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Potassium 2-(*N*-hydroxycarbamoyl)-acetate monohydrateElena V. Prisyazhnaya,^a Irina Odarich,^b Igor O. Fritsky,^c Elżbieta Gumienna-Kontecka^d and Turganbay S. Iskenderov^{e*}

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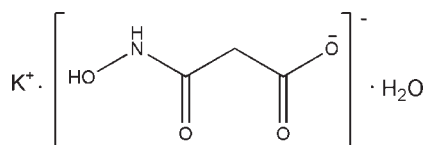
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.025; wR factor = 0.064; data-to-parameter ratio = 14.0.

The crystal structure of the title compound, $\text{K}^+ \cdot \text{C}_3\text{H}_4\text{NO}_4^- \cdot \text{H}_2\text{O}$, consists of potassium cations, monoanions of 2-carboxy-acetohydroxamic acid [namely 2-(*N*-hydroxycarbamoyl)-acetate] and solvent water molecules. The elements of the structure are united in a three-dimensional network by numerous $\text{K} \cdots \text{O}$ coordinate bonds and $\text{O}-\text{H} \cdots \text{O}$ and $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds. The coordination sphere of the K^+ ions may be described as a distorted double capped octahedron. Bond lengths and angles are similar to those in related compounds.

Related literature

For background to hydroxamic acids in biological and coordination chemistry, see: Kaczka *et al.* (1962); Hershko *et al.* (1992); Ghio *et al.* (1992); Shao *et al.* (2004). For hydroxamic acids as versatile bridging ligands, see: Bodwin *et al.* (2001); Cutland-Van Noord *et al.* (2002). For related structures, see: Golenya *et al.* (2007); Gumienna-Kontecka *et al.* (2007); Wörl *et al.* (2005). For $\text{K}-\text{O}$ bond lengths, see: Świątek-Kozłowska *et al.* (2000); Mokhir *et al.* (2002).



Experimental

Crystal data

$\text{K}^+ \cdot \text{C}_3\text{H}_4\text{NO}_4^- \cdot \text{H}_2\text{O}$
 $M_r = 175.19$
 Monoclinic, $P2_1/c$
 $a = 7.457$ (1) Å
 $b = 13.002$ (3) Å
 $c = 6.816$ (1) Å
 $\beta = 105.41$ (3)°

$V = 637.1$ (2) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.80$ mm⁻¹
 $T = 100$ K
 $0.25 \times 0.20 \times 0.12$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 2001)
 $T_{\min} = 0.829$, $T_{\max} = 0.914$

3974 measured reflections
 1498 independent reflections
 1398 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.064$
 $S = 1.10$
 1498 reflections
 107 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.36$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.43$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O4}-\text{H4O} \cdots \text{O1}^{\text{i}}$	0.89 (2)	1.79 (2)	2.6820 (13)	177 (2)
$\text{N1}-\text{H1N} \cdots \text{O2}^{\text{ii}}$	0.79 (2)	2.12 (2)	2.9025 (16)	166.7 (18)
$\text{O1W}-\text{H1W} \cdots \text{O2}^{\text{iii}}$	0.82 (2)	1.97 (2)	2.7811 (15)	171 (2)
$\text{O1W}-\text{H2W} \cdots \text{O2}^{\text{iv}}$	0.84 (3)	1.97 (3)	2.8046 (14)	175 (2)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2123).

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supplementary materials

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Potassium 2-(*N*-hydroxycarbamoyl)acetate monohydrate

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Comment

Hydroxamic acids represent an important class of chelating agents and enzyme inhibitors (Kaczka *et al.*, 1962; Hershko *et al.*, 1992; Ghio *et al.*, 1992; Shao *et al.*, 2004). In recent years hydroxamic acids have also been widely used in coordination chemistry as versatile bridging ligands able to produce multinuclear compounds containing a large number of metal ions, such as metallacrowns (Bodwin *et al.*, 2001; Cutland-Van Noord *et al.*, 2002). Recently we reported that 2-carboxyacetohydroxamic acid is an efficient ligand for obtaining 12-metallacrown-4 complexes with copper(II) ions which can be used as pentanuclear building blocks for preparation of one-dimensional coordination polymers (Gumienna-Kontecka *et al.*, 2007). The present investigation is aimed at the study of the molecular structure of the title compound (**I**) which is a suitable ligand for preparation of polynuclear complexes and coordination polymers.

