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Hydrogen bonding in ethylenediammonium bis(hydrogen malonate) monohydrate

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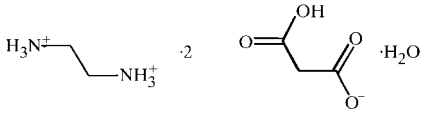
In the title compound, $C_2H_{10}N_2^{2+}\cdot 2C_3H_3O_4^-\cdot H_2O$, the hydrogen malonate anion has an intramolecular O—H···O hydrogen bond of 2.430 (2) Å. The water molecule lies on a twofold axis and connects the anions into pairs through hydrogen bonds of 2.734 (1) Å. The ethylenediammonium cation lies across an inversion centre. Each of the ammonium protons is involved in hydrogen bonding to an anion or a water molecule [N···O 2.815 (2)–2.875 (2) Å].

Comment

Reaction of an amine with a polycarboxylic acid often gives crystals of only one of the possible salts. Frequently, the dominant compound contains a partially ionized acid group, such as hydrogen oxalate or hydrogen malonate, to optimize hydrogen bonding. The hydrogen bonding may be extended further by water molecules (Barnes & Barnes, 1996; Barnes *et al.*, 1998). In the present work, ethylenediammonium bis(hydrogen malonate) monohydrate, (I), was the only crystalline product from aqueous mixtures of 1,2-diaminoethane and malonic acid.



(I)

Golic (Djinovic *et al.*, 1990; Djinovic & Golic, 1991) has discussed the structural patterns of the hydrogen malonate ion. The acidic proton is used either to form chains by interanion hydrogen bonding or, as in (I), in an asymmetric intramolecular hydrogen bond. In (I), the $R_1^1(6)$ ring (Bernstein *et al.*, 1995)., *i.e.* ...O═C—C—C—O—H..., is more symmetrical than in, for example, benzylammonium hydrogen malonate (Djinovic *et al.*, 1990) [values in brackets], with O8—H81 1.13 (2) Å [0.80 Å] and H8···O7 1.33 (2) Å [1.67 Å]. In (I), C4—C5 is only 3σ shorter than C3—C4 compared with 7σ in benzylammonium hydrogen malonate.

The water molecule, O10, lies on the twofold axis. Hydrogen bonds connect the anions into pairs [O10—H101···O6 2.734 (1) Å] and the centrosymmetric cations into chains [N1—H1*B*···O10 2.848 (2) Å]. The ammonium protons H1*A* and H1*C* form hydrogen bonds to O6 and O9 of separate anions (see Table 2).

Experimental

Crystals were grown by slow evaporation of an aqueous mixture of 1,2-diaminoethane and malonic acid (1:1).

Crystal data

$C_2H_{10}N_2^{2+}\cdot 2C_3H_3O_4^-\cdot H_2O$
M_r = 286.24
Monoclinic, $P2/n$
a = 8.0276 (10) Å
b = 8.7013 (11) Å
c = 9.543 (2) Å
β = 105.901 (13)°
V = 641.05 (18) Å^3
Z = 2
D_x = 1.483 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 14.4–15.0°
μ = 0.136 mm^{-1}
T = 293 (2) K
Block, colourless
0.52 × 0.50 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
2227 measured reflections
1865 independent reflections
1441 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 29.97°
$h = -1 \rightarrow 11$
$k = 0 \rightarrow 12$
$l = -13 \rightarrow 13$
3 standard reflections
frequency: 60 min
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.159
S = 0.787
1865 reflections
100 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1366P)^2 + 0.1458P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.35 (3)

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.4711 (18)	C3—C4	1.5203 (17)
C2—C2^{i}	1.507 (2)	C4—C5	1.5154 (19)
C3—O6	1.2340 (17)	C5—O9	1.2192 (19)
C3—O7	1.2693 (15)	C5—O8	1.2975 (19)
N1—C2—C2^{i}	110.44 (13)	C5—C4—C3	117.33 (11)
O6—C3—O7	123.27 (12)	O9—C5—O8	121.92 (15)
O6—C3—C4	118.59 (11)	O9—C5—C4	121.31 (14)
O7—C3—C4	118.14 (12)	O8—C5—C4	116.77 (12)
O6—C3—C4—C5	−171.40 (12)	C3—C4—C5—O8	−7.13 (18)

Symmetry codes: (i) $-x, 2-y, 1-z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O6	0.89	1.94	2.8152 (16)	165.7
N1—H1B···O10^{i}	0.89	2.12	2.8483 (15)	138.5
N1—H1C···O9^{ii}	0.89	2.02	2.875 (2)	161.9
O8—H81···O7	1.13 (2)	1.33 (2)	2.4297 (17)	162 (2)
O10—H101···O6	0.87 (2)	1.889 (19)	2.7342 (13)	165 (2)

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

Data collection: *CAD*-4/*PC* (Enraf–Nonius, 1993); cell refinement: *CAD*-4/*PC*; data reduction: *XCAD*4 (Harms & Wocadlo, 1996); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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