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Melaminium bis(hydrogen oxalate)

Xiu-Lian Zhang,[a] Xiao-Ming Chen[a] and Seik Weng Ng[b]*

[a]School of Chemistry and Chemical Engineering, Sun Yat-Sen University, Guangzhou 510275, People's Republic of China, and [b]Department of Chemistry, University of Malaya, Kuala Lumpur 50603, Malaysia

Correspondence e-mail: seikweng@um.edu.my

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Key indicators

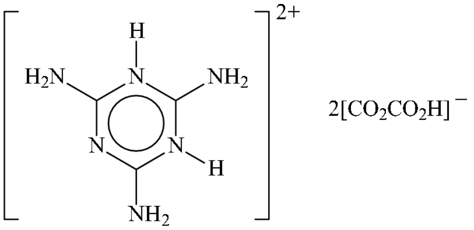
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.116
Data-to-parameter ratio = 10.8

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

The crystal structure of melaminium bis(hydrogen oxalate), $C_3H_8N_6^{2+}\cdot 2C_2HO_4^-$, contains hydrogen oxalate anions that are linked by hydrogen bonds into a linear chain; adjacent chains are linked through the dication (which has crystallographic twofold symmetry), forming a layer structure.

Comment

A number of melaminium salts of carboxylic acids have been reported, *e.g.* melaminium acetate hydrate–acetic acid (Perpétuo & Janczak, 2002), melaminium levulinate hydrate (Choi *et al.*, 2004), dimelaminium glutarate monohydrate (Janczak & Perpétuo, 2002), dimelaminium malate tetrahydrate (Janczak & Perpétuo, 2003), dimelaminium maleate monohydrate (Janczak & Perpétuo, 2004), dimelaminium phthalate (Janczak & Perpétuo, 2001*a*) and dimelaminium terephthalate dihydrate (Zhang *et al.*, 2004). These carboxylates have the melamine moiety as a monoprotonated cation; possibly, the carboxylic acids are not acidic enough to protonate the monoprotonated cation. An exception is pyromellitic acid, which has been isolated as the dihydrated melaminium(2+) salt; the synthesis required rather vigorous hydrothermal conditions (Karle *et al.*, 2003). Otherwise, the melaminium(2+) entity has been documented only for the 4-hydroxybenzenesulfonate (Janczak & Perpétuo, 2001*b*), perchlorate hydrate (Martin & Pinkerton, 1995) and tetrachlorocuprate (Colombo *et al.*, 1985). The title salt, (I), is an example of a melaminium(2+) carboxylate; in the dication, the three amino substituents are planar, and are coplanar with the ring.



(I)

The hydrogen oxalate counter-ion interacts by a hydrogen bond to another anion to furnish a linear chain that propogates along the b axis. The melaminium(2+) dications surround the chain. Each dication lies on a special position of site symmetry 2, so that they connect adjacent chains into a layer structure (Fig. 1). The hydrogen oxalate ion is known to prefer chain formation through extremely short (*ca* 2.5 Å) hydrogen bonds; it usually adopts a planar conformation (Barnes *et al.*, 1998). In the title compound, the C3—C4 bond