The atom-numbering scheme of compound (**I**) is shown in Fig. 1. The crystal structure of (**I**) is ionic and consists of potassium cations, monoanions of 2-carboxyacetohydroxamic acid and solvate water molecules. The elements of the structure are united in three-dimensional-network by numerous K \cdots O coordination bonds and the O—H \cdots O and N—H \cdots O hydrogen bonds (Fig. 2, Table 1).

The residue of 2-carboxyacetohydroxamic acid is a monoanion bearing the deprotonated carboxylic group with the hydroxamic function remaining protonated. The anion exhibits C—O, N—O, C—N bond lengths which are typical for carboxylic and hydroxamic groups (Wörl *et al.*, 2005, Golenya *et al.*, 2007). The conformation of monoanion of 2-carboxyacetohydroxamic acid is significantly non-planar due to the presence of the flexible C—CH₂—C moiety uniting two planar hydroxamic and carboxylic fragments. The mentioned groups are disposed nearly perpendicularly; the dihedral angle between their planes is equal to 86.37 (5) $^{\circ}$.

The potassium cation exhibits coordination number 8, and its coordination polyhedron can be considered as severely distorted double capped octahedron. Its coordination environment is formed by two solvate water molecules and six oxygen atoms of monoanion of 2-carboxyacetohydroxamic acid belonging to the deprotonated carboxylic groups (O(1)) and both oxygen atoms of the hydroxamic functions (O(3) and O(4)) belonging to the different translational anions. Each potassium cation has in its coordination sphere the oxygen atoms belonging to five different translational monoanions of 2-carboxyacetohydroxamic acid. The K—O bond lengths lie in the range 2.711 (1) - 3.058 (1) Å which is normal for potassium cations (Świątek-Kozłowska *et al.*, 2000; Mokhir *et al.*, 2002).

Experimental

I was obtained as white powder precipitate by addition of 1 equiv. of KOH (1 M aqueous solution) to warm solution of 2-carboxyacetohydroxamic acid (1.19 g, 10 mmol) in water (40 ml) with consequent reduction in volume of the obtained solution. Single crystals suitable for X-ray analysis were grown by slow isothermal evaporation of aqueous solution at room

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temperature. Anal. For $C_3H_6NO_5K$ (175.18) calcd.: C - 20.57, H - 3.45, N - 8.00. Found: C - 20.7, H - 3.5, N - 7.8. - IR (cm^{-1}): 1062 ($\nu(N-O)$), 1380 ($\nu_s(COO^-)$), 1580 ($\nu_{as}(COO^-)$), 1672 ($\nu(C=O)$ Amide I).

Refinement

The O—H and N—H H atoms were located from the difference Fourier map and refined isotropically. The methylene H atoms were positioned geometrically and were constrained to ride on their parent atoms, with C—H = 0.975–0.98 Å, and $U_{iso} = 1.2 U_{eq}(\text{parent atom})$.

Figures

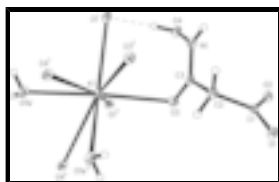


Fig. 1. A view of compound (I), with displacement ellipsoids shown at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. The hydrogen bonding is shown by dashed lines [symmetry codes: (i) $1 + x, y, z$; (ii) $x, 1.5 - y, -1/2 + z$; (iii) $1 + x, 1.5 - y, 1/2 + z$; (iv) $1 - x, 1 - y, -z$; (v) $1 - x, -1/2 + y, 1/2 - z$].

