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Bis(tetramethylammonium) oxalate monohydrate

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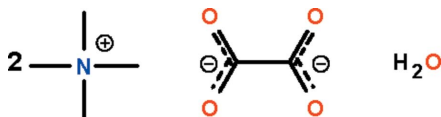
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.053; wR factor = 0.164; data-to-parameter ratio = 13.8.

In the crystal structure of the title hydrated salt, $2\text{C}_4\text{H}_{12}\text{N}^+\cdot\text{C}_2\text{O}_4^{2-}\cdot\text{H}_2\text{O}$, the two independent cations, the anion and the water molecule all lie on special positions of m site symmetry. In both cations, the mirror plane passes through the nitrogen atom and two methyl groups; in the anion, the mirror plane passes through two carbon and two oxygen atoms. The anions and water molecules interact by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonding, forming a chain running along the b axis.

Related literature

For the crystal structure of tetramethylammonium hydrogen oxalate, see: Mascal *et al.* (2000).



Experimental

Crystal data

 $2\text{C}_4\text{H}_{12}\text{N}^+\cdot\text{C}_2\text{O}_4^{2-}\cdot\text{H}_2\text{O}$ $M_r = 254.33$ Orthorhombic, $Pnma$ $a = 24.614$ (4) Å $b = 6.738$ (1) Å $c = 8.633$ (2) Å $V = 1431.8$ (4) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.09$ mm⁻¹ $T = 293$ K $0.50 \times 0.10 \times 0.10$ mm

Data collection

Bruker APEX2 diffractometer

Absorption correction: none

3915 measured reflections

1367 independent reflections

1043 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.024$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.053$ $wR(F^2) = 0.164$ $S = 1.01$

1367 reflections

99 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.25$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1W}-\text{H1}\cdots\text{O3}$	0.95 (3)	1.82 (3)	2.764 (2)	171 (3)

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

We thank Beijing Normal University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2618).

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supplementary materials

Acta Cryst. (2009). E65, o2602 [doi:10.1107/S1600536809039099]

Bis(tetramethylammonium) oxalate monohydrate

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Experimental

Oxalic acid (0.126 g, 1 mmol) was dissolved in a water-ethanol (1:2 v/v) mixture and a 25% solution of tetramethylammonium hydroxide was added to neutralize the acid. Colorless block crystals were separated after several weeks.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.96 Å) and were included in the refinement in the riding model approximation, with $U(\text{H})$ set to $1.5U(\text{C})$. The water H-atom was freely refined.

Figures

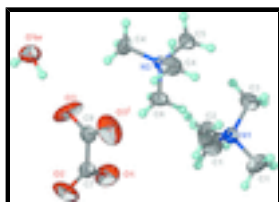
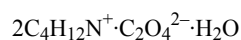


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $2[(\text{CH}_3)_4\text{N}](\text{C}_2\text{O}_4)\cdot\text{H}_2\text{O}$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Bis(tetramethylammonium) oxalate monohydrate

Crystal data



$$M_r = 254.33$$

Orthorhombic, *Pnma*

Hall symbol: -P 2ac 2n

$$a = 24.614 (4) \text{ \AA}$$

$$b = 6.738 (1) \text{ \AA}$$

$$c = 8.633 (2) \text{ \AA}$$

$$V = 1431.8 (4) \text{ \AA}^3$$

$$Z = 4$$

$$F_{000} = 560$$

$$D_x = 1.180 \text{ Mg m}^{-3}$$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1201 reflections

$$\theta = 2.5\text{--}25.0^\circ$$

$$\mu = 0.09 \text{ mm}^{-1}$$

$$T = 293 \text{ K}$$

Block, colorless

$$0.50 \times 0.10 \times 0.10 \text{ mm}$$

Data collection

Bruker APEX2
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$$T = 293 \text{ K}$$

1043 reflections with $I > 2\sigma(I)$

$$R_{\text{int}} = 0.024$$

$$\theta_{\text{max}} = 25.0^\circ$$

$$\theta_{\text{min}} = 2.5^\circ$$

supplementary materials

φ and ω scans $h = -29 \rightarrow 11$
Absorption correction: None $k = -8 \rightarrow 8$
3915 measured reflections $l = -10 \rightarrow 8$
1367 independent reflections

