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3,6-Dioxaoctane-1,8-diammonium oxalate

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.076
Data-to-parameter ratio = 12.3

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

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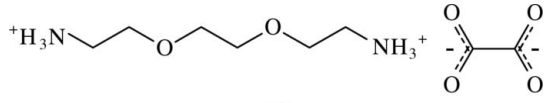
The title compound, $C_6H_{18}N_2O_2^{2+}\cdot C_2O_4^{2-}$, crystallizes with one half-cation and one half-anion in the asymmetric unit. It contains cyclic N—H···O hydrogen-bonded rings involving 3,6-dioxaoctane-1,8-diammonium and oxalate ions, forming a three-dimensional network.

Comment

Analysis of intermolecular interactions in crystalline systems is very important in supramolecular chemistry (Braga *et al.*, 2002). These interactions influence the crystal packing and can provide insight into the collective properties of materials as well as leading to the design of new crystals with specific physical and chemical properties (Lam & Mak, 2000). We have been interested in supramolecular hydrogen-bonded systems formed by organic amines and carboxylic acids (Odabaşoğlu, Büyükgüngör & Lönnecke, 2003; Odabaşoğlu, Büyükgüngör, Turgut *et al.*,2003; Odabaşoğlu & Büyükgüngör, 200a,*b*). The structure presented here, (I), is another example of this type of supramolecular assembly (Fig. 1).



(I)

In (I), the 3,6-dioxaoctane-1,8-diammonium ions are linked to the oxalate ions through N—H···O hydrogen bonds, resulting in the formation of spirocyclic $R_2^2(5)$ and $R_2^2(4)$ hydrogen-bonded rings (Fig. 2 and Table 2). The asymmetric unit contains half of each ion.

N—H···O hydrogen bonds found in amine carboxylates with essentially linear hydrogen bonds exhibit an average N···O bond distance of 2.811 Å and an average N—H···O angle of 158.2° (Vaidhyanathan *et al.*, 2002). Compound (I) has a slightly longer N···O average bond distance of 2.829 (12) Å and a slightly smaller N—H···O average bond angle of 149.9 (12)°. In (I), the C1—N1 bond exhibits a normal Csp^3—Nsp^3 single-bond length (Vaidhyanathan *et al.*, 2002; Odabaşoğlu & Büyükgüngör, 2006), while the C1—C2 single bond is shorter than normal. This shortening can be attributed to the positive inductive effect of the O and N atoms.

Experimental

The title compound was prepared by mixing 2-[2-(2-aminoethoxy)ethoxy]ethanamine and oxalic acid in a 1:1 molar ratio in water at 353 K. Crystals of (I) were obtained by slow evaporation of the solvent (m.p. 494–495 K).

Crystal data

$C_6H_{18}N_2O_2^{2+}\cdot C_2O_4^{2-}$
M_r = 238.24
Monoclinic, $P2_1/c$
a = 7.5119 (7) Å
b = 7.7072 (5) Å
c = 10.879 (1) Å
β = 121.494 (6)°
V = 537.07 (9) Å^3
Z = 2
D_x = 1.473 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7458 reflections
θ = 3.2–28.0°
μ = 0.13 mm^{-1}
T = 100 K
Rounded prism, colorless
0.35 × 0.28 × 0.17 mm

Data collection

Stoe IPDS 2 diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.961, T_{max} = 0.983
7458 measured reflections
1058 independent reflections
952 reflections with $I > 2\sigma(I)$
R_{int} = 0.105
θ_{max} = 26.0°
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.076
S = 1.08
1058 reflections
86 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0231P)^2 + 0.1294P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.093 (6)

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.4817 (15)	C3—C3^{i}	1.521 (2)
C1—C2	1.5047 (15)	C4—O3	1.2434 (13)
C2—O1	1.4247 (13)	C4—O2	1.2541 (13)
C3—O1	1.4241 (14)		
O3—C4—O2	126.71 (9)	O2—C4—C4^{ii}	116.08 (11)
O3—C4—C4^{ii}	117.21 (11)		

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

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H11A···O2^{iii}	0.913 (16)	1.904 (15)	2.7614 (12)	155.6 (12)
N1—H11A···O3^{iv}	0.913 (16)	2.352 (14)	2.9866 (12)	126.5 (10)
N1—H11B···O3^{v}	0.901 (15)	1.976 (15)	2.8138 (12)	154.0 (12)
N1—H11C···O2^{vi}	0.909 (16)	1.869 (16)	2.7530 (12)	163.6 (14)

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

All C-bound H atoms were refined using the riding-model approximation, with C—H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C). N-bound H atoms were located in a Fourier difference map and refined freely.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

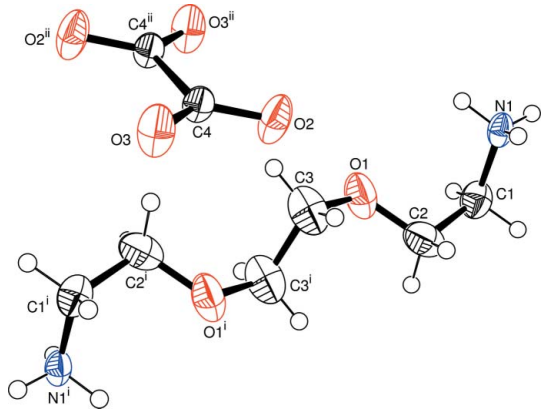


Figure 1
A view of the title molecule with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Symmetry codes are as in Table 1.

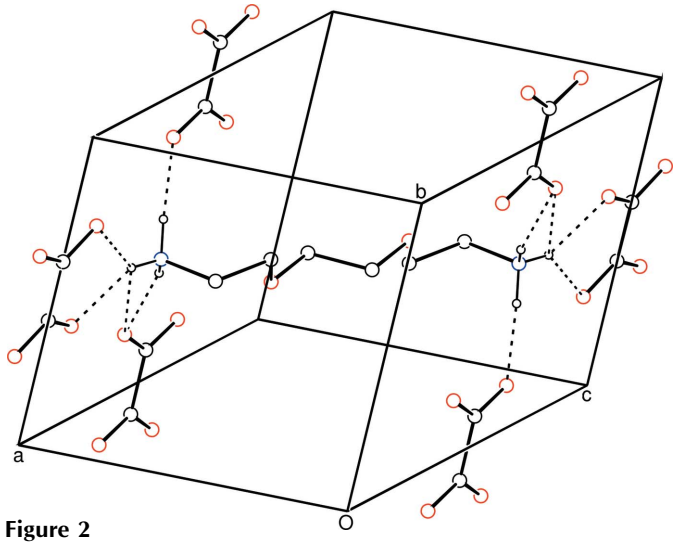


Figure 2
A packing diagram of the title compound, showing the hydrogen-bonding (dashed lines) scheme.

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