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Bis(*tert*-butylammonium) oxalate

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Key indicators

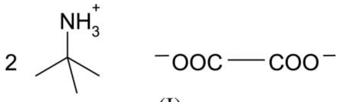
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.035
wR factor = 0.107
Data-to-parameter ratio = 14.6

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

The component species in the title compound, $2C_4H_{12}N_2^+\cdot C_2O_4^{2-}$, interact by way of N—H···O hydrogen bonds, resulting in a layered network. N and two C atoms of the cation possess site symmetry *m* and the C atom of the anion has site symmetry 2.

Comment

The adducts of oxalic acid and various organic amines (e.g. alkylamines, guanidine and 1,4-diazabicyclo[2,2,2]octane) 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 O—H···O hydrogen bonds; (ii) dimers of dicarboxylic acid molecules; (iii) isolated oxalate monoanions or dianion units (MacDonald *et al.*, 2001; Vaidhyanathan *et al.*, 2002).



(I)

We present here the crystal structure of the title salt, (I), obtained from the solution-phase reaction of *tert*-butylamine and oxalic acid (Fig. 1). The complete cation is generated by mirror symmetry, with atoms N1, C2 and C3 lying on the mirror plane. Its geometry is normal. The complete oxalate dianion is generated by 2/*m* point symmetry at the mid-point of the C—C bond and is thus constrained to be planar. The C—O bond distance of 1.240 (1)Å is consistent with related structures (e.g. Alagar *et al.*, 2003).

The crystal packing (Fig. 2) in (I) is stabilized by strong N—H···O hydrogen bonds (Table 1), resulting in (001) layers. Each of the anions is surrounded by six *tert*-butylammonium cations. One of the hydrogen bonds (N1—H1*A*···O6^{i}) is almost linear and the other is bifurcated and much longer. This may explain the increase of the N1—H1*A* bond length from 0.85 (2) to 0.96 (1)Å. Similar effects have been observed in other crystal structures in which the NH hydrogens of the *tert*-butylammonium cations take part in strong hydrogen bonds (Kinbara *et al.*, 1996; Sada *et al.*, 2004; Nagahama *et al.*, 2003).

Experimental

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

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Crystal data

$2C_4H_{12}N^+\cdot C_2O_4^{2-}$
$M_r = 236.31$
Monoclinic, $C2/m$
$a = 11.264$ (2) Å
$b = 7.512$ (1) Å
$c = 9.324$ (1) Å
$\beta = 111.91$ (1)°
$V = 731.97$ (19) Å^3
$Z = 2$
$D_x = 1.072$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.08$ mm^{-1}
$T = 298$ (2) K
Irregular fragment, colourless
0.50 × 0.48 × 0.45 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
ω scans
Absorption correction: none
2385 measured reflections
731 independent reflections
626 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.013$
$\theta_{\rm max} = 25.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.107$
$S = 1.09$
731 reflections
50 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.1997P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.17$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.14$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.069 (10)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O6$^{\rm i}$	0.96 (1)	1.84 (1)	2.785 (1)	171 (1)
N1—H1B···O6	0.85 (2)	2.20 (2)	2.919 (2)	143 (1)
N1—H1B···O6$^{\rm ii}$	0.85 (2)	2.20 (2)	2.919 (2)	143 (1)

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

N-bound H atoms were freely refined. C-bound H atoms were positioned geometrically (C—H = 0.96Å) and refined as riding, with $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(C).

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*.

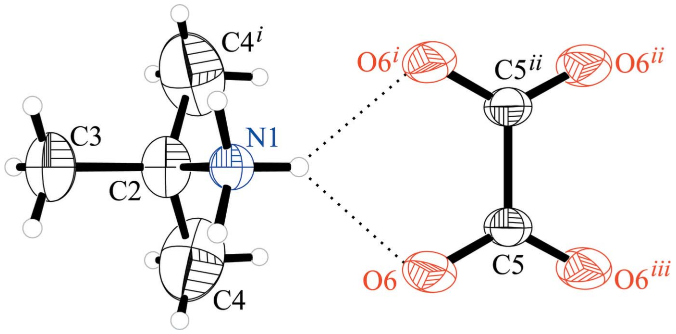

Figure 1
The molecular structure of (I), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). [Symmetry codes: (i) $x, -y, z$; (ii) $-x, -y, 2-z$; (iii) $-x, y, 2-z$.]

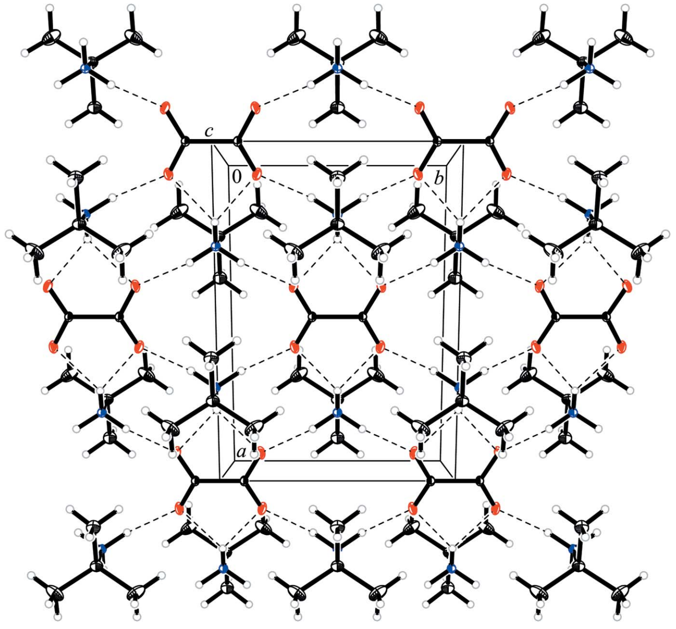

Figure 2
Part of a (001) sheet in (I), showing the hydrogen-bonding scheme (dashed lines).

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