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Allylammonium hydrogen oxalate hemihydrate

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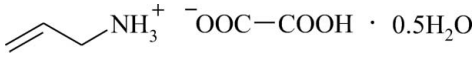
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.043; wR factor = 0.098; data-to-parameter ratio = 10.1.

In the title hydrated molecular salt, $C_3H_8N^+\cdot C_2HO_4^-\cdot 0.5H_2O$, the water O atom lies on a crystallographic twofold axis. The C=C—C—N torsion angle in the cation is 2.8 (3)° and the dihedral angle between the CO_2 and CO_2H planes in the anion is 1.0 (4)°. In the crystal, the hydrogen oxalate ions are linked by O—H···O hydrogen bonds, generating [010] chains. The allylammonium cations bond to the chains through N—H···O and N—H···(O,O) hydrogen bonds. The water molecule accepts two N—H···O hydrogen bonds and makes two O—H···O hydrogen bonds. Together, the hydrogen bonds generate (100) sheets.

Related literature

For the crystal structures of oxalic acid salts with aliphatic amines, see: Ejsmont (2006), (2007); Ejsmont & Zaleski (2006*a*,*b*); Vaidhyanathan *et al.* (2001, 2002); MacDonald *et al.* (2001) For information on the Cambridge Database, see: Allen (2002).



Experimental

Crystal data

$C_3H_8N^+\cdot C_2HO_4^-\cdot 0.5H_2O$
M_r = 156.14
Monoclinic, $C2/c$
a = 21.578 (3) Å
b = 5.6521 (4) Å
c = 13.8629 (17) Å
β = 118.415 (17)°
V = 1487.0 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 100 K
0.33 × 0.18 × 0.14 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
4525 measured reflections
1376 independent reflections
958 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.098
S = 0.90
1376 reflections
136 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*B*···O9	0.98 (3)	1.86 (3)	2.825 (2)	169 (2)
N1—H1*A*···O11	0.98 (3)	1.82 (3)	2.769 (2)	161 (2)
N1—H1*C*···O8^{i}	0.91 (3)	2.19 (3)	3.014 (2)	151 (2)
N1—H1*C*···O10^{i}	0.91 (3)	2.16 (3)	2.853 (2)	132 (2)
O7—H7···O10^{ii}	0.94 (3)	1.62 (3)	2.5563 (19)	179 (4)
O11—H11···O9^{iii}	0.88 (3)	1.86 (3)	2.739 (2)	176 (3)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2008); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2008); data reduction: *CrysAlis RED* ; 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*.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7243).

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Allylammonium hydrogen oxalate hemihydrate

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S1. Comment

Oxalic acid, together with its anions, is one of the best building blocks for the construction of supramolecular structures based on hydrogen bonds. The adducts of oxalic acid and aliphatic amines have been examined by single-crystal X-ray diffraction and other techniques. Three types of characteristic structural motifs are present: (i) linear chains of dicarboxylic acids formed by strong hydrogen bonds; (ii) dimers of dicarboxylic acid molecules; (iii) isolated oxalate monoanions or dianion units (MacDonald *et al.*, 2001; Vaidhyanathan *et al.*, 2001, 2002); Ejsmont, 2006, 2007; Ejsmont & Zaleski 2006*a*, 2006*b*).

The crystal structure of the title salt, (I), consists of allyloammonium cations, hydrogen oxalate anions and water molecules (Fig. 1). A search of the Cambridge Structural Database (CSD; CONQUEST Version 1.16; Allen, 2002) afforded that the geometrical parameters of the allyloammonium cation (Table 1) compare well with those found in other crystal structures which include this cation (Allen, 2002). The oxalate monoanions are nearly planar and are connected to each other by strong O—H···O hydrogen bonds along the *b* axis. The allyloammonium cations form N—H···O H atoms bonds to the anions and water molecules (Fig. 2 and Table 2).

S2. Experimental

Colourless prisms of (I) were grown at room temperature by slow evaporation of an aqueous solution of allylamine and oxalic acid in a 1:1 stoichiometric ratio.