Refinement

Refinement on F^2 Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full H atoms treated by a mixture of independent and constrained refinement
 $R[F^2 > 2\sigma(F^2)] = 0.053$ $w = 1/[\sigma^2(F_o^2) + (0.0847P)^2 + 0.5757P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.164$ $(\Delta/\sigma)_{\max} = 0.001$
 $S = 1.01$ $\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$
1367 reflections $\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$
99 parameters Extinction correction: SHELXL97 (Sheldrick, 2008),
 $F_c^* = kFc[1 + 0.001x Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods Extinction coefficient: 0.022 (5)
Secondary atom site location: difference Fourier map

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.29989 (10)	0.7500	0.4672 (3)	0.0944 (10)	
O2	0.38001 (14)	0.7500	0.3510 (3)	0.1103 (12)	
O3	0.38835 (9)	0.5888 (4)	0.6773 (3)	0.1216 (10)	
O1W	0.40771 (12)	0.2500	0.8491 (3)	0.0790 (8)	
H1	0.3983 (11)	0.358 (4)	0.784 (3)	0.098 (9)*	
N1	0.04672 (8)	0.7500	0.7763 (3)	0.0442 (6)	
N2	0.27226 (8)	0.7500	1.0044 (2)	0.0372 (6)	
C1	0.02478 (11)	0.5681 (4)	0.7022 (3)	0.0864 (9)	
H1A	-0.0141	0.5665	0.7121	0.130*	
H1B	0.0397	0.4531	0.7522	0.130*	
H1C	0.0345	0.5669	0.5945	0.130*	
C2	0.10673 (11)	0.7500	0.7595 (5)	0.0679 (10)	
H2A	0.1218	0.8579	0.8181	0.102*	0.50
H2B	0.1161	0.7654	0.6522	0.102*	0.50
H2C	0.1211	0.6267	0.7974	0.102*	0.50
C3	0.03268 (14)	0.7500	0.9431 (3)	0.0652 (9)	
H3A	-0.0060	0.7379	0.9548	0.098*	0.50
H3B	0.0447	0.8719	0.9894	0.098*	0.50
H3C	0.0502	0.6402	0.9932	0.098*	0.50
C4	0.30685 (10)	0.5697 (3)	1.0196 (3)	0.0620 (7)	
H4A	0.3247	0.5708	1.1185	0.093*	
H4B	0.3336	0.5689	0.9387	0.093*	
H4C	0.2846	0.4533	1.0113	0.093*	

C5	0.22984 (12)	0.7500	1.1282 (3)	0.0522 (8)	
H5A	0.2459	0.7116	1.2249	0.078*	0.50
H5B	0.2016	0.6577	1.1014	0.078*	0.50
H5C	0.2147	0.8807	1.1377	0.078*	0.50
C6	0.24524 (12)	0.7500	0.8504 (3)	0.0548 (8)	
H6A	0.2219	0.8636	0.8423	0.082*	0.50
H6B	0.2241	0.6311	0.8391	0.082*	0.50
H6C	0.2723	0.7554	0.7704	0.082*	0.5
C7	0.35047 (12)	0.7500	0.4666 (3)	0.0486 (7)	
C8	0.37795 (10)	0.7500	0.6197 (3)	0.0479 (7)	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0651 (17)	0.124 (3)	0.0938 (19)	0.000	-0.0318 (14)	0.000
O2	0.134 (3)	0.146 (3)	0.0503 (15)	0.000	0.0259 (16)	0.000
O3	0.1149 (17)	0.126 (2)	0.1237 (18)	0.0185 (14)	-0.0231 (13)	0.0734 (15)
O1W	0.113 (2)	0.0643 (16)	0.0591 (15)	0.000	-0.0145 (14)	0.000
N1	0.0386 (12)	0.0467 (13)	0.0473 (13)	0.000	0.0020 (9)	0.000
N2	0.0416 (11)	0.0366 (11)	0.0334 (11)	0.000	-0.0011 (9)	0.000
C1	0.0809 (17)	0.093 (2)	0.0849 (17)	-0.0298 (16)	0.0007 (14)	-0.0317 (15)
C2	0.0404 (15)	0.060 (2)	0.103 (3)	0.000	0.0112 (16)	0.000
C3	0.069 (2)	0.078 (2)	0.0486 (17)	0.000	0.0046 (15)	0.000
C4	0.0677 (13)	0.0551 (14)	0.0631 (13)	0.0215 (11)	-0.0031 (10)	0.0016 (11)
C5	0.0562 (17)	0.0583 (18)	0.0419 (15)	0.000	0.0097 (12)	0.000
C6	0.0586 (17)	0.071 (2)	0.0352 (14)	0.000	-0.0093 (12)	0.000
C7	0.0614 (18)	0.0383 (15)	0.0461 (15)	0.000	-0.0003 (13)	0.000
C8	0.0356 (14)	0.0592 (18)	0.0489 (15)	0.000	0.0053 (11)	0.000

