

Potassium oxalurate monohydrate

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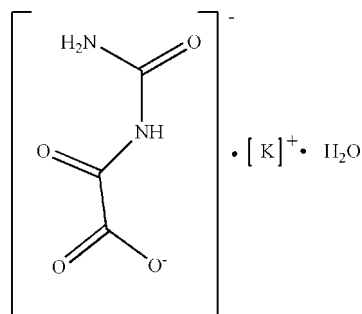
Received 7 February 2009; accepted 17 February 2009

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å;
 R factor = 0.026; wR factor = 0.077; data-to-parameter ratio = 10.6.

The title salt, poly[aqua- μ_3 -oxalurate-potassium(I)], $[\text{K}(\text{C}_3\text{H}_3\text{N}_2\text{O}_4)(\text{H}_2\text{O})]_n$, which was obtained from a water solution of oxaluric acid and KOH at room temperature, crystallizes as potassium and oxalurate ions along with a water molecule. The K^+ cation lies on a crystallographic twofold rotation axis (site symmetry 2, Wyckoff position f), and the water and oxalurate molecules are located within different mirror planes (site symmetry m , Wyckoff position g). The K^+ cation is eight-coordinated by six O atoms of six oxalurate ligands and two O atoms from two water molecules in a distorted square-antiprismatic geometry. All of the eight coordinated O atoms are in a monodentate bridging mode, with alternate bridged $\text{K}\cdots\text{K}$ distances of 3.5575 (12) and 3.3738 (12) Å. The oxalurate ligand shows a μ_3 -bridging coordination mode, which links the K^+ cation into a three-dimensional network. The oxalurate ligands and the water molecules are involved in inter- and intramolecular $\text{N}-\text{H}\cdots\text{O}$, and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, which stabilize the network.

Related literature

For oxalurate metal complexes, see: Falvello *et al.* (2002). For elongated $\text{K}-\text{O}$ bonds, see: Karapetyan (2008); Kunz *et al.* (2009).



Experimental

Crystal data

$[\text{K}(\text{C}_3\text{H}_3\text{N}_2\text{O}_4)(\text{H}_2\text{O})]$	$V = 685.9$ (3) Å ³
$M_r = 188.19$	$Z = 4$
Orthorhombic, $Pnmm$	Mo $K\alpha$ radiation
$a = 7.7313$ (17) Å	$\mu = 0.75$ mm ⁻¹
$b = 12.799$ (3) Å	$T = 296$ K
$c = 6.9313$ (16) Å	$0.41 \times 0.39 \times 0.28$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	3320 measured reflections
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 1997)	699 independent reflections
$T_{\min} = 0.748$, $T_{\max} = 0.816$	633 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.013$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$	66 parameters
$wR(F^2) = 0.077$	H-atom parameters constrained
$S = 1.09$	$\Delta\rho_{\text{max}} = 0.17$ e Å ⁻³
699 reflections	$\Delta\rho_{\text{min}} = -0.34$ e Å ⁻³

Table 1

Selected bond lengths (Å).

$\text{K1}-\text{O1}$	2.7291 (11)	$\text{K1}-\text{O5}$	2.8458 (13)
$\text{K1}-\text{O3}^{\text{i}}$	2.7812 (11)	$\text{K1}-\text{O4}^{\text{ii}}$	2.9775 (13)

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

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O4}^{\text{iii}}$	0.86	2.10	2.936 (2)	164
$\text{N2}-\text{H2A}\cdots\text{O1}^{\text{iv}}$	0.86	2.17	2.997 (2)	163
$\text{N2}-\text{H2A}\cdots\text{O2}^{\text{iv}}$	0.86	2.37	3.069 (2)	139
$\text{N2}-\text{H2B}\cdots\text{O3}$	0.86	2.01	2.667 (2)	133
$\text{N2}-\text{H2B}\cdots\text{O5}^{\text{v}}$	0.86	2.38	3.076 (3)	138
$\text{O5}-\text{H1W}\cdots\text{O2}^{\text{i}}$	0.83	1.97	2.791 (2)	172
$\text{O5}-\text{H1W}\cdots\text{O3}^{\text{i}}$	0.83	2.59	3.068 (2)	118
$\text{O5}-\text{H2W}\cdots\text{O2}^{\text{vi}}$	0.83	2.14	2.973 (2)	178

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors thank Nan Yang Normal University for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2155).

