetate units bound to copper(II) through a single oxygen per unit at 2.359 Å, a closer distance than any found in either the title structure or ZAXLOB structure to the potassium ion. An additional five CSD entries were found containing any metal bound to an oxalate group where a single oxygen atom is bonded to a methyl or *tert*-butyl group with another two entries having metal bound to oxalate where two oxygen atoms are bonded to methyl groups. The bound metals include titanium (PUTRUV, Okumura *et al.*, 2025), lithium (OXATIT and OXATOZ; Sun *et al.*, 2021), iridium (IKUJII, Padilla *et al.*, 2010; ILEZEE and ILEZII, Paneque *et al.*, 2003), and osmium (UKEZUI, Lin *et al.*, 2020).

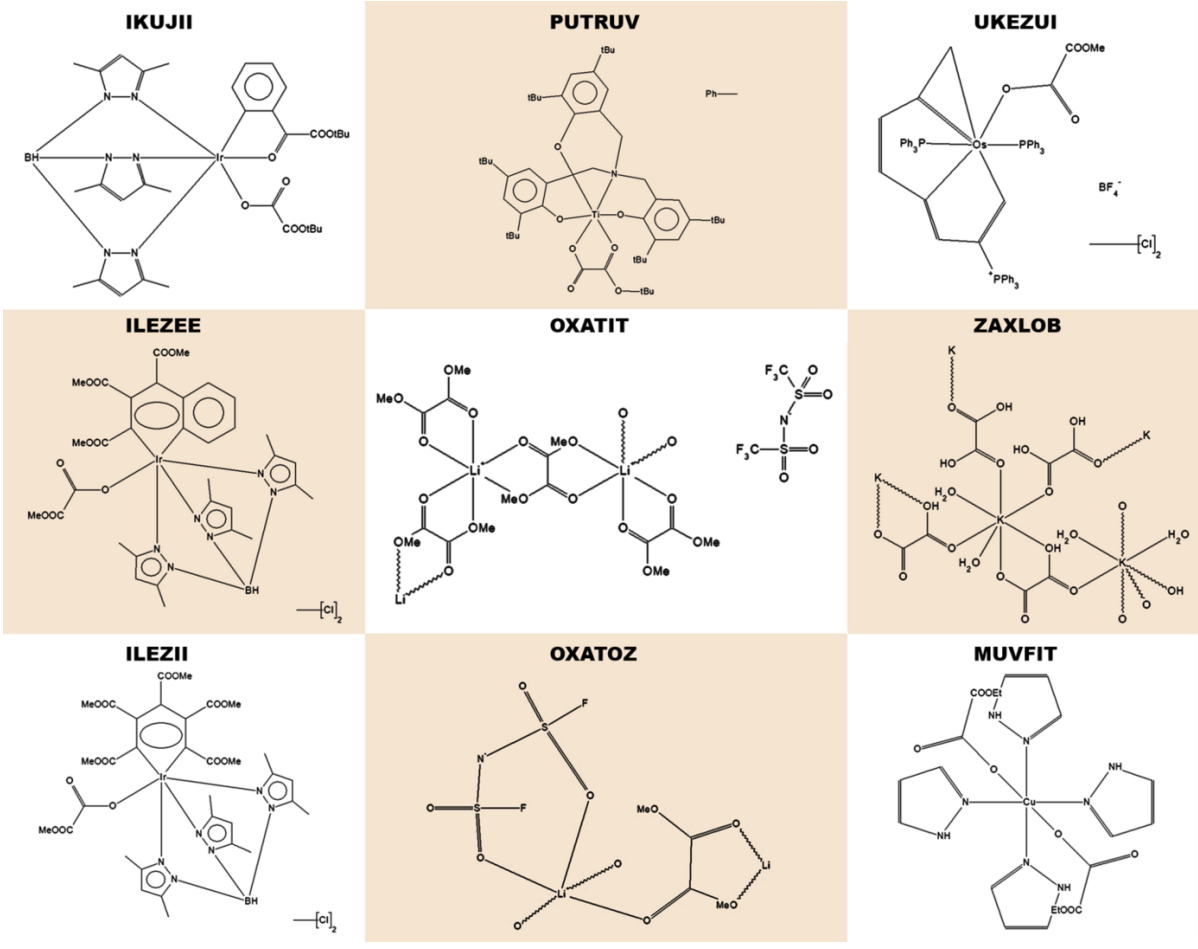


Figure 7
Chemical structures of the compounds referenced in the database survey.