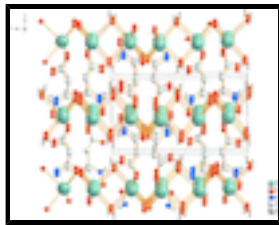


Fig. 2. A packing diagram of the title compound. Hydrogen bonds are indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

Potassium 2-(N-hydroxycarbamoyl)acetate monohydrate

Crystal data



$$M_r = 175.19$$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$$a = 7.4570 (10) \text{ \AA}$$

$$b = 13.002 (3) \text{ \AA}$$

$$c = 6.8160 (10) \text{ \AA}$$

$$\beta = 105.41 (3)^\circ$$

$$V = 637.1 (2) \text{ \AA}^3$$

$$Z = 4$$

$$F_{000} = 360$$

$$D_x = 1.826 \text{ Mg m}^{-3}$$

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

Cell parameters from 567 reflections

$$\theta = 3.5\text{--}27.5^\circ$$

$$\mu = 0.80 \text{ mm}^{-1}$$

$$T = 100 \text{ K}$$

Needle, colourless

$$0.25 \times 0.20 \times 0.12 \text{ mm}$$

Data collection

Bruker SMART CCD area-detector diffractometer

1498 independent reflections

Radiation source: fine-focus sealed tube

1398 reflections with $I > 2\sigma(I)$

Monochromator: graphite

$$R_{int} = 0.025$$

$$T = 100 \text{ K}$$

$$\theta_{max} = 28.4^\circ$$

ω scans $\theta_{\min} = 3.5^\circ$
 Absorption correction: multi-scan $h = -9 \rightarrow 9$
 (SADABS; Sheldrick, 2001)
 $T_{\min} = 0.829$, $T_{\max} = 0.914$ $k = -17 \rightarrow 17$
 3974 measured reflections $l = -6 \rightarrow 9$

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
 Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites
 $R[F^2 > 2\sigma(F^2)] = 0.025$ H atoms treated by a mixture of independent and constrained refinement
 $wR(F^2) = 0.064$ $w = 1/[\sigma^2(F_o^2) + (0.0348P)^2 + 0.2753P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.10$ $(\Delta/\sigma)_{\max} < 0.001$
 1498 reflections $\Delta\rho_{\max} = 0.36 \text{ e } \text{\AA}^{-3}$
 107 parameters $\Delta\rho_{\min} = -0.43 \text{ e } \text{\AA}^{-3}$
 Primary atom site location: structure-invariant direct methods Extinction correction: none

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
K1	0.50362 (4)	0.64050 (2)	0.09788 (4)	0.01167 (10)
O1	-0.30644 (12)	0.82467 (7)	0.01504 (14)	0.0127 (2)
O2	-0.09437 (12)	0.92917 (7)	0.20776 (13)	0.01173 (19)
O3	0.27158 (12)	0.76799 (7)	0.24610 (13)	0.01273 (19)
O4	0.46616 (12)	0.93401 (7)	0.18090 (13)	0.0122 (2)
O1W	0.77186 (13)	0.51272 (8)	0.03917 (15)	0.0155 (2)
N1	0.28881 (15)	0.90970 (9)	0.06042 (16)	0.0110 (2)
C1	-0.14337 (17)	0.85843 (9)	0.07756 (18)	0.0091 (2)
C2	0.00201 (17)	0.81466 (10)	-0.02077 (18)	0.0112 (2)
H2A	-0.0220	0.7419	-0.0464	0.013*
H2B	-0.0110	0.8482	-0.1510	0.013*
C3	0.19948 (16)	0.82835 (10)	0.10799 (18)	0.0096 (2)