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supplementary materials

Acta Cryst. (2009). E65, m308-m309 [doi:10.1107/S1600536809005637]

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Comment

Oxaluric acid is the condensation product of oxalic acid and urea. Deprotonated oxalurate possesses four oxygen atoms and two amine N atoms, which can serve as hydrogen-bond acceptors and hydrogen-bond donors, respectively. In addition, one or more of six different atoms can bind to metal centers in any of at least three distinct coordination modes, namely, chelating, terminal, or bridging coordination (Falvello, 2002).

As shown in Fig. 1, the asymmetric structure unit consists of one K^+ cation, one $C_3H_3N_2O_4^-$ anion, and one water molecule. The K^+ cation is surrounded by six oxalurate ligands and two water molecules, making close contacts with eight O atoms at 2.7291 (11)–2.9775 (13) Å in a distorted square antiprismatic geometry of the central atom (Karapetyan, 2008; Kunz, 2009) (Table 1). All the eight coordinated O atoms are in the monodentate bridging mode, with the bridged $K\cdots K$ distance of 3.558 (1) and 3.374 (1) Å alternately. The oxalurate ligand, which is planar, shows a μ_3 -bridging coordination mode and links the K^+ cation into a three-dimensional network (Fig. 2). The oxalurate ligands and water molecules are involved in inter- and intramolecular $N-H\cdots O$, and $O-H\cdots O$ hydrogen bonds, which stabilize the network (Table 2).

Experimental

A 10 ml sample of a KOH solution (0.5 mol/L) was added to a water suspension of oxaluric acid, $HOCCONHCONH_2$ (0.5 mmol/10 ml). The KOH addition produced a partial solubilization of the acid and then the precipitation of a white solid. After 20 min of stirring, the solid was filtered off, washed with *i*-PrOH. The single crystals suitable for X-ray analysis were obtained by slow diffusion of Et_2O into the water solution of the solid.

Refinement

Water H atoms were located in a difference Fourier and allowed to ride at the value approximately 0.83 Å with $U_{iso}(H) = 1.5U_{eq}(O)$. Other H atoms were positioned geometrically and treated as riding, with $N-H = 0.86$ Å (NH and NH_2) and $U_{iso}(H) = 1.2U_{eq}(N)$.

Figures

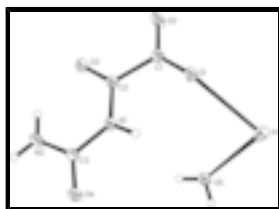


Fig. 1. A perspective view of the asymmetric unit, showing the atomic numbering and displacement ellipsoids drawn at the 30% probability level.

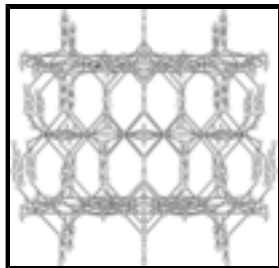


Fig. 2. A view of the compound packing down the *a* axis.

poly[aqua- μ_3 -oxalurate-potassium(I)]

Crystal data

[K(C₃H₃N₂O₄)(H₂O)]

$M_r = 188.19$

Orthorhombic, *Pnmm*

Hall symbol: -P 2 2n

$a = 7.7313 (17) \text{ \AA}$

$b = 12.799 (3) \text{ \AA}$

$c = 6.9313 (16) \text{ \AA}$

$V = 685.9 (3) \text{ \AA}^3$

$Z = 4$

$F_{000} = 384$

$D_x = 1.823 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1939 reflections