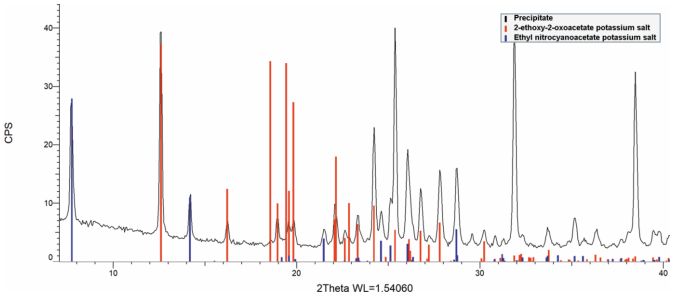


Figure 8
XRD pattern of isolated bulk material with comparison to the calculated pattern of the byproduct potassium 2-ethoxy-2-oxoacetate (red) and the intended product potassium ethyl nitrocyanoacetate (blue). XRD pattern plotted with *DIFFRAC·EVA* with X-offset of 0.09° to correct for sample height.

4. Synthesis and crystallization

A solution of $KMnO_4$ (11.85 g, 75 mmol) in water (200 ml) was added dropwise over 1 h to a warm solution of KOH (0.9 g, 16 mmol) and ethyl (hydroxyimino)cyanoacetate (7.15 g, 50.3 mmol) in water (140 ml). The solution was stirred overnight, until the $KMnO_4$ was fully consumed as shown by the depletion of the dark purple coloring in favor of black. The solution was filtered, removing the black colored solids, and the filtrate concentrated on a rotary evaporator, producing pale-yellow crystalline material. The solids were washed with ethanol and isolated by filtration. The yellow filtrate was stored overnight in a refrigerator and additional material precipitated. From this precipitate, a crystal of the salt potassium 2-ethoxy-2-oxoacetate was isolated for single-crystal X-ray diffraction. Comparison of the calculated pattern to the experimental powder pattern of bulk material collected showed both the byproduct potassium 2-ethoxy-2-oxoacetate and potassium ethyl nitrocyanoacetate present, next to some unassigned reflections (Fig. 8).

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. A PART command in *SHELXL* (Sheldrick, 2015*b*), accompanied by SIMU and RIGU restraints set to 0.01, were used to model the positional disorder within the ethyl group and resulted in a refined ratio of 0.792 (13):0.208 (2). Corresponding H atoms were included using a riding model for the methyl and methylene groups.

Funding information

We would like to thank the Office of Naval Research (ONR) and the U·S. Naval Research Laboratory (NRL) for their generous support of this base 6.1 program.

Table 2
Experimental details.

Crystal data	
Chemical formula	$K^+·C_4H_5O_4^-$
M_r	156.18
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	14.052 (2), 5.9280 (8), 8.0579 (10)
β (°)	93.242 (5)
V (Å^3)	670.15 (16)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.73
Crystal size (mm)	0.12 × 0.09 × 0.02
Data collection	
Diffractometer	Photon II CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.660, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10487, 1593, 1192
R_{int}	0.059
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.657
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.064, 0.150, 1.10
No. of reflections	1593
No. of parameters	103
No. of restraints	43
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.42, −0.31

Computer programs: *APEX3* and *SAINT* (Bruker, 2019), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *SHELXTL* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

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Byproduct identification: crystal structure of potassium 2-ethoxy-2-oxoacetate