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H4O	0.543 (3)	0.8999 (18)	0.124 (3)	0.031 (5)*
H1N	0.243 (3)	0.9497 (15)	-0.027 (3)	0.024 (5)*
H1W	0.863 (3)	0.4904 (18)	0.125 (3)	0.036 (6)*
H2W	0.807 (3)	0.5327 (18)	-0.062 (4)	0.043 (6)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.01153 (15)	0.01230 (16)	0.01062 (15)	0.00162 (9)	0.00197 (10)	-0.00076 (9)
O1	0.0095 (4)	0.0153 (4)	0.0134 (4)	-0.0009 (3)	0.0029 (3)	-0.0024 (3)
O2	0.0112 (4)	0.0130 (4)	0.0108 (4)	0.0004 (3)	0.0024 (3)	-0.0031 (3)
O3	0.0131 (4)	0.0140 (4)	0.0112 (4)	0.0013 (3)	0.0034 (3)	0.0021 (3)
O4	0.0078 (4)	0.0165 (5)	0.0118 (4)	-0.0021 (3)	0.0020 (3)	-0.0035 (3)
O1W	0.0120 (4)	0.0218 (5)	0.0130 (5)	0.0038 (4)	0.0041 (4)	0.0064 (4)
N1	0.0096 (5)	0.0121 (5)	0.0097 (5)	0.0001 (4)	-0.0002 (4)	0.0006 (4)
C1	0.0103 (5)	0.0096 (6)	0.0070 (5)	0.0016 (4)	0.0018 (4)	0.0026 (4)
C2	0.0104 (5)	0.0133 (6)	0.0099 (5)	-0.0001 (4)	0.0031 (4)	-0.0029 (4)
C3	0.0096 (5)	0.0113 (6)	0.0092 (5)	0.0009 (4)	0.0047 (4)	-0.0028 (4)

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

K1—O1W	2.7105 (11)	O4—N1	1.3951 (14)
K1—O3	2.7734 (10)	O4—H4O	0.89 (2)
K1—O3 ⁱ	2.8202 (12)	O1W—H1W	0.82 (2)
K1—O1W ⁱⁱ	2.8358 (12)	O1W—H2W	0.84 (3)
K1—O1 ⁱⁱⁱ	2.8558 (12)	N1—C3	1.3351 (17)
K1—O1 ^{iv}	2.9128 (11)	N1—H1N	0.79 (2)
K1—O4 ⁱ	2.9448 (10)	C1—C2	1.5279 (17)
K1—O4 ^v	3.0580 (11)	C2—C3	1.5111 (17)
O1—C1	1.2560 (15)	C2—H2A	0.9700
O2—C1	1.2628 (15)	C2—H2B	0.9700
O3—C3	1.2337 (16)		
O1W—K1—O3	167.55 (3)	O3 ⁱ —K1—O4 ^v	137.54 (3)
O1W—K1—O3 ⁱ	116.31 (3)	O1W ⁱⁱ —K1—O4 ^v	59.85 (3)
O3—K1—O3 ⁱ	75.90 (2)	O1 ⁱⁱⁱ —K1—O4 ^v	72.32 (3)
O1W—K1—O1W ⁱⁱ	91.10 (3)	O1 ^{iv} —K1—O4 ^v	147.29 (3)
O3—K1—O1W ⁱⁱ	94.15 (3)	O4 ⁱ —K1—O4 ^v	99.36 (3)
O3 ⁱ —K1—O1W ⁱⁱ	77.83 (4)	N1—O4—H4O	104.5 (13)
O1W—K1—O1 ⁱⁱⁱ	93.09 (4)	H1W—O1W—H2W	108 (2)
O3—K1—O1 ⁱⁱⁱ	74.63 (3)	C3—N1—O4	119.52 (10)
O3 ⁱ —K1—O1 ⁱⁱⁱ	144.05 (3)	C3—N1—H1N	124.0 (14)
O1W ⁱⁱ —K1—O1 ⁱⁱⁱ	124.30 (3)	O4—N1—H1N	116.1 (14)
O1W—K1—O1 ^{iv}	93.40 (3)	O1—C1—O2	124.49 (11)
O3—K1—O1 ^{iv}	87.81 (3)	O1—C1—C2	117.18 (11)
O3 ⁱ —K1—O1 ^{iv}	73.05 (3)	O2—C1—C2	118.23 (11)