$\theta = 2.9\text{--}28.2^\circ$

$\mu = 0.75 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Block, pink

$0.41 \times 0.39 \times 0.28 \text{ mm}$

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 296 \text{ K}$

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 1997)

$T_{\min} = 0.748$, $T_{\max} = 0.816$

3320 measured reflections

699 independent reflections

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

$R_{\text{int}} = 0.013$

$\theta_{\max} = 25.5^\circ$

$\theta_{\min} = 3.1^\circ$

$h = -9 \rightarrow 9$

$k = -15 \rightarrow 15$

$l = -8 \rightarrow 6$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.026$

$wR(F^2) = 0.077$

$S = 1.09$

699 reflections

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 0.2142P]$

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

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

$\Delta\rho_{\max} = 0.17 \text{ e \AA}^{-3}$

66 parameters

$$\Delta\rho_{\min} = -0.34 \text{ e } \text{\AA}^{-3}$$

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
K1	1.0000	0.5000	0.24337 (6)	0.0336 (2)
O1	0.89541 (18)	0.34475 (10)	0.0000	0.0329 (4)
O2	0.90620 (19)	0.17010 (11)	0.0000	0.0478 (5)
O3	0.55768 (18)	0.16337 (10)	0.0000	0.0349 (4)
O4	0.30509 (18)	0.44721 (10)	0.0000	0.0396 (4)
N1	0.5439 (2)	0.34235 (12)	0.0000	0.0278 (4)
H1	0.6069	0.3977	0.0000	0.033*
N2	0.2646 (2)	0.27328 (14)	0.0000	0.0414 (5)
H2A	0.1538	0.2794	0.0000	0.050*
H2B	0.3111	0.2122	0.0000	0.050*
C1	0.8300 (3)	0.25554 (15)	0.0000	0.0266 (5)
C2	0.6290 (2)	0.24914 (14)	0.0000	0.0239 (4)
C3	0.3632 (2)	0.35754 (14)	0.0000	0.0281 (5)
O5	0.7184 (2)	0.46547 (13)	0.5000	0.0490 (5)
H1W	0.6919	0.5284	0.5000	0.073*
H2W	0.6298	0.4287	0.5000	0.073*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0406 (3)	0.0241 (3)	0.0362 (4)	-0.00314 (16)	0.000	0.000
O1	0.0213 (7)	0.0206 (7)	0.0567 (10)	-0.0038 (5)	0.000	0.000
O2	0.0210 (7)	0.0224 (7)	0.0999 (15)	0.0037 (6)	0.000	0.000
O3	0.0226 (7)	0.0171 (7)	0.0650 (11)	-0.0027 (6)	0.000	0.000
O4	0.0227 (7)	0.0203 (7)	0.0758 (12)	0.0033 (6)	0.000	0.000
N1	0.0175 (8)	0.0165 (8)	0.0493 (11)	-0.0020 (6)	0.000	0.000
N2	0.0172 (8)	0.0219 (8)	0.0852 (16)	0.0006 (7)	0.000	0.000
C1	0.0194 (10)	0.0224 (9)	0.0379 (11)	-0.0015 (7)	0.000	0.000
C2	0.0197 (10)	0.0190 (9)	0.0330 (11)	-0.0007 (7)	0.000	0.000
C3	0.0184 (9)	0.0221 (9)	0.0438 (13)	0.0014 (7)	0.000	0.000
O5	0.0247 (8)	0.0271 (8)	0.0952 (14)	0.0024 (7)	0.000	0.000

Geometric parameters (\AA , $^\circ$)