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Computing details

Potassium 2-ethoxy-2-oxoacetate

Crystal data

$K^+ \cdot C_4H_5O_4^-$
$M_r = 156.18$
Monoclinic, $P2_1/c$
$a = 14.052$ (2) Å
$b = 5.9280$ (8) Å
$c = 8.0579$ (10) Å
$\beta = 93.242$ (5)°
$V = 670.15$ (16) Å^3
$Z = 4$
$F(000) = 320$
$D_x = 1.548$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1931 reflections
$\theta = 2.9$–27.2°
$\mu = 0.73$ mm^{-1}
$T = 296$ K
Plate, yellow
0.12 × 0.09 × 0.02 mm

Data collection

Photon II CCD diffractometer
Radiation source: 1uS 3.0 microfocus
Helios multilayer optics monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
$T_{min} = 0.660$, $T_{max} = 0.746$
10487 measured reflections
1593 independent reflections
1192 reflections with $I > 2\sigma(I)$
$R_{int} = 0.059$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.9°$
$h = -17 \rightarrow 18$
$k = -7 \rightarrow 7$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.150$
$S = 1.10$
1593 reflections
103 parameters
43 restraints
Primary atom site location: intrinsic phasing
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2 + 0.7606P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
K1	0.09360 (6)	0.26080 (10)	0.61193 (8)	0.0455 (3)	
O1	0.0930 (2)	0.5589 (4)	0.3575 (3)	0.0552 (7)	
O2	0.1067 (2)	0.9317 (4)	0.3806 (3)	0.0650 (8)	
O3	0.2563 (3)	0.9045 (6)	0.1792 (5)	0.0985 (12)	
O4	0.2588 (2)	0.5356 (5)	0.2237 (4)	0.0761 (9)	
C1	0.1325 (3)	0.7420 (5)	0.3364 (4)	0.0436 (8)	
C2	0.2238 (3)	0.7407 (6)	0.2392 (5)	0.0552 (9)	
C3	0.3425 (7)	0.521 (2)	0.1236 (14)	0.114 (3)	0.792 (13)
H3A	0.325405	0.450607	0.017359	0.137*	0.792 (13)
H3B	0.367178	0.670628	0.103228	0.137*	0.792 (13)
C4	0.4131 (6)	0.388 (2)	0.2152 (16)	0.144 (4)	0.792 (13)
H4A	0.467205	0.366411	0.149261	0.216*	0.792 (13)
H4B	0.386482	0.243678	0.241020	0.216*	0.792 (13)
H4C	0.432783	0.464288	0.316375	0.216*	0.792 (13)
C3A	0.376 (3)	0.547 (8)	0.198 (6)	0.122 (5)	0.208 (13)
H3AA	0.395966	0.686855	0.149019	0.147*	0.208 (13)
H3AB	0.414355	0.513027	0.298734	0.147*	0.208 (13)
C4A	0.369 (3)	0.362 (7)	0.083 (5)	0.125 (6)	0.208 (13)
H4AA	0.345767	0.416681	−0.024271	0.187*	0.208 (13)
H4AB	0.325420	0.251309	0.121351	0.187*	0.208 (13)
H4AC	0.430521	0.295372	0.074146	0.187*	0.208 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0772 (6)	0.0234 (3)	0.0364 (4)	0.0041 (3)	0.0082 (3)	0.0011 (3)
O1	0.0809 (19)	0.0348 (12)	0.0516 (14)	−0.0030 (12)	0.0190 (13)	0.0021 (10)
O2	0.103 (2)	0.0317 (13)	0.0605 (17)	0.0068 (13)	0.0097 (16)	−0.0088 (11)
O3	0.103 (3)	0.068 (2)	0.128 (3)	−0.0207 (19)	0.040 (2)	0.016 (2)
O4	0.078 (2)	0.0634 (18)	0.089 (2)	0.0200 (15)	0.0244 (17)	0.0030 (16)
C1	0.069 (2)	0.0284 (14)	0.0332 (14)	0.0018 (15)	0.0006 (14)	−0.0003 (12)
C2	0.066 (2)	0.0461 (19)	0.053 (2)	−0.0061 (18)	0.0035 (18)	−0.0002 (16)
C3	0.074 (6)	0.145 (7)	0.125 (8)	0.045 (5)	0.029 (5)	0.008 (6)
C4	0.082 (6)	0.163 (8)	0.188 (10)	0.035 (6)	0.021 (6)	0.045 (8)
C3A	0.080 (10)	0.147 (10)	0.144 (12)	0.047 (9)	0.035 (9)	0.015 (10)
C4A	0.081 (12)	0.151 (12)	0.146 (13)	0.033 (11)	0.032 (11)	0.018 (12)

