Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Diethylammonium hydrogen oxalate

Krzysztof Ejsmont

Institute of Chemistry, University of Opole, Oleska 48, 45-052 Opole, Poland

Correspondence e-mail: eismont@uni.opole.pl

Received 30 November 2006
Accepted 30 November 2006

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.116
Data-to-parameter ratio = 15.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of the title compound, $C_4H_{12}N^+\cdot C_2HO_4^-$, consists of discrete oxalate monoanions and diethylammonium cations. The N atom lies on a crystallographic twofold rotation axis and the oxalate ion is centrosymmetric. The oxalate monoanions are present as hydrogen-bonded linear chains. Conformationally extended diethylammonium cations link the linear chains through three–centre N–H···O hydrogen bonds.

Comment

The study of amine oxalates demonstrates the occurrence of different types of supramolecular structures arising from hydrogen bonds (MacDonald *et al.*, 2001; Vaidhyanathan *et al.*, 2001, 2002; Ejsmont & Zaleski, 2006*a*,*b*).

(I)

The crystal structure of the title compound, (I), shows the presence of conformationally extended diethylammonium cations (the values of torsion angles are close to 180°; Table 1) and oxalate monoanions (Fig. 1). The complete cation is generated by a twofold rotation axis, with atom N3 lying on the axis. Its geometry is normal and compares well with those found in other crystal structures which include this cation (Castellari *et al.*, 2001; Bolte *et al.*, 2003; Emsley *et al.*, 1986;

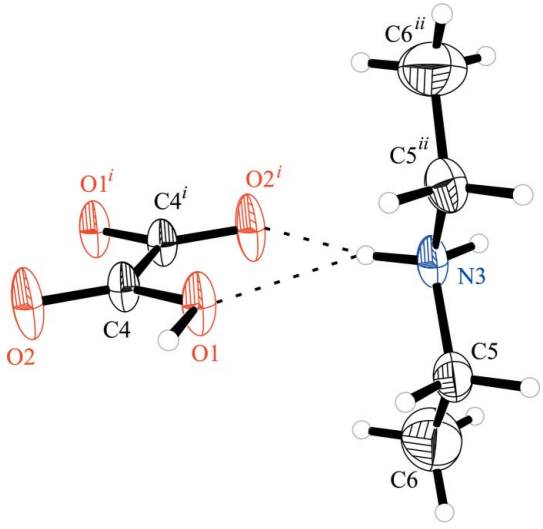

Figure 1
The molecular structure of (I), showing 30% displacement ellipsoids (arbitrary spheres for the H atoms). Dashed lines indicate hydrogen bonds. [Symmetry codes: (i) 1 − *x*, −*y*, 1 − *y*; (ii) 1 − *x*, *y*, 0.5 − *z*.]

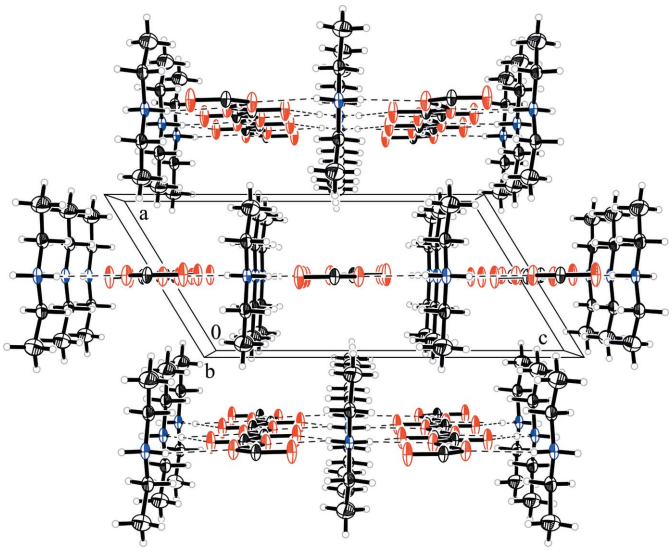

Figure 2
The packing of (I), showing the intermolecular hydrogen-bonding scheme (dashed lines).