K1—O1	2.7291 (11)	O3—K1 ^{vii}	2.7812 (11)
K1—O1 ⁱ	2.7291 (11)	O3—K1 ^{viii}	2.7812 (11)
K1—O3 ⁱⁱ	2.7812 (11)	O4—C3	1.232 (2)
K1—O3 ⁱⁱⁱ	2.7812 (11)	O4—K1 ^v	2.9775 (13)
K1—O5 ^{iv}	2.8458 (13)	O4—K1 ^{ix}	2.9775 (13)
K1—O5	2.8458 (13)	N1—C2	1.362 (2)

supplementary materials

K1—O4 ^v	2.9775 (13)	N1—C3	1.410 (2)
K1—O4 ^{vi}	2.9775 (13)	N1—H1	0.8600
K1—K1 ⁱ	3.3738 (12)	N2—C3	1.321 (3)
K1—K1 ^{iv}	3.5575 (12)	N2—H2A	0.8600
K1—H1W	2.9950	N2—H2B	0.8600
O1—C1	1.249 (2)	C1—C2	1.556 (3)
O1—K1 ⁱ	2.7291 (11)	O5—K1 ^{iv}	2.8458 (13)
O2—C1	1.242 (2)	O5—H1W	0.8312
O3—C2	1.228 (2)	O5—H2W	0.8317
O1—K1—O1 ⁱ	103.64 (4)	O3 ⁱⁱ —K1—K1 ^{iv}	50.24 (2)
O1—K1—O3 ⁱⁱ	153.54 (4)	O3 ⁱⁱⁱ —K1—K1 ^{iv}	50.24 (2)
O1 ⁱ —K1—O3 ⁱⁱ	84.00 (3)	O5 ^{iv} —K1—K1 ^{iv}	51.32 (2)
O1—K1—O3 ⁱⁱⁱ	84.00 (3)	O5—K1—K1 ^{iv}	51.32 (2)
O1 ⁱ —K1—O3 ⁱⁱⁱ	153.54 (4)	O4 ^v —K1—K1 ^{iv}	124.509 (19)
O3 ⁱⁱ —K1—O3 ⁱⁱⁱ	100.48 (4)	O4 ^{vi} —K1—K1 ^{iv}	124.509 (19)
O1—K1—O5 ^{iv}	136.56 (4)	K1 ⁱ —K1—K1 ^{iv}	180.0
O1 ⁱ —K1—O5 ^{iv}	92.67 (4)	C1—O1—K1	141.79 (2)
O3 ⁱⁱ —K1—O5 ^{iv}	66.81 (4)	C1—O1—K1 ⁱ	141.79 (2)
O3 ⁱⁱⁱ —K1—O5 ^{iv}	66.07 (4)	K1—O1—K1 ⁱ	76.36 (4)
O1—K1—O5	92.67 (4)	C2—O3—K1 ^{vii}	138.06 (4)
O1 ⁱ —K1—O5	136.56 (4)	C2—O3—K1 ^{viii}	138.06 (4)
O3 ⁱⁱ —K1—O5	66.07 (4)	K1 ^{vii} —O3—K1 ^{viii}	79.52 (4)
O3 ⁱⁱⁱ —K1—O5	66.81 (4)	C3—O4—K1 ^v	120.01 (9)
O5 ^{iv} —K1—O5	102.63 (4)	C3—O4—K1 ^{ix}	120.01 (9)
O1—K1—O4 ^v	65.19 (4)	K1 ^v —O4—K1 ^{ix}	69.02 (4)
O1 ⁱ —K1—O4 ^v	73.70 (4)	C2—N1—C3	126.81 (16)
O3 ⁱⁱ —K1—O4 ^v	93.71 (3)	C2—N1—H1	116.6
O3 ⁱⁱⁱ —K1—O4 ^v	131.30 (4)	C3—N1—H1	116.6
O5 ^{iv} —K1—O4 ^v	157.65 (5)	C3—N2—H2A	120.0
O5—K1—O4 ^v	77.49 (3)	C3—N2—H2B	120.0
O1—K1—O4 ^{vi}	73.70 (4)	H2A—N2—H2B	120.0
O1 ⁱ —K1—O4 ^{vi}	65.19 (4)	O2—C1—O1	127.80 (18)
O3 ⁱⁱ —K1—O4 ^{vi}	131.30 (4)	O2—C1—C2	115.29 (17)
O3 ⁱⁱⁱ —K1—O4 ^{vi}	93.71 (3)	O1—C1—C2	116.91 (17)
O5 ^{iv} —K1—O4 ^{vi}	77.49 (3)	O3—C2—N1	124.44 (17)
O5—K1—O4 ^{vi}	157.65 (5)	O3—C2—C1	119.70 (17)
O4 ^v —K1—O4 ^{vi}	110.98 (4)	N1—C2—C1	115.87 (16)
O1—K1—K1 ⁱ	51.82 (2)	O4—C3—N2	123.36 (18)
O1 ⁱ —K1—K1 ⁱ	51.82 (2)	O4—C3—N1	119.31 (17)
O3 ⁱⁱ —K1—K1 ⁱ	129.76 (2)	N2—C3—N1	117.34 (17)
O3 ⁱⁱⁱ —K1—K1 ⁱ	129.76 (2)	K1 ^{iv} —O5—K1	77.37 (4)

