organic papers

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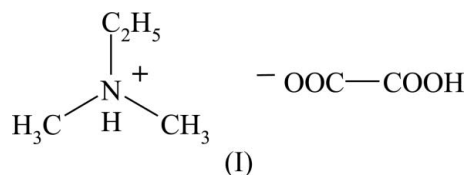# Ethyldimethylammonium oxalate

The title crystal structure, $C_4H_{12}N^+ \cdot C_2HO_4^-$, contains discrete ethyldimethylammonium cations and oxalate monoanions. Linear chains of oxalate monoanions are formed by strong O—H···O hydrogen bonds. These chains are further connected by N—H···O and C—H···O hydrogen bonds through ethyldimethylammonium cations, forming channels along the a axis.

Comment

Supramolecular hydrogen–bonded assemblies of oxalic acid and organic amines (alkylammonium, guanidine, 1,4-diaza-bicyclo[2.2.2]octane, *etc.*) have been examined by single-crystal X-ray diffraction and other techniques (MacDonald *et al.*, 2001; Vaidhyanathan *et al.*, 2001, 2002; Ejsmont & Zaleski, 2006*a*,b).

The asymmetric unit of the title compound, (I), consists of one ethyldimethylammonium cation and one oxalate mono-anion (Fig. 1). The geometrical parameters of the ethyl-dimethylammonium cation (Table 1) compare well with those
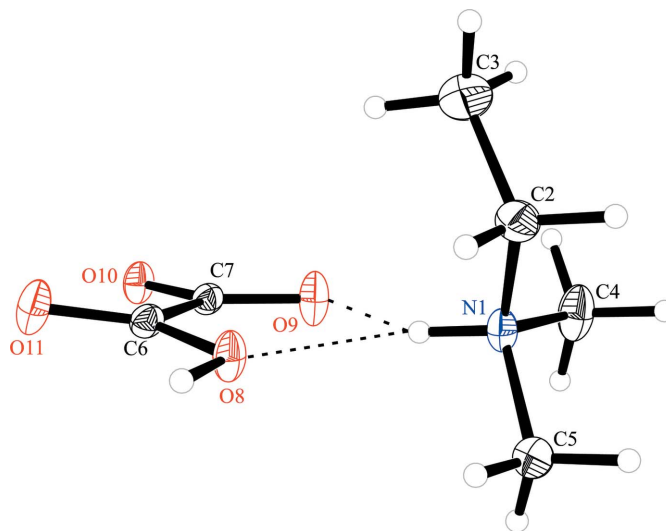

Figure 1
The asymetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines.
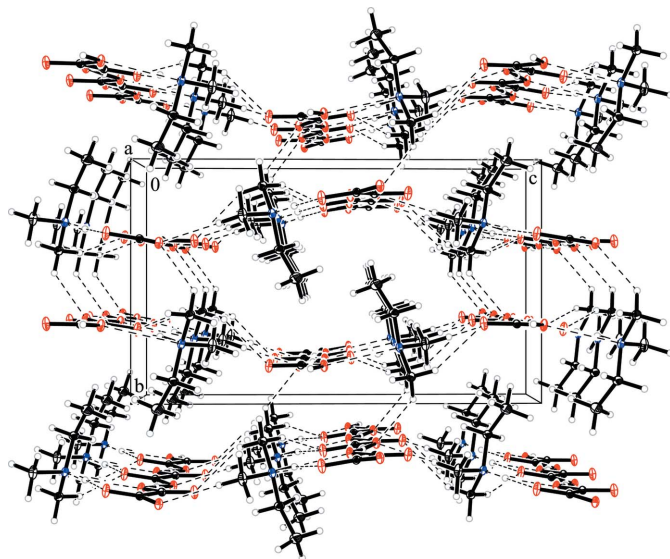
Figure 2
A packing plot of (I), showing the intermolecular hydrogen-bonding scheme (dashed lines).

found in other crystal structures which include this cation (Bujak & Zaleski, 1999; Scheibitz *et al.*, 2003; Molteni *et al.*, 2004). The oxalate monoanions are essentially planar, with dihedral angles between the carboxylate groups of less than $10°$. A strong O8—H8···O10[i] (see Table 2 for symmetry code) hydrogen bond generates linear oxalate chains running parallel to the *a* axis. The geometrical parameters of this chain correlate well with the corresponding values found in related crystal structures (Vaidhyanathan *et al.*, 2002; Ejsmont & Zaleski, 2006b). In addtion, the crystal structure of (I) is stabilized by ionic interactions between the ethyldimethyl-ammonium cations and the oxalate monoanions chains, as well as by a network of N—H···O and C—H···O hydrogen bonds (Fig. 2 and Table 2). In the crystal structure of (I), there are channels running along the *a* axis. The dimensions of these channels, measured by the distance between methyl H atoms of the ethyldimethylammonium cation and between the mean planes of the oxalate monoanions chains, are 5.073 (5) and 3.267 (4) Å, respectively.

