organic compounds

Bis(tetramethylammonium) oxalate monohydrate

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In the crystal structure of the title hydrated salt, $2C_4H_{12}N^+ \cdot C_2O_4^{2-} \cdot H_2O$, 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 O—H···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).
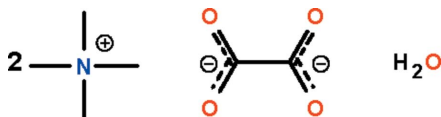
Experimental

Crystal data

$2C_4H_{12}N^+ \cdot C_2O_4^{2-} \cdot H_2O$

$M_r = 254.33$

Orthorhombic, *Pnma*

$a = 24.614$ (4) Å

$b = 6.738$ (1) Å

$c = 8.633$ (2) Å

$V = 1431.8$ (4) Å^3

$Z = 4$

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 $I > 2\sigma(I)$

$R_{int} = 0.024$

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

$\Delta\rho_{max} = 0.25$ e Å^{-3}

$\Delta\rho_{min} = -0.25$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1···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).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.

Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Mascal, M., Marjo, C. E. & Blake, A. J. (2000). *Chem. Commun.* pp. 1591–1592.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Westrip, S. P. (2009). *publCIF*. In preparation.

supporting information

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S1. Experimental

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

S2. 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(H) set to $1.5U$(C). The water H-atom was freely refined.
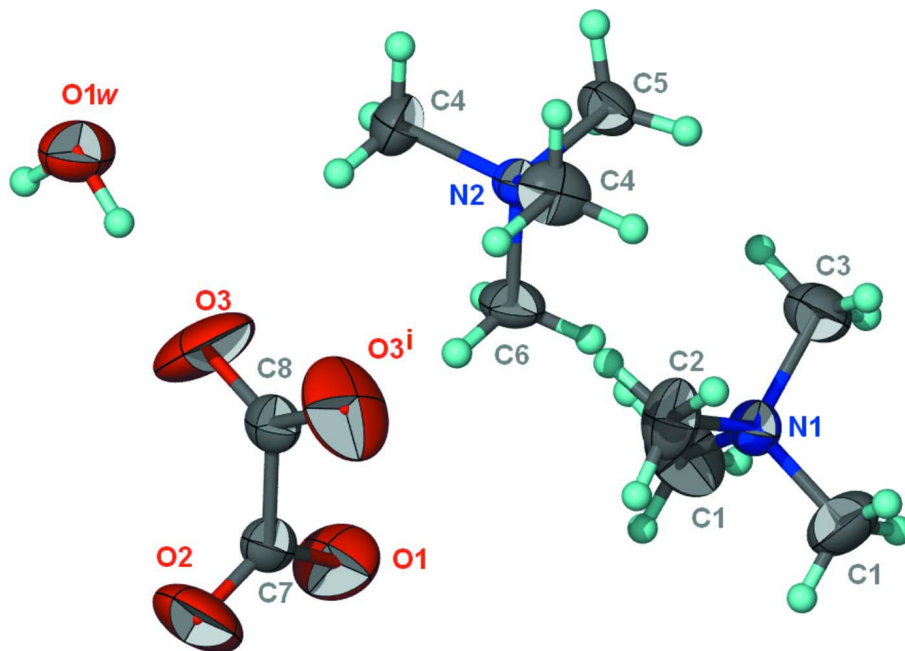
Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $2[(CH_3)_4N](C_2O_4)\cdot H_2O$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Bis(tetramethylammonium) oxalate monohydrate

Crystal data

$2C_4H_{12}N^+\cdot C_2O_4{}^{2-}\cdot H_2O$	$b = 6.738$ (1) Å
$M_r = 254.33$	$c = 8.633$ (2) Å
Orthorhombic, $Pnma$	$V = 1431.8$ (4) Å³
Hall symbol: -P 2ac 2n	$Z = 4$
$a = 24.614$ (4) Å	$F(000) = 560$

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

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1201 reflections

$\theta = 2.5–25.0°$

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

$T = 293$ K

Block, colorless

$0.50 \times 0.10 \times 0.10$ mm

Data collection

Bruker APEX2
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

3915 measured reflections

1367 independent reflections

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

$R_{int} = 0.024$

$\theta_{max} = 25.0°$, $\theta_{min} = 2.5°$

$h = -29\rightarrow11$

$k = -8\rightarrow8$

$l = -10\rightarrow8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.053$

$wR(F^2) = 0.164$

$S = 1.01$

1367 reflections

99 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0847P)^2 + 0.5757P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.25$ e Å^{-3}

$\Delta\rho_{min} = -0.25$ e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick,
2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$

Extinction coefficient: 0.022 (5)

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

	x	y	z	$U_{iso}*/U_{eq}$	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 (Å^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 (Å, º)

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

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1···O3	0.95 (3)	1.82 (3)	2.764 (2)	171 (3)