Geometric parameters (Å, º)

K1—O1	2.706 (2)	O4—C3	1.465 (9)
K1—O2^{i}	2.712 (2)	O4—C3A	1.67 (4)
K1—O1^{ii}	2.741 (2)	C1—C2	1.540 (5)
K1—O2^{iii}	2.828 (3)	C3—C4	1.439 (12)
K1—O1^{iv}	2.855 (3)	C3—H3A	0.9700
K1—O4^{ii}	3.008 (3)	C3—H3B	0.9700

K1—O2^{iv}	3.041 (3)	C4—H4A	0.9600
K1—O3^{iii}	3.053 (4)	C4—H4B	0.9600
K1—C1^{iv}	3.228 (4)	C4—H4C	0.9600
K1—C1^{iii}	3.485 (3)	C3A—C4A	1.44 (2)
K1—C1^{ii}	3.513 (3)	C3A—H3AA	0.9700
K1—K1^{ii}	4.0310 (5)	C3A—H3AB	0.9700
O1—C1	1.235 (4)	C4A—H4AA	0.9600
O2—C1	1.240 (4)	C4A—H4AB	0.9600
O3—C2	1.187 (5)	C4A—H4AC	0.9600
O4—C2	1.320 (4)		

O1—K1—O2^{i}	86.93 (8)	C1^{iv}—K1—K1^{ii}	79.35 (6)
O1—K1—O1^{ii}	176.99 (7)	C1^{iii}—K1—K1^{ii}	61.12 (5)
O2^{i}—K1—O1^{ii}	90.11 (8)	C1^{ii}—K1—K1^{ii}	57.81 (5)
O1—K1—O2^{iii}	98.99 (7)	C1—O1—K1	134.1 (2)
O2^{i}—K1—O2^{iii}	170.36 (12)	C1—O1—K1^{v}	119.2 (2)
O1^{ii}—K1—O2^{iii}	84.01 (7)	K1—O1—K1^{v}	95.47 (7)
O1—K1—O1^{iv}	81.80 (8)	C1—O1—K1^{iv}	96.1 (2)
O2^{i}—K1—O1^{iv}	115.28 (9)	K1—O1—K1^{iv}	98.20 (8)
O1^{ii}—K1—O1^{iv}	98.99 (6)	K1^{v}—O1—K1^{iv}	111.19 (10)
O2^{iii}—K1—O1^{iv}	73.33 (8)	C1—O2—K1^{vi}	151.9 (2)
O1—K1—O4^{ii}	125.26 (8)	C1—O2—K1^{vii}	111.8 (2)
O2^{i}—K1—O4^{ii}	72.80 (9)	K1^{vi}—O2—K1^{vii}	93.36 (7)
O1^{ii}—K1—O4^{ii}	54.12 (8)	C1—O2—K1^{iv}	87.2 (2)
O2^{iii}—K1—O4^{ii}	97.56 (9)	K1^{vi}—O2—K1^{iv}	99.06 (9)
O1^{iv}—K1—O4^{ii}	152.84 (8)	K1^{vii}—O2—K1^{iv}	103.70 (9)
O1—K1—O2^{iv}	107.27 (8)	C2—O3—K1^{vii}	107.6 (3)
O2^{i}—K1—O2^{iv}	80.94 (9)	C2—O4—C3	114.9 (6)
O1^{ii}—K1—O2^{iv}	71.67 (8)	C2—O4—C3A	110.5 (16)
O2^{iii}—K1—O2^{iv}	104.30 (6)	C2—O4—K1^{v}	106.3 (3)
O1^{iv}—K1—O2^{iv}	44.20 (7)	C3—O4—K1^{v}	115.5 (6)
O4^{ii}—K1—O2^{iv}	118.32 (8)	O1—C1—O2	128.0 (4)
O1—K1—O3^{iii}	71.36 (10)	O1—C1—C2	117.4 (3)
O2^{i}—K1—O3^{iii}	120.69 (9)	O2—C1—C2	114.6 (3)
O1^{ii}—K1—O3^{iii}	110.74 (10)	O1—C1—K1^{iv}	61.6 (2)
O2^{iii}—K1—O3^{iii}	55.21 (9)	O2—C1—K1^{iv}	70.2 (2)