S3. Refinement

All H atoms were positioned geometrically and their parameters are refined independently.

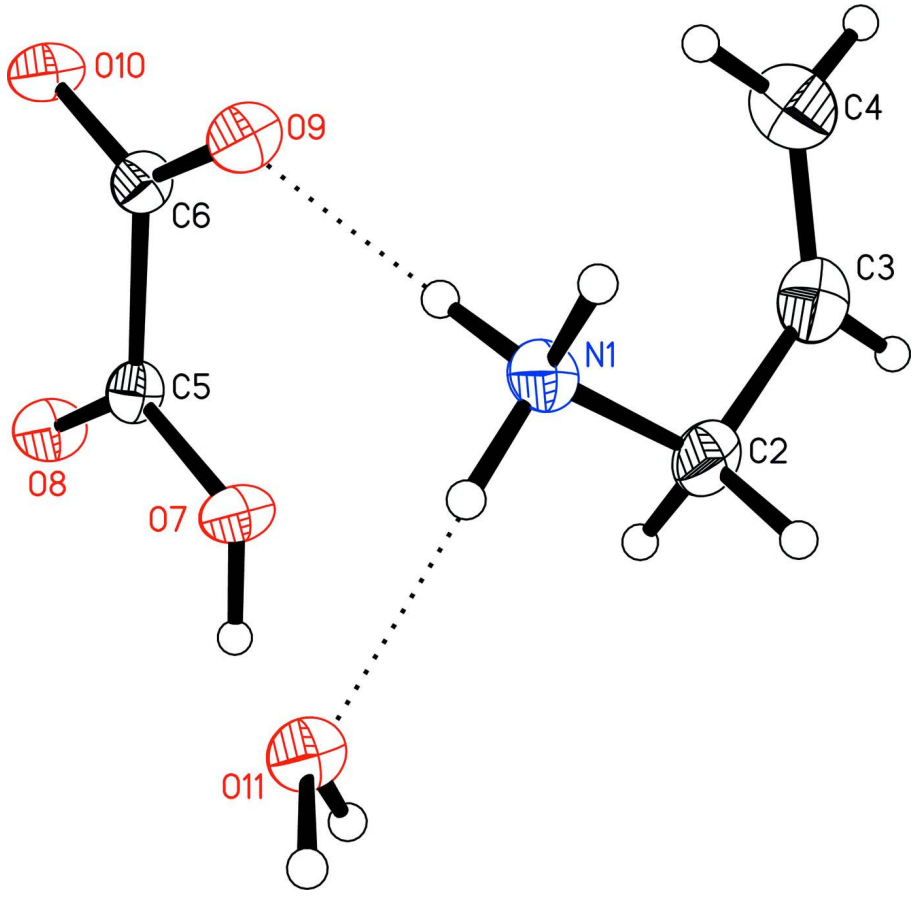

Figure 1

The molecular structure of (I), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). Hydrogen bonds are shown as dotted lines.