O5 ^{iv} —K1—K1 ⁱ	128.68 (2)	K1 ^{iv} —O5—H1W	92.2
O5—K1—K1 ⁱ	128.68 (2)	K1—O5—H1W	92.2
O4 ^v —K1—K1 ⁱ	55.491 (19)	K1 ^{iv} —O5—H2W	135.9
O4 ^{vi} —K1—K1 ⁱ	55.491 (19)	K1—O5—H2W	135.9
O1—K1—K1 ^{iv}	128.18 (2)	H1W—O5—H2W	110.2
O1 ⁱ —K1—K1 ^{iv}	128.18 (2)		
O1 ⁱ —K1—O1—C1	-177.2 (2)	K1 ^{vii} —O3—C2—C1	-73.10 (14)
O3 ⁱⁱ —K1—O1—C1	-72.9 (2)	K1 ^{viii} —O3—C2—C1	73.10 (14)
O3 ⁱⁱⁱ —K1—O1—C1	28.56 (19)	C3—N1—C2—O3	0.0
O5 ^{iv} —K1—O1—C1	73.8 (2)	C3—N1—C2—C1	180.0
O5—K1—O1—C1	-37.78 (19)	O2—C1—C2—O3	0.0
O4 ^v —K1—O1—C1	-112.67 (19)	O1—C1—C2—O3	180.0
O4 ^{vi} —K1—O1—C1	124.22 (19)	O2—C1—C2—N1	180.0
K1 ⁱ —K1—O1—C1	-177.2 (2)	O1—C1—C2—N1	0.0
K1 ^{iv} —K1—O1—C1	2.8 (2)	K1 ^v —O4—C3—N2	-40.86 (6)
O1 ⁱ —K1—O1—K1 ⁱ	0.0	K1 ^{ix} —O4—C3—N2	40.86 (6)
O3 ⁱⁱ —K1—O1—K1 ⁱ	104.25 (5)	K1 ^v —O4—C3—N1	139.14 (6)
O3 ⁱⁱⁱ —K1—O1—K1 ⁱ	-154.26 (4)	K1 ^{ix} —O4—C3—N1	-139.14 (6)
O5 ^{iv} —K1—O1—K1 ⁱ	-109.03 (4)	C2—N1—C3—O4	180.0
O5—K1—O1—K1 ⁱ	139.40 (3)	C2—N1—C3—N2	0.0
O4 ^v —K1—O1—K1 ⁱ	64.50 (3)	O1—K1—O5—K1 ^{iv}	139.06 (3)
O4 ^{vi} —K1—O1—K1 ⁱ	-58.60 (3)	O1 ⁱ —K1—O5—K1 ^{iv}	-107.82 (4)
K1 ^{iv} —K1—O1—K1 ⁱ	180.0	O3 ⁱⁱ —K1—O5—K1 ^{iv}	-57.25 (3)
K1—O1—C1—O2	-92.22 (16)	O3 ⁱⁱⁱ —K1—O5—K1 ^{iv}	56.75 (3)
K1 ⁱ —O1—C1—O2	92.22 (16)	O5 ^{iv} —K1—O5—K1 ^{iv}	0.0
K1—O1—C1—C2	87.78 (16)	O4 ^v —K1—O5—K1 ^{iv}	-157.09 (5)
K1 ⁱ —O1—C1—C2	-87.78 (16)	O4 ^{vi} —K1—O5—K1 ^{iv}	87.77 (8)
K1 ^{vii} —O3—C2—N1	106.90 (14)	K1 ⁱ —K1—O5—K1 ^{iv}	180.0
K1 ^{viii} —O3—C2—N1	-106.90 (14)		