O1^{iv}—K1—O3^{iii}	115.07 (8)	C2—C1—K1^{iv}	156.9 (2)
O4^{ii}—K1—O3^{iii}	76.63 (9)	O1—C1—K1^{vii}	139.1 (3)
O2^{iv}—K1—O3^{iii}	157.70 (8)	O2—C1—K1^{vii}	48.87 (17)
O1—K1—C1^{iv}	98.06 (8)	C2—C1—K1^{vii}	81.45 (18)
O2^{i}—K1—C1^{iv}	100.99 (9)	K1^{iv}—C1—K1^{vii}	86.81 (8)
O1^{ii}—K1—C1^{iv}	81.96 (8)	O1—C1—K1^{v}	42.90 (15)
O2^{iii}—K1—C1^{iv}	85.78 (8)	O2—C1—K1^{v}	151.4 (3)
O1^{iv}—K1—C1^{iv}	22.36 (7)	C2—C1—K1^{v}	81.02 (18)
O4^{ii}—K1—C1^{iv}	134.99 (8)	K1^{iv}—C1—K1^{v}	86.35 (8)
O2^{iv}—K1—C1^{iv}	22.57 (6)	K1^{vii}—C1—K1^{v}	115.78 (9)
O3^{iii}—K1—C1^{iv}	135.50 (8)	O3—C2—O4	124.1 (4)
O1—K1—C1^{iii}	80.47 (7)	O3—C2—C1	123.6 (4)

O2^{i}—K1—C1^{iii}	161.88 (9)	O4—C2—C1	112.2 (3)
O1^{ii}—K1—C1^{iii}	102.53 (7)	C4—C3—O4	107.6 (8)
O2^{iii}—K1—C1^{iii}	19.29 (7)	C4—C3—H3A	110.2
O1^{iv}—K1—C1^{iii}	75.92 (8)	O4—C3—H3A	110.2
O4^{ii}—K1—C1^{iii}	104.05 (9)	C4—C3—H3B	110.2
O2^{iv}—K1—C1^{iii}	115.18 (8)	O4—C3—H3B	110.2
O3^{iii}—K1—C1^{iii}	42.61 (9)	H3A—C3—H3B	108.5
C1^{iv}—K1—C1^{iii}	93.68 (9)	C3—C4—H4A	109.5
O1—K1—C1^{ii}	159.67 (7)	C3—C4—H4B	109.5
O2^{i}—K1—C1^{ii}	74.27 (8)	H4A—C4—H4B	109.5
O1^{ii}—K1—C1^{ii}	17.87 (7)	C3—C4—H4C	109.5
O2^{iii}—K1—C1^{ii}	98.69 (7)	H4A—C4—H4C	109.5
O1^{iv}—K1—C1^{ii}	112.97 (8)	H4B—C4—H4C	109.5
O4^{ii}—K1—C1^{ii}	41.87 (8)	C4A—C3A—O4	91 (3)
O2^{iv}—K1—C1^{ii}	77.81 (8)	C4A—C3A—H3AA	113.5
O3^{iii}—K1—C1^{ii}	111.51 (10)	O4—C3A—H3AA	113.5
C1^{iv}—K1—C1^{ii}	93.16 (8)	C4A—C3A—H3AB	113.5
C1^{iii}—K1—C1^{ii}	115.79 (9)	O4—C3A—H3AB	113.5
O1—K1—K1^{ii}	141.05 (6)	H3AA—C3A—H3AB	110.8
O2^{i}—K1—K1^{ii}	131.92 (6)	C3A—C4A—H4AA	109.5
O1^{ii}—K1—K1^{ii}	41.94 (5)	C3A—C4A—H4AB	109.5
O2^{iii}—K1—K1^{ii}	42.18 (5)	H4AA—C4A—H4AB	109.5
O1^{iv}—K1—K1^{ii}	82.75 (5)	C3A—C4A—H4AC	109.5
O4^{ii}—K1—K1^{ii}	74.10 (6)	H4AA—C4A—H4AC	109.5
O2^{iv}—K1—K1^{ii}	85.18 (5)	H4AB—C4A—H4AC	109.5
O3^{iii}—K1—K1^{ii}	83.42 (8)		