Geometric parameters (\AA , $^\circ$)

O1—C7	1.245 (4)	C2—H2B	0.9600
O2—C7	1.235 (4)	C2—H2C	0.9600
O3—C8	1.222 (2)	C3—H3A	0.9600
O1W—H1	0.95 (3)	C3—H3B	0.9600
N1—C3	1.481 (3)	C3—H3C	0.9600
N1—C2	1.484 (3)	C4—H4A	0.9600
N1—C1	1.484 (3)	C4—H4B	0.9600
N1—C1 ⁱ	1.484 (3)	C4—H4C	0.9600
N2—C6	1.487 (3)	C5—H5A	0.9600
N2—C4	1.489 (2)	C5—H5B	0.9600
N2—C4 ⁱ	1.489 (2)	C5—H5C	0.9600
N2—C5	1.494 (3)	C6—H6A	0.9600
C1—H1A	0.9600	C6—H6B	0.9600
C1—H1B	0.9600	C6—H6C	0.9600
C1—H1C	0.9600	C7—C8	1.484 (4)
C2—H2A	0.9600	C8—O3 ⁱ	1.222 (2)
C3—N1—C2	109.1 (3)	N1—C3—H3C	109.5

supplementary materials

C3—N1—C1	109.52 (16)	H3A—C3—H3C	109.5
C2—N1—C1	108.66 (17)	H3B—C3—H3C	109.5
C3—N1—C1 ⁱ	109.52 (16)	N2—C4—H4A	109.5
C2—N1—C1 ⁱ	108.66 (17)	N2—C4—H4B	109.5
C1—N1—C1 ⁱ	111.3 (3)	H4A—C4—H4B	109.5
C6—N2—C4	109.54 (13)	N2—C4—H4C	109.5
C6—N2—C4 ⁱ	109.54 (13)	H4A—C4—H4C	109.5
C4—N2—C4 ⁱ	109.3 (2)	H4B—C4—H4C	109.5
C6—N2—C5	109.1 (2)	N2—C5—H5A	109.5
C4—N2—C5	109.68 (14)	N2—C5—H5B	109.5
C4 ⁱ —N2—C5	109.68 (14)	H5A—C5—H5B	109.5
N1—C1—H1A	109.5	N2—C5—H5C	109.5
N1—C1—H1B	109.5	H5A—C5—H5C	109.5
H1A—C1—H1B	109.5	H5B—C5—H5C	109.5
N1—C1—H1C	109.5	N2—C6—H6A	109.5
H1A—C1—H1C	109.5	N2—C6—H6B	109.5
H1B—C1—H1C	109.5	H6A—C6—H6B	109.5
N1—C2—H2A	109.5	N2—C6—H6C	109.5
N1—C2—H2B	109.5	H6A—C6—H6C	109.5
H2A—C2—H2B	109.5	H6B—C6—H6C	109.5
N1—C2—H2C	109.5	O2—C7—O1	126.3 (3)
H2A—C2—H2C	109.5	O2—C7—C8	116.8 (3)
H2B—C2—H2C	109.5	O1—C7—C8	116.9 (3)
N1—C3—H3A	109.5	O3 ⁱ —C8—O3	125.5 (3)
N1—C3—H3B	109.5	O3 ⁱ —C8—C7	117.26 (17)
H3A—C3—H3B	109.5	O3—C8—C7	117.26 (17)
O2—C7—C8—O3 ⁱ	89.9 (2)	O2—C7—C8—O3	-89.9 (2)
O1—C7—C8—O3 ⁱ	-90.1 (2)	O1—C7—C8—O3	90.1 (2)

Symmetry codes: (i) $x, -y+3/2, z$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—H1 \cdots O3	0.95 (3)	1.82 (3)	2.764 (2)	171 (3)

Fig. 1

