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Bis(tetramethylammonium) thiosulfate tetrahydrate

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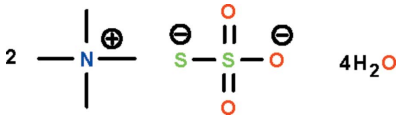
Key indicators: single-crystal X-ray study; T = 130 K; mean σ(N–C) = 0.003 Å; R factor = 0.045; wR factor = 0.127; data-to-parameter ratio = 20.0.

The anion of the title salt, $2C_4H_{12}N^+\cdot S_2O_3^{2-}\cdot 4H_2O$, possesses approximate C_{3v} symmetry. The water molecules themselves engage in hydrogen bonding, forming a ribbon running along the a axis; adjacent chains are linked to the thiosulfate anions by hydrogen bonds, forming a three-dimensional network. The cavities in the network are occupied by the tetramethylammonium counter ions.

Related literature

For tetraethylammonium thiosulfate dihydrate, see: Leyten *et al.* (1988).



Experimental

Crystal data

$2C_4H_{12}N^+\cdot S_2O_3^{2-}\cdot 4H_2O$
M_r = 332.48
Monoclinic, $P2_1/n$
a = 8.1869 (1) Å
b = 15.4342 (2) Å
c = 14.0867 (2) Å
β = 94.074 (1)°
V = 1775.47 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 130 K
0.25 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.923, T_{max} = 0.953
11725 measured reflections
4080 independent reflections
3531 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.127
S = 1.02
4080 reflections
204 parameters
12 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.74 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H11···O1	0.83 (1)	1.88 (1)	2.706 (3)	174 (3)
O1W—H12···O3W	0.84 (2)	1.92 (2)	2.758 (2)	178 (1)
O2W—H21···O1W	0.84 (2)	1.86 (2)	2.689 (2)	172 (2)
O2W—H22···O4W	0.84 (2)	1.90 (3)	2.736 (2)	173 (3)
O3W—H31···O2^{i}	0.83 (2)	1.93 (2)	2.759 (2)	172 (2)
O3W—H32···O2W^{ii}	0.84 (2)	1.88 (2)	2.713 (2)	173 (2)
O4W—H41···S2^{iii}	0.83 (2)	2.51 (2)	3.3280 (19)	170 (2)
O4W—H42···O3W^{iii}	0.83 (2)	1.93 (2)	2.760 (2)	179 (2)

Symmetry codes: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, -y+2, -z+1$; (iii) $x-1, y, z$.

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, 2010).

We thank Northwest Normal University, China, and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5240).

References

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Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

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Acta Cryst. (2011). E**67**, o1664 [doi:10.1107/S1600536811021672]

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S1. Comment

The thiosulfate anion of tetramethylammonium thiosulfate tetrahydrate (Scheme I, Fig. 1) resulted from the decomposition of 1,2-hydrazinedicarbothioamide under basic conditions. The anion of the salt, tetramethylammonium thiosulfate tetrahydrate, possesses approximate C_{3v} symmetry. The four water molecules themselves engage in hydrogen bonding to form a ribbon running along the *a*-axis of the monoclinic unit cell; adjacent chains are linked to the thiosulfate anion by hydrogen bonds to form a three-dimensional network. The cavities in the network are occupied by the ammonium counterions. Tetraethylammonium thiosulfate exists as a dihydrate; in this salt, the sulfur-sulfur bond is 2.028 (1) Å (Leyten *et al.*, 1988).

S2. Experimental

1,2-Hydrazinedicarbothioamide (0.25 mmol, 0.038 g) was dissolved in tetramethylammonium hydroxide (25% aqueous solution) in a 1:2 molar ratio. A small quantity of water-ethanol (1:2) was added to dissolve the reactants completely. The mixture was set aside for the growth of colorless crystals, which separated after several days.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.98 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.5*U*(C).

The water H-atoms were located in a difference Fourier map, and were refined with a distance restraint of O–H 0.84±0.01 Å; their temperature factors were freely refined.