Ishida & Kashino, 2000). The centrosymmetric oxalate monoanion in (I) is exactly planar. The difference in the C4—O1 and C4—O2 bond distances (0.053 Å, Table 1) clearly indicates that O1 of the carboxylate group is protonated.

In the crystal structure of (I), there is a linear O—H···O hydrogen bond between the oxalate monoanions, which can be identified as a very strong interaction (Steiner, 2002). The H atom in this interaction is located on a centre of inversion, resulting in equal donor–hydrogen and hydrogen–acceptor distances. This O—H···O hydrogen bond generates linear oxalate chains with an O1···O1^{i} distance of 2.452 (1) Å (Table 2). The O···O distance in this interaction is shorter than that observed for O—H···O hydrogen bonds formed between the monoanionic oxalate units in the structures of amine oxalates (MacDonald *et al.*, 2001; Vaidhyanathan *et al.*, 2001, 2002; Ejsmont & Zaleski, 2006*a*,*b*). As can be seen from the packing diagram of (I), the monohydrogen oxalate linear chains run parallel to the *b* axis, with the diethylammonium cations between the chains (Fig. 2). The anion chains and diethylammonium cations are held together by three-centre N—H···O hydrogen bonds (Table 2).

Experimental

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

Crystal data

$C_4H_{12}N^+\cdot C_2HO_4^-$
M_r = 163.17
Monoclinic, $P2_1/c$
a = 7.0527 (4) Å
b = 5.5465 (2) Å
c = 14.0317 (6) Å
β = 121.544 (4)°
V = 467.78 (4) Å^3
Z = 2
D_x = 1.158 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (1) K
Block, colourless
0.39 × 0.37 × 0.36 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
ω scans
Absorption correction: none
2852 measured reflections
872 independent reflections
744 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 25.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.117
S = 1.09
872 reflections
56 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0778P)^2 + 0.1154P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C4	1.268 (2)	O2—C4	1.215 (2)
C5^{i}—N3—C5—C6	175.9 (2)		

Symmetry code: (i) $-x+1, y, -z+\frac{1}{2}$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O1^{ii}	1.23 (1)	1.23 (1)	2.452 (1)	180
N3—H3···O1	0.91 (2)	2.42 (2)	3.0231 (9)	123 (2)
N3—H3···O2^{iii}	0.91 (2)	2.01 (2)	2.894 (2)	163 (2)

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

H atoms bonded to C were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C)] using a riding model, with C—H = 0.97 Å for methylene or 0.96 Å for methyl H atoms. H atoms bonded to O and N atoms were freely refined; since the H atom bonded to O is located on a crystallographic centre of inversion, its coordinates were not refined.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); 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

Bolte, M., Panisch, R. & Müller, T. (2003). *Acta Cryst.* E**59**, o929–o930.
Castellari, C., Comelli, F. & Ottani, S. (2001). *Acta Cryst.* C**57**, 437–438.
Ejsmont, K. & Zaleski, J. (2006*a*). *Acta Cryst.* E**62**, o2512–o2513.
Ejsmont, K. & Zaleski, J. (2006*b*). *Acta Cryst.* E**62**, o3879–o3880.
Emsley, J., Freeman, N. J., Parker, R. J., Dawes, H. M. & Hursthouse, M. B. (1986). *J. Chem. Soc. Perkin Trans. 1*, pp. 471–473.
Ishida, H. & Kashino, S. (2000). *Acta Cryst.* C**56**, e202–e204.
MacDonald, J. C., Doeewstein, C. P. & Pilley, M. M. (2001). *Cryst. Grow. Des.* **1**, 29–38.
Oxford Diffraction (2002). *CrysAlis CCD*. Version 1.171. Oxford Diffraction, Wrocław, Poland.
Oxford Diffraction (2006). *CrysAlis RED*. Version 1.171.31.5. Oxford Diffraction, Wrocław, Poland.
Sheldrick, G. M. (1990). *SHELXTL*. Siemens Analytical X-ray Instrument Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Steiner, T. (2002). *Angew. Chem. Int. Ed.* **41**, 48–76.
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.