K1—O1—C1—O2	−83.4 (5)	K1^{vii}—O2—C1—K1^{v}	−66.8 (5)
K1^{v}—O1—C1—O2	142.7 (3)	K1^{iv}—O2—C1—K1^{v}	36.9 (5)
K1^{iv}—O1—C1—O2	24.2 (4)	K1^{vii}—O3—C2—O4	151.2 (4)
K1—O1—C1—C2	98.6 (3)	K1^{vii}—O3—C2—C1	−25.4 (5)
K1^{v}—O1—C1—C2	−35.4 (4)	C3—O4—C2—O3	−0.5 (8)
K1^{iv}—O1—C1—C2	−153.8 (3)	C3A—O4—C2—O3	28.4 (19)
K1—O1—C1—K1^{iv}	−107.6 (3)	K1^{v}—O4—C2—O3	−129.6 (4)
K1^{v}—O1—C1—K1^{iv}	118.4 (2)	C3—O4—C2—C1	176.4 (6)
K1—O1—C1—K1^{vii}	−151.46 (18)	C3A—O4—C2—C1	−154.7 (18)
K1^{v}—O1—C1—K1^{vii}	74.6 (4)	K1^{v}—O4—C2—C1	47.4 (3)
K1^{iv}—O1—C1—K1^{vii}	−43.8 (3)	O1—C1—C2—O3	162.8 (4)
K1—O1—C1—K1^{v}	134.0 (4)	O2—C1—C2—O3	−15.6 (6)
K1^{iv}—O1—C1—K1^{v}	−118.4 (2)	K1^{iv}—C1—C2—O3	81.5 (7)
K1^{vi}—O2—C1—O1	81.6 (7)	K1^{vii}—C1—C2—O3	21.2 (4)
K1^{vii}—O2—C1—O1	−126.2 (3)	K1^{v}—C1—C2—O3	139.2 (4)
K1^{iv}—O2—C1—O1	−22.6 (4)	O1—C1—C2—O4	−14.2 (5)
K1^{vi}—O2—C1—C2	−100.3 (5)	O2—C1—C2—O4	167.5 (3)
K1^{vii}—O2—C1—C2	51.9 (4)	K1^{iv}—C1—C2—O4	−95.4 (6)
K1^{iv}—O2—C1—C2	155.5 (3)	K1^{vii}—C1—C2—O4	−155.7 (3)
K1^{vi}—O2—C1—K1^{iv}	104.1 (5)	K1^{v}—C1—C2—O4	−37.7 (3)
K1^{vii}—O2—C1—K1^{iv}	−103.65 (16)	C2—O4—C3—C4	132.1 (10)

K1^{vi}—O2—C1—K1^{vii}	−152.2 (6)	K1^{v}—O4—C3—C4	−103.5 (11)
K1^{iv}—O2—C1—K1^{vii}	103.65 (16)	C2—O4—C3A—C4A	−143 (3)
K1^{vi}—O2—C1—K1^{v}	141.0 (4)	K1^{v}—O4—C3A—C4A	5 (5)

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