O1W ⁱⁱ —K1—O1 ^{iv}	149.40 (3)	C3—C2—C1	113.38 (10)
O1 ⁱⁱⁱ —K1—O1 ^{iv}	85.67 (3)	C3—C2—H2A	108.9
O1W—K1—O4 ⁱ	62.64 (4)	C1—C2—H2A	108.9
O3—K1—O4 ⁱ	129.78 (3)	C3—C2—H2B	108.9
O3 ⁱ —K1—O4 ⁱ	55.86 (3)	C1—C2—H2B	108.9
O1W ⁱⁱ —K1—O4 ⁱ	64.93 (3)	H2A—C2—H2B	107.7
O1 ⁱⁱⁱ —K1—O4 ⁱ	155.21 (3)	O3—C3—N1	123.01 (11)
O1 ^{iv} —K1—O4 ⁱ	90.56 (3)	O3—C3—C2	121.88 (11)
O1W—K1—O4 ^v	64.79 (3)	N1—C3—C2	115.11 (11)
O3—K1—O4 ^v	108.45 (3)		
O1—C1—C2—C3	-159.16 (11)	O4—N1—C3—C2	174.68 (10)
O2—C1—C2—C3	24.31 (15)	C1—C2—C3—O3	83.28 (14)
O4—N1—C3—O3	-5.58 (18)	C1—C2—C3—N1	-96.98 (13)

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O4—H4O \cdots O1 ^{iv}	0.89 (2)	1.79 (2)	2.6820 (13)	177 (2)
N1—H1N \cdots O2 ^{vi}	0.79 (2)	2.12 (2)	2.9025 (16)	166.7 (18)
O1W—H1W \cdots O2 ^v	0.82 (2)	1.97 (2)	2.7811 (15)	171 (2)
O1W—H2W \cdots O2 ^{vii}	0.84 (3)	1.97 (3)	2.8046 (14)	175 (2)

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

Fig. 1

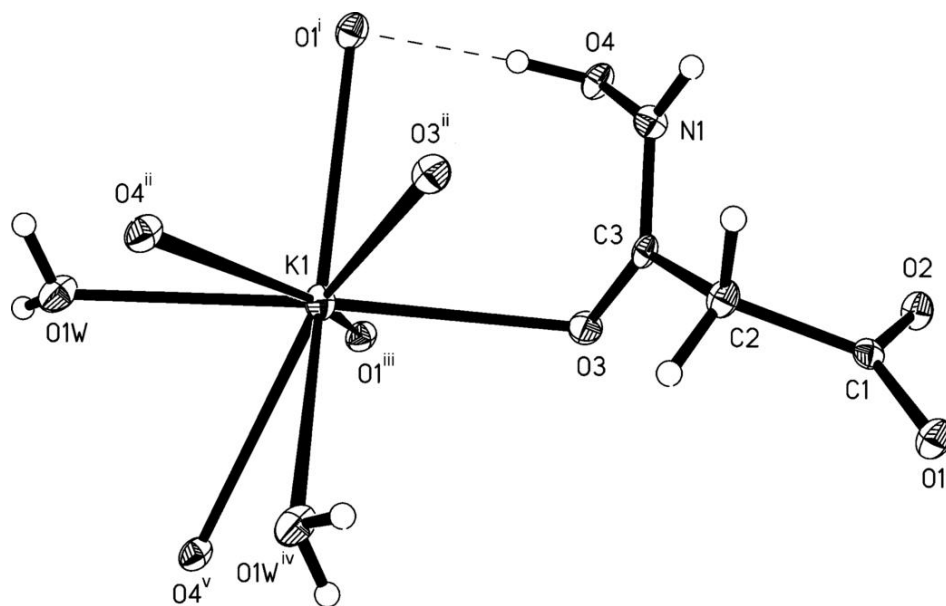


Fig. 2