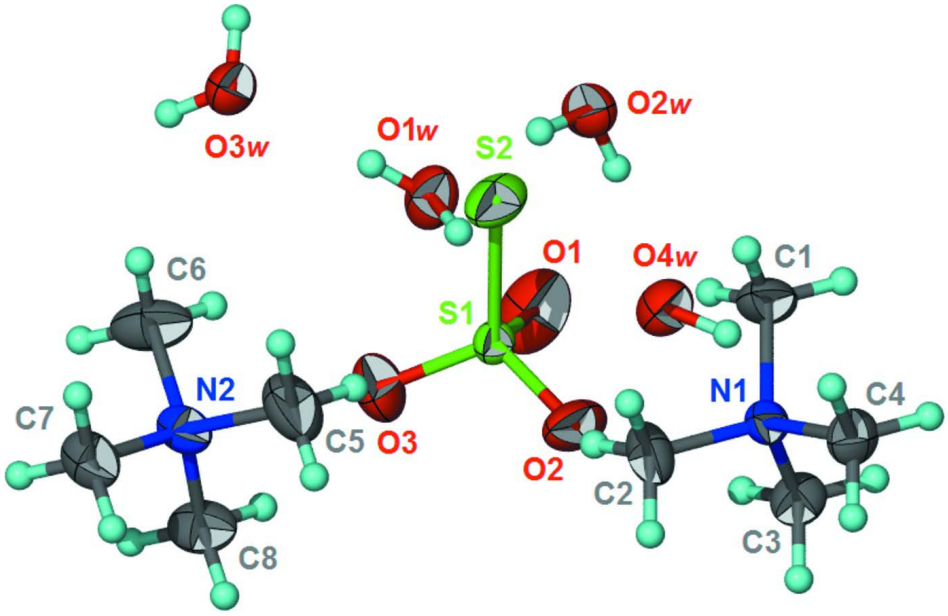

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $2(CH_3)_4N^+ S_2O_3^{2-}\cdot 4H_2O$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

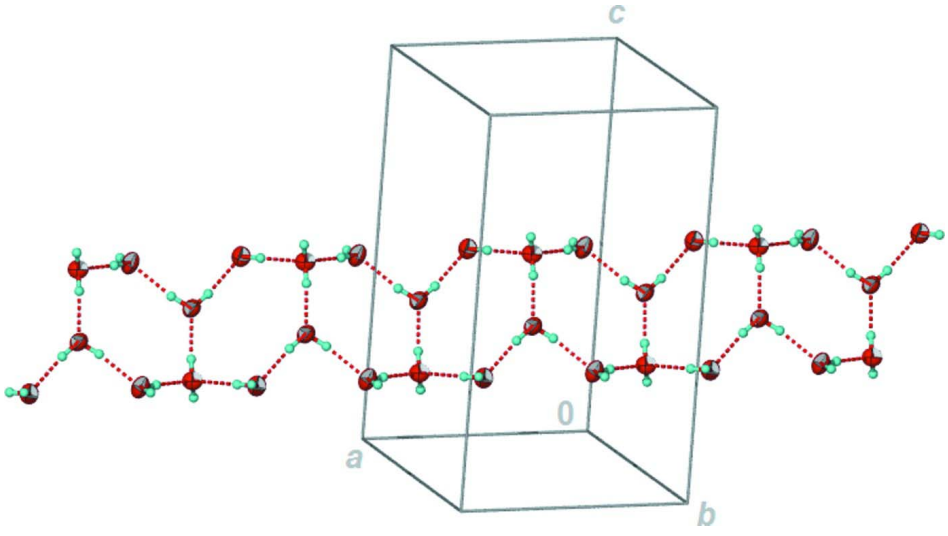

Figure 2

Ribbon motif arising from hydrogen bonds involving water molecules.

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$c = 14.0867$ (2) Å
$\beta = 94.074$ (1)°
$V = 1775.47$ (4) Å^3
$Z = 4$
$F(000) = 728$
$D_x = 1.244$ Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5675 reflections
θ = 2.8–27.6°
μ = 0.33 mm^{-1}
T = 130 K
Block, colorless
0.25 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.923, T_{max} = 0.953
11725 measured reflections
4080 independent reflections
3531 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 27.6°, θ_{min} = 2.0°
h = −10→10
k = −20→20
l = −18→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.127
S = 1.02
4080 reflections
204 parameters
12 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
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 2.4726P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.74 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
S1	0.69577 (6)	0.66503 (3)	0.67106 (3)	0.02068 (13)
S2	0.83493 (9)	0.67111 (4)	0.56042 (5)	0.04154 (18)
O1	0.5466 (2)	0.71568 (13)	0.64692 (18)	0.0556 (6)
O2	0.6474 (3)	0.57509 (11)	0.68608 (12)	0.0437 (5)
O3	0.7837 (3)	0.70123 (13)	0.75481 (13)	0.0495 (5)
O1W	0.4917 (2)	0.88844 (12)	0.63615 (14)	0.0384 (4)
O2W	0.2208 (2)	0.92082 (12)	0.52239 (12)	0.0346 (4)
O3W	0.7668 (2)	0.99207 (10)	0.64424 (12)	0.0322 (4)
O4W	−0.0083 (2)	0.85939 (12)	0.63760 (13)	0.0353 (4)
N1	0.7309 (2)	0.37242 (11)	0.56622 (11)	0.0223 (3)
N2	1.2319 (2)	0.64015 (13)	0.83568 (12)	0.0277 (4)
C1	0.7226 (3)	0.43773 (15)	0.48818 (16)	0.0361 (5)
H1A	0.7122	0.4958	0.5152	0.054*
H1B	0.6274	0.4257	0.4440	0.054*
H1C	0.8226	0.4346	0.4540	0.054*
C2	0.8740 (3)	0.39114 (17)	0.63428 (16)	0.0344 (5)
H2A	0.8628	0.4494	0.6608	0.052*
H2B	0.9749	0.3880	0.6010	0.052*
H2C	0.8785	0.3484	0.6859	0.052*
C3	0.5774 (3)	0.37662 (15)	0.61729 (16)	0.0301 (5)
H3A	0.5661	0.4346	0.6444	0.045*