Experimental

Crystals of (I) were grown at room temperature by slow evaporation of an aqueous solution containing ethyldimethylamine and oxalic acid in a 2:1 stoichiometric ratio.

Crystal data

$C_4H_{12}N^+ \cdot C_2HO_4^-$
$M_r = 163.17$
Monoclinic, $P2_1/n$
$a = 5.5953 (7)$ Å
$b = 9.3979 (9)$ Å
$c = 15.804 (2)$ Å
$\beta = 92.158 (9)°$
$V = 830.45 (17)$ Å^3

$Z = 4$
$D_x = 1.305$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 100.0 (1)$ K
Block, colourless
$0.42 \times 0.40 \times 0.38$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer
ω scans
Absorption correction: none
4871 measured reflections

1545 independent reflections
1185 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 25.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.082$
$S = 0.98$
1545 reflections
152 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0778P)^2 + 0.1154P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C4	1.485 (2)	C6—O8	1.306 (1)
N1—C5	1.490 (2)	C6—C7	1.552 (2)
N1—C2	1.499 (2)	C7—O9	1.237 (2)
C6—O11	1.212 (2)	C7—O10	1.266 (2)
O11—C6—O8	125.9 (1)	O9—C7—O10	126.7 (1)
O11—C6—C7	121.7 (1)	O9—C7—C6	118.6 (1)
O8—C6—C7	112.4 (1)	O10—C7—C6	114.7 (1)
O11—C6—C7—O9	170.4 (1)	O11—C6—C7—O10	−8.4 (2)
O8—C6—C7—O9	−8.8 (2)	O8—C6—C7—O10	172.4 (1)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O8	0.92 (2)	2.19 (2)	2.931 (1)	136 (1)
N1—H1···O9	0.92 (2)	1.98 (2)	2.781 (1)	144 (1)
O8—H8···O10[ii]	1.12 (2)	1.35 (2)	2.473 (1)	177 (2)
C4—H4B···O10[ii]	1.00 (2)	2.30 (2)	3.264 (2)	161 (1)
C5—H5A···O10[iii]	0.99 (2)	2.40 (2)	3.213 (2)	140 (1)
C5—H5B···O9[i]	0.98 (2)	2.49 (2)	3.421 (2)	160 (1)
C5—H5C···O11[ii]	1.00 (1)	2.54 (1)	3.449 (2)	151 (1)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2002); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

References

Bujak, M. & Zaleski, J. (1999). *Acta Cryst.* C**55**, 1775–1778.
Ejsmont, K. & Zaleski, J. (2006a). *Acta Cryst.* E**62**, o2512–o2513.
Ejsmont, K. & Zaleski, J. (2006b). *Acta Cryst.* E**62**, o3879–o3880.
MacDonald, J. C., Doeewstein, C. P. & Pilley, M. M. (2001). *Cryst. Growth Des.* **1**, 29–38.
Molteni, V., Penzotti, J., Wilson, D. M., Termin, A. P., Mao, L., Crane, C. M., Hassman, F., Wang, T., Wong, H., Miller, K. J., Grossman, S. & Grootenhuis, P. D. J. (2004). *J. Med. Chem.* **47**, 2426–2429.
Oxford Diffraction (2002). *CrysAlis CCD* (Version 1.170) and *CrysAlis RED* (Version 1.170.16). Oxford Diffraction, Wrocław, Poland.
Scheibitz, M., Lerner, H.-W. & Bolte, M. (2003). *Acta Cryst.* E**59**, o253–o254.

Sheldrick, G. M. (1990). *SHELXTL*. Siemens Analytical X-ray Instrument Inc., Madison, Winsconsin, USA.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Vaidhyanathan, R., Natarajan, S. & Rao, C. N. R. (2001). *J. Chem. Soc. Dalton Trans.* pp. 699–706.

Vaidhyanathan, R., Natarajan, S. & Rao, C. N. R. (2002). *J. Mol. Struct.* **608**, 123–133.