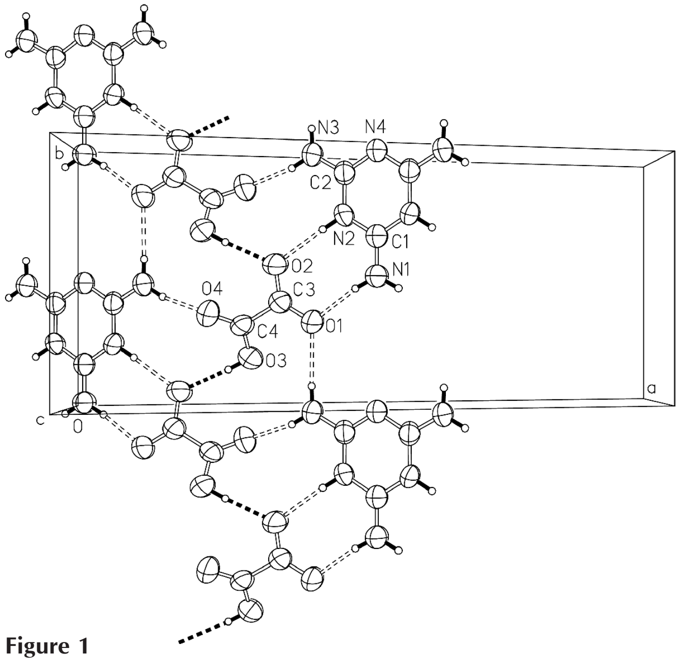


Figure 1
*ORTEP*II (Johnson, 1976) plot of a part of the packing of melaminium bis(hydrogen oxalate), illustrating the hydrogen bonds (dashed lines). Displacement ellipsoids are drawn at the 90% probability level and H atoms are drawn as spheres of arbitrary radii

is relatively long and the anion nearly planar, though the anion is not required by symmetry to be so.

Experimental

A hot aqueous solution of melamine (0.50 mmol, 0.063 g) was mixed with a hot aqueous solution of oxalic acid dihydrate (1 mmol, 0.126 g). Colorless crystals separated from the solution after a week. Analysis found: C 27.53, H 3.33, N 27.43%; calculated for $C_7H_{10}N_6O_8$: C 27.55, H 3.29, N 27.41%.

Crystal data

$C_3H_8N_6^{2+}\cdot 2C_2HO_4^-$
$M_r = 306.21$
Monoclinic, $C2/c$
$a = 20.829$ (2) Å
$b = 8.428$ (1) Å
$c = 6.8415$ (8) Å
$\beta = 108.402$ (2)°
$V = 1139.6$ (2) Å^3
$Z = 4$
$D_x = 1.785$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1045 reflections
$\theta = 3.3$–27.3°
$\mu = 0.16$ mm^{-1}
$T = 295$ (2) K
Block, colorless
0.20 × 0.16 × 0.15 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: none
3287 measured reflections
1269 independent reflections
1068 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\rm max} = 27.5°$
$h = -25 \rightarrow 26$
$k = -10 \rightarrow 9$
$l = -4 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.116$
$S = 1.06$
1269 reflections
117 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0711P)^2 + 0.2079P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.26$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.242 (2)	C1—N2	1.349 (2)
O2—C3	1.257 (2)	C2—N2	1.374 (2)
O3—C4	1.305 (2)	C2—N3	1.308 (2)
O4—C4	1.214 (2)	C2—N4	1.326 (2)
C1—N1	1.294 (3)	C3—C4	1.535 (2)
C1—N2—C2	120.5 (1)	O1—C3—O2	126.8 (1)
C2$^{\rm i}$—N4—C2	117.5 (2)	O1—C3—C4	117.9 (1)
N1—C1—N2	121.3 (1)	O2—C3—C4	115.4 (1)
N2—C1—N2$^{\rm i}$	117.4 (2)	O3—C4—O4	125.1 (2)
N2—C2—N3	118.0 (1)	O3—C4—C3	113.4 (1)
N2—C2—N4	122.0 (1)	O4—C4—C3	121.5 (1)
N3—C2—N4	119.9 (1)		

Symmetry code: (i) $1 - x, y, \frac{3}{2} - z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3o···O2$^{\rm ii}$	0.86 (1)	1.84 (1)	2.699 (2)	176 (2)
N1—H1···O1	0.85 (1)	1.90 (1)	2.748 (2)	173 (2)
N2—H2···O2	0.86 (1)	1.94 (1)	2.792 (2)	173 (2)
N3—H3a···O1$^{\rm iii}$	0.85 (1)	2.11 (1)	2.959 (2)	175 (2)
N3—H3b···O4$^{\rm iv}$	0.86 (1)	2.03 (1)	2.861 (2)	164 (2)

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

H atoms were located and refined with a distance restraint of O—H = N—H = 0.85 (1) Å.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; 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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