H3B	0.5823	0.3335	0.6685	0.045*
H3C	0.4831	0.3645	0.5725	0.045*
C4	0.7481 (3)	0.28336 (14)	0.52585 (16)	0.0309 (5)
H4A	0.6541	0.2711	0.4808	0.046*
H4B	0.7520	0.2407	0.5775	0.046*
H4C	0.8492	0.2800	0.4928	0.046*
C5	1.2059 (4)	0.5696 (2)	0.76359 (19)	0.0487 (7)
H5A	1.2113	0.5132	0.7957	0.073*
H5B	1.2911	0.5727	0.7183	0.073*
H5C	1.0981	0.5766	0.7295	0.073*
C6	1.2232 (3)	0.72610 (19)	0.7873 (2)	0.0452 (7)
H6A	1.1151	0.7332	0.7536	0.068*
H6B	1.3079	0.7295	0.7417	0.068*
H6C	1.2407	0.7721	0.8349	0.068*
C7	1.3963 (3)	0.62940 (17)	0.88745 (16)	0.0337 (5)
H7A	1.4021	0.5728	0.9189	0.051*
H7B	1.4132	0.6753	0.9353	0.051*
H7C	1.4815	0.6331	0.8421	0.051*
C8	1.1031 (3)	0.63532 (18)	0.90535 (17)	0.0374 (5)
H8A	1.1080	0.5786	0.9368	0.056*
H8B	0.9950	0.6431	0.8719	0.056*
H8C	1.1217	0.6811	0.9532	0.056*
H11	0.515 (3)	0.8363 (7)	0.641 (2)	0.036 (8)*
H12	0.576 (2)	0.9191 (13)	0.638 (2)	0.058 (10)*
H21	0.3104 (18)	0.912 (2)	0.5532 (18)	0.046 (8)*
H22	0.145 (2)	0.903 (3)	0.554 (2)	0.085 (14)*
H31	0.791 (4)	1.0218 (15)	0.6926 (11)	0.049 (9)*
H32	0.768 (5)	1.0224 (17)	0.5952 (11)	0.076 (12)*
H41	−0.053 (3)	0.8120 (9)	0.625 (2)	0.053 (9)*
H42	−0.077 (3)	0.8988 (12)	0.640 (2)	0.055 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0216 (2)	0.0191 (2)	0.0214 (2)	−0.00144 (18)	0.00176 (17)	−0.00134 (17)
S2	0.0512 (4)	0.0395 (3)	0.0368 (3)	−0.0140 (3)	0.0236 (3)	−0.0079 (3)
O1	0.0277 (10)	0.0471 (11)	0.0919 (16)	0.0091 (8)	0.0034 (10)	0.0030 (11)
O2	0.0689 (13)	0.0263 (8)	0.0374 (9)	−0.0122 (8)	0.0146 (9)	0.0000 (7)
O3	0.0599 (13)	0.0547 (12)	0.0332 (9)	−0.0133 (10)	−0.0017 (9)	−0.0143 (8)
O1W	0.0280 (9)	0.0380 (10)	0.0478 (10)	−0.0030 (8)	−0.0066 (7)	0.0038 (8)
O2W	0.0292 (9)	0.0409 (9)	0.0332 (8)	−0.0024 (7)	−0.0026 (7)	0.0054 (7)
O3W	0.0383 (9)	0.0256 (8)	0.0327 (8)	−0.0050 (7)	0.0021 (7)	−0.0009 (7)
O4W	0.0325 (9)	0.0321 (9)	0.0413 (9)	−0.0017 (7)	0.0022 (7)	0.0032 (7)
N1	0.0268 (9)	0.0203 (8)	0.0198 (8)	−0.0010 (7)	0.0009 (6)	−0.0012 (6)
N2	0.0214 (9)	0.0385 (10)	0.0233 (8)	0.0043 (8)	0.0016 (7)	0.0029 (7)
C1	0.0504 (15)	0.0282 (11)	0.0295 (11)	−0.0031 (10)	0.0016 (10)	0.0086 (9)
C2	0.0273 (11)	0.0459 (13)	0.0294 (11)	−0.0015 (10)	−0.0028 (9)	−0.0093 (10)
C3	0.0276 (11)	0.0300 (11)	0.0334 (11)	0.0010 (9)	0.0073 (9)	−0.0009 (9)