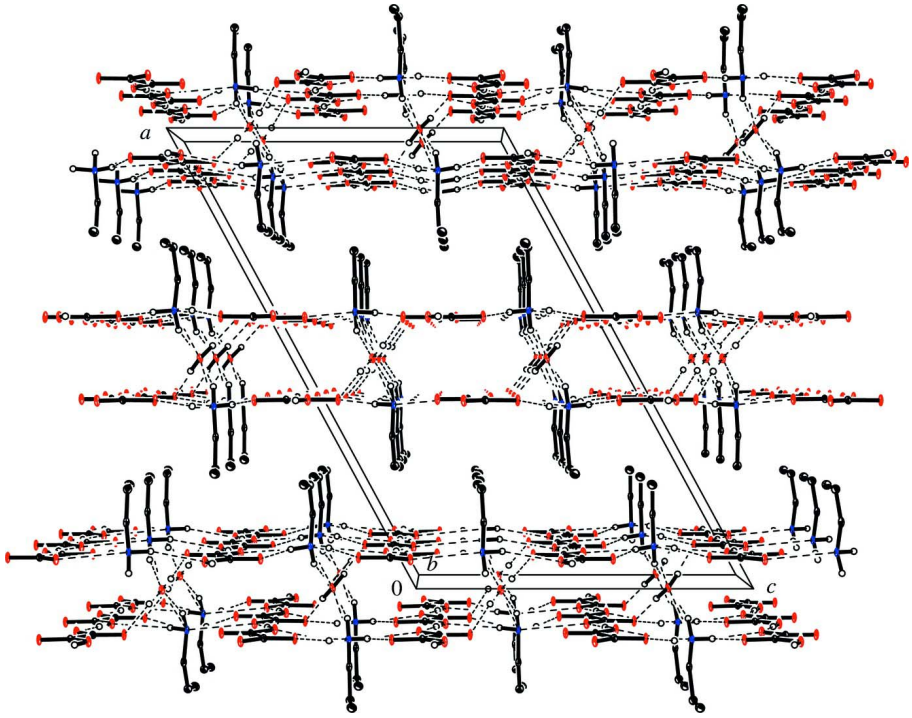

Figure 2

The packing diagram of (I), viewed along the *b* axis, showing the intermolecular hydrogen-bonding scheme (dashed lines).

Allylammonium hydrogen oxalate hemihydrate

Crystal data

$C_3H_8N^+·C_2HO_4^-·0.5H_2O$
M_r = 156.14
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 21.578 (3) Å
b = 5.6521 (4) Å
c = 13.8629 (17) Å
β = 118.415 (17)°
V = 1487.0 (3) Å^3
Z = 8
F(000) = 664
D_x = 1.395 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 4525 reflections
θ = 3.3–25.5°
μ = 0.12 mm^{-1}
T = 100 K
Prism, colourless
0.33 × 0.18 × 0.14 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 1024 x 1024 with blocks 2 x 2 pixels mm^{-1}
ω–scan
4525 measured reflections
1376 independent reflections
958 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 25.5°, θ_{min} = 3.3°
h = −26→26
k = −6→5
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.098$
$S = 0.90$
1376 reflections
136 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.
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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.40092 (10)	0.2394 (3)	0.23240 (15)	0.0196 (4)
H1A	0.4411 (13)	0.348 (4)	0.2556 (19)	0.033 (6)*
H1B	0.4018 (14)	0.132 (5)	0.177 (2)	0.050 (8)*
H1C	0.4052 (14)	0.154 (5)	0.291 (2)	0.046 (8)*
C2	0.33560 (12)	0.3802 (4)	0.18227 (19)	0.0231 (5)
H2A	0.3367 (11)	0.466 (4)	0.1271 (18)	0.021 (5)*
H2B	0.3376 (12)	0.486 (4)	0.233 (2)	0.032 (6)*
C3	0.27053 (13)	0.2364 (4)	0.13940 (19)	0.0284 (5)
H3	0.2311 (12)	0.325 (4)	0.1035 (18)	0.026 (6)*
C4	0.26561 (15)	0.0080 (5)	0.1452 (2)	0.0327 (6)
H4A	0.2203 (14)	−0.075 (4)	0.111 (2)	0.042 (7)*
H4B	0.3053 (13)	−0.085 (4)	0.1769 (19)	0.028 (6)*
C5	0.41682 (10)	0.1591 (3)	−0.03835 (16)	0.0167 (5)
C6	0.41572 (10)	−0.0892 (3)	0.00740 (16)	0.0177 (5)
O7	0.41788 (8)	0.3311 (2)	0.02573 (11)	0.0211 (4)
H7	0.4173 (16)	0.482 (6)	−0.002 (3)	0.077 (10)*
O8	0.41599 (8)	0.1838 (2)	−0.12581 (11)	0.0228 (4)
O9	0.41549 (8)	−0.1043 (2)	0.09658 (11)	0.0231 (4)
O10	0.41539 (8)	−0.2570 (2)	−0.05214 (11)	0.0221 (4)
O11	0.5000	0.5720 (4)	0.2500	0.0199 (5)
H11	0.5269 (14)	0.672 (5)	0.302 (2)	0.055 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0289 (11)	0.0222 (10)	0.0119 (8)	−0.0017 (8)	0.0133 (8)	0.0000 (8)
C2	0.0356 (13)	0.0234 (12)	0.0166 (11)	0.0039 (10)	0.0175 (10)	0.0007 (10)
C3	0.0273 (14)	0.0331 (14)	0.0255 (12)	0.0051 (11)	0.0132 (11)	0.0020 (10)
C4	0.0290 (14)	0.0362 (15)	0.0294 (13)	−0.0024 (12)	0.0110 (11)	0.0021 (12)
C5	0.0189 (11)	0.0201 (10)	0.0113 (10)	−0.0001 (8)	0.0073 (8)	−0.0027 (8)
C6	0.0202 (11)	0.0194 (10)	0.0136 (10)	0.0009 (8)	0.0082 (9)	−0.0002 (8)
O7	0.0383 (9)	0.0158 (7)	0.0148 (7)	−0.0003 (7)	0.0172 (7)	0.0003 (6)
O8	0.0394 (9)	0.0224 (8)	0.0128 (7)	−0.0011 (6)	0.0173 (7)	0.0005 (6)
O9	0.0416 (9)	0.0222 (8)	0.0145 (8)	0.0049 (6)	0.0206 (7)	0.0037 (6)
O10	0.0395 (9)	0.0172 (7)	0.0148 (7)	−0.0003 (6)	0.0170 (7)	−0.0014 (6)
O11	0.0283 (12)	0.0207 (11)	0.0119 (10)	0.000	0.0107 (9)	0.000

