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Bis(2-hydroxyethylammonium) oxalate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.041
wR factor = 0.114
Data-to-parameter ratio = 12.5

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

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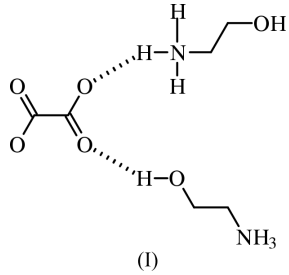
In bis(2-hydroxyethylammonium) oxalate, $2C_2H_8NO^+\cdot C_2O_4^{2-}$, hydrogen bonds involving the hydroxy and ammonium groups connect the carboxyl O atoms of the oxalate anion into a three-dimensional network structure.

Comment

Double salts with small-length alkylammonium cations are of interest because of their optical (Jayasree *et al.*, 1996; Mahadevan Pillai *et al.*, 1997, 1998), ferroelectric, ferroelastic (Kirpichnikova *et al.*, 1990; Vlokh, Bublyk *et al.*, 1991; Vlokh, Kapustyanyuk *et al.*, 1991) and structural (Bator *et al.*, 1998; Kearley, 1983) properties, these compounds often exhibiting several phase transitions at lower temperature. The divalent metal 2-ethanolammonium (Jordanovska & Trojko, 1993) and trivalent metal bis- and tris(2-ethanol)ammonium sulfates (Jordanovska *et al.*, 1996) exist as double salts; however, the reaction of M^{II} oxalates (*M* = Mn, Co, Ni, Cu, Zn and Cd) yields only 2-ethanolamine adducts (Jordanovska & Trojko, 1995).

In our hands, the synthesis of the cadmium oxalate adduct gave only bis(2-ethanolammonium) oxalate. The structures of some ammonium oxalates have been described recently (Krishnakumar *et al.*, 1998; Paixao *et al.*, 1999). The title ammonium oxalate, (I), displays no unusual features; the C—C bond [1.559 (5) Å] in the oxalate anion is also characteristically long. Its ammonium unit is engaged in three hydrogen-bonding interactions, whereas the hydroxyl unit is engaged in one hydrogen-bonding interaction.



(I)

Experimental

An aqueous solution of oxalic acid was neutralized with aqueous ethanolamine to pH 8. The solution was concentrated by evaporating the water. Crystals deposited after cooling the solution to room temperature and these were washed with ethanol and air-dried. A yellow form of the compound was obtained in the attempt to prepare the 2-ethanolamine complex of cadmium(II) oxalate (Jordanovska & Trojko, 1995) by refluxing cadmium oxalate and aqueous 2-ethanolamine in chloroform.

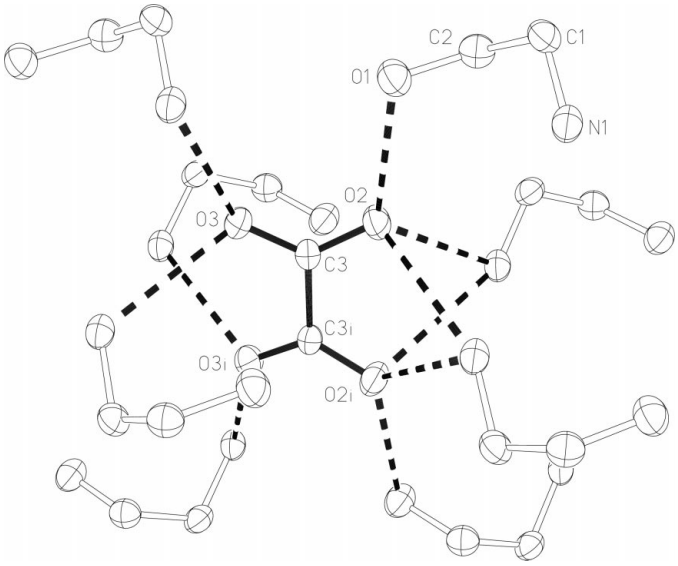


Figure 1
*ORTEP*II (Johnson, 1976) plot with displacement ellipsoids at the 50% probability level.

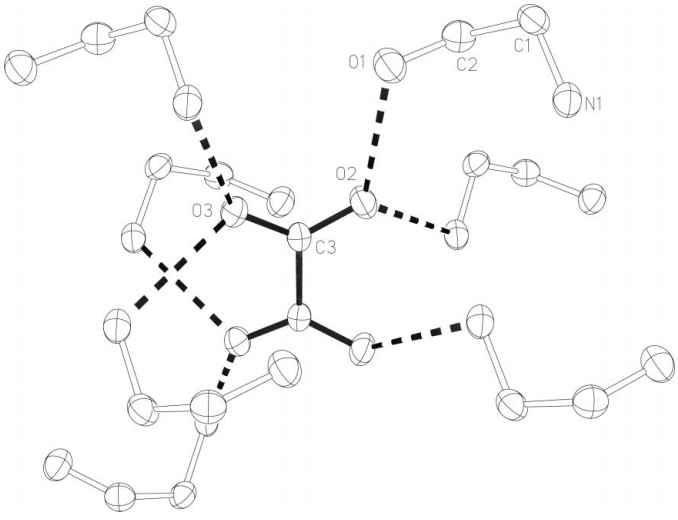


Figure 2
Hydrogen-bonding interactions of the anion.

Crystal data

$2C_2H_8NO^+\cdot C_2O_4^{2-}$
M_r = 212.21
Monoclinic, $C2/c$
a = 17.473 (5) Å
b = 5.916 (2) Å
c = 10.346 (3) Å
β = 118.69 (2)°
V = 938.2 (5) Å^3
Z = 4
D_x = 1.502 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 6.8–18.1°
μ = 0.13 mm^{-1}
T = 298 (2) K
Block, colorless
0.20 × 0.10 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
872 measured reflections
822 independent reflections
550 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 25.0°
$h = -20 \rightarrow 18$
$k = 0 \rightarrow 7$
$l = 0 \rightarrow 12$
3 standard reflections
frequency: 120 min
intensity decay: 7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.114
S = 1.01
822 reflections
66 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0543P)^2 + 0.7120P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.412 (3)	N1—C1	1.481 (3)
O2—C3	1.250 (3)	C1—C2	1.501 (4)
O3—C3	1.248 (3)	C3—C3^{i}	1.559 (5)
N1—C1—C2	111.6 (2)	O3—C3—C3^{i}	117.4 (2)
O1—C2—C1	111.3 (2)	O2—C3—C3^{i}	116.2 (2)
O3—C3—O2	126.4 (2)		

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.82	1.92	2.720 (3)	167
N1—H1A···O3^{i}	0.89	1.97	2.835 (3)	163
N1—H1B···O2^{ii}	0.89	2.00	2.878 (3)	171
N1—H1C···O3^{iii}	0.89	1.98	2.857 (3)	167

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

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CELDIM* in *CAD*-4 *Software* (Enraf–Nonius, 1989); data reduction: *XCAD*4 (Harms, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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