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

<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>
N1—H1...O4 ^v	0.86	2.10	2.936 (2)	164
N2—H2A...O1 ^{ix}	0.86	2.17	2.997 (2)	163
N2—H2A...O2 ^{ix}	0.86	2.37	3.069 (2)	139
N2—H2B...O3	0.86	2.01	2.667 (2)	133
N2—H2B...O5 ^{vii}	0.86	2.38	3.076 (3)	138
O5—H1W...O2 ⁱⁱ	0.83	1.97	2.791 (2)	172
O5—H1W...O3 ⁱⁱ	0.83	2.59	3.068 (2)	118

supplementary materials

O5—H2W···O2^x

0.83

2.14

2.973 (2)

178

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

Fig. 1

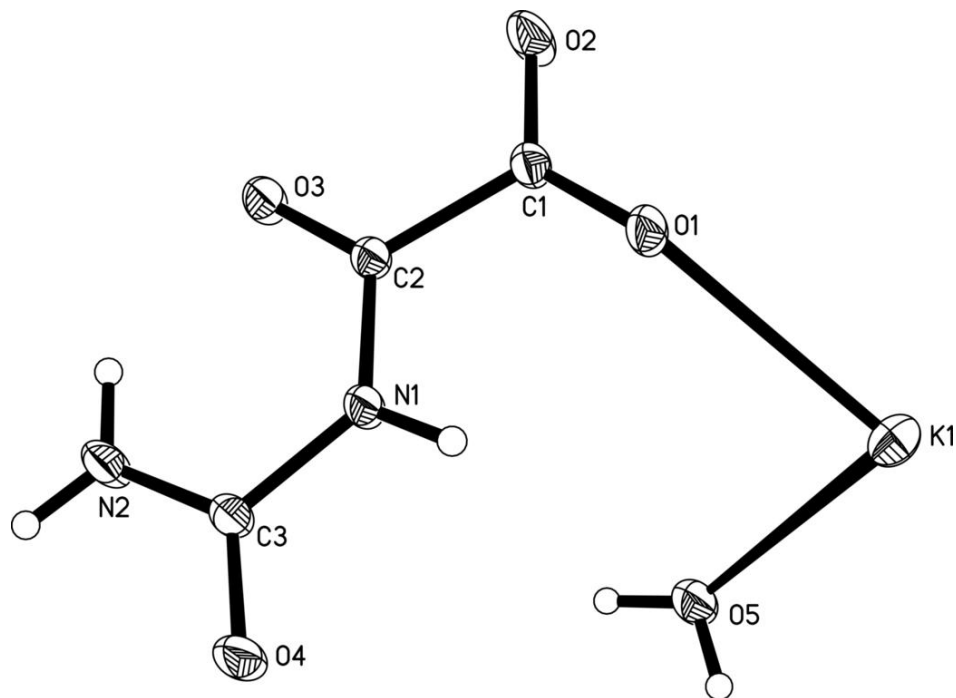


Fig. 2