Geometric parameters (Å, º)

N1—C2	1.473 (3)	C4—H4A	0.98 (3)
N1—H1A	0.98 (3)	C4—H4B	0.92 (2)
N1—H1B	0.98 (3)	C5—O8	1.212 (2)
N1—H1C	0.91 (3)	C5—O7	1.309 (2)
C2—C3	1.480 (3)	C5—C6	1.545 (3)
C2—H2A	0.92 (2)	C6—O9	1.242 (2)
C2—H2B	0.91 (2)	C6—O10	1.255 (2)
C3—C4	1.301 (3)	O7—H7	0.94 (3)
C3—H3	0.91 (2)	O11—H11	0.88 (3)
C2—N1—H1A	108.3 (13)	C4—C3—H3	120.1 (14)
C2—N1—H1B	109.5 (15)	C2—C3—H3	112.3 (14)
H1A—N1—H1B	108 (2)	C3—C4—H4A	122.4 (14)
C2—N1—H1C	112.2 (17)	C3—C4—H4B	120.8 (14)
H1A—N1—H1C	110 (2)	H4A—C4—H4B	116.6 (19)
H1B—N1—H1C	109 (2)	O8—C5—O7	125.49 (18)
N1—C2—C3	113.87 (18)	O8—C5—C6	121.27 (17)
N1—C2—H2A	106.4 (13)	O7—C5—C6	113.24 (15)
C3—C2—H2A	110.6 (13)	O9—C6—O10	126.98 (18)
N1—C2—H2B	108.0 (14)	O9—C6—C5	118.63 (16)
C3—C2—H2B	111.1 (15)	O10—C6—C5	114.39 (16)
H2A—C2—H2B	106.5 (19)	C5—O7—H7	113.9 (19)
C4—C3—C2	127.6 (2)		
N1—C2—C3—C4	2.8 (3)	O8—C5—C6—O10	1.4 (3)
O8—C5—C6—O9	−178.8 (2)	O7—C5—C6—O10	−179.34 (17)
O7—C5—C6—O9	0.4 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···O9	0.98 (3)	1.86 (3)	2.825 (2)	169 (2)
N1—H1*A*···O11	0.98 (3)	1.82 (3)	2.769 (2)	161 (2)
N1—H1*C*···O8^{i}	0.91 (3)	2.19 (3)	3.014 (2)	151 (2)
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O11—H11···O9^{iii}	0.88 (3)	1.86 (3)	2.739 (2)	176 (3)

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