C4	0.0390 (13)	0.0214 (10)	0.0326 (11)	−0.0012 (9)	0.0045 (9)	−0.0062 (8)
C5	0.0539 (17)	0.0598 (18)	0.0321 (12)	−0.0035 (14)	0.0000 (11)	−0.0122 (12)
C6	0.0370 (14)	0.0514 (16)	0.0482 (15)	0.0112 (12)	0.0110 (11)	0.0238 (13)
C7	0.0226 (11)	0.0459 (13)	0.0321 (11)	0.0077 (10)	−0.0022 (9)	0.0019 (10)
C8	0.0278 (12)	0.0495 (14)	0.0364 (12)	0.0050 (10)	0.0116 (9)	0.0074 (11)

Geometric parameters (Å, º)

S1—O3	1.4496 (18)	C1—H1C	0.9800
S1—O2	1.4631 (17)	C2—H2A	0.9800
S1—O1	1.4696 (19)	C2—H2B	0.9800
S1—S2	1.9970 (8)	C2—H2C	0.9800
O1W—H11	0.828 (9)	C3—H3A	0.9800
O1W—H12	0.834 (10)	C3—H3B	0.9800
O2W—H21	0.837 (10)	C3—H3C	0.9800
O2W—H22	0.841 (10)	C4—H4A	0.9800
O3W—H31	0.834 (10)	C4—H4B	0.9800
O3W—H32	0.835 (10)	C4—H4C	0.9800
O4W—H41	0.831 (10)	C5—H5A	0.9800
O4W—H42	0.832 (10)	C5—H5B	0.9800
N1—C2	1.488 (3)	C5—H5C	0.9800
N1—C1	1.489 (3)	C6—H6A	0.9800
N1—C3	1.493 (3)	C6—H6B	0.9800
N1—C4	1.498 (3)	C6—H6C	0.9800
N2—C6	1.490 (3)	C7—H7A	0.9800
N2—C8	1.493 (3)	C7—H7B	0.9800
N2—C7	1.494 (3)	C7—H7C	0.9800
N2—C5	1.494 (3)	C8—H8A	0.9800
C1—H1A	0.9800	C8—H8B	0.9800
C1—H1B	0.9800	C8—H8C	0.9800
O3—S1—O2	111.83 (11)	N1—C3—H3B	109.5
O3—S1—O1	109.87 (13)	H3A—C3—H3B	109.5
O2—S1—O1	108.00 (12)	N1—C3—H3C	109.5
O3—S1—S2	109.83 (9)	H3A—C3—H3C	109.5
O2—S1—S2	109.44 (8)	H3B—C3—H3C	109.5
O1—S1—S2	107.78 (10)	N1—C4—H4A	109.5
H11—O1W—H12	111.2 (16)	N1—C4—H4B	109.5
H21—O2W—H22	108.9 (16)	H4A—C4—H4B	109.5
H31—O3W—H32	110.5 (16)	N1—C4—H4C	109.5
H41—O4W—H42	111.3 (16)	H4A—C4—H4C	109.5
C2—N1—C1	109.68 (18)	H4B—C4—H4C	109.5
C2—N1—C3	109.39 (16)	N2—C5—H5A	109.5
C1—N1—C3	109.27 (17)	N2—C5—H5B	109.5
C2—N1—C4	109.40 (17)	H5A—C5—H5B	109.5
C1—N1—C4	109.96 (16)	N2—C5—H5C	109.5
C3—N1—C4	109.11 (17)	H5A—C5—H5C	109.5
C6—N2—C8	109.37 (19)	H5B—C5—H5C	109.5

C6—N2—C7	109.53 (19)	N2—C6—H6A	109.5
C8—N2—C7	109.12 (17)	N2—C6—H6B	109.5
C6—N2—C5	109.8 (2)	H6A—C6—H6B	109.5
C8—N2—C5	109.7 (2)	N2—C6—H6C	109.5
C7—N2—C5	109.33 (19)	H6A—C6—H6C	109.5
N1—C1—H1A	109.5	H6B—C6—H6C	109.5
N1—C1—H1B	109.5	N2—C7—H7A	109.5
H1A—C1—H1B	109.5	N2—C7—H7B	109.5
N1—C1—H1C	109.5	H7A—C7—H7B	109.5
H1A—C1—H1C	109.5	N2—C7—H7C	109.5
H1B—C1—H1C	109.5	H7A—C7—H7C	109.5
N1—C2—H2A	109.5	H7B—C7—H7C	109.5
N1—C2—H2B	109.5	N2—C8—H8A	109.5
H2A—C2—H2B	109.5	N2—C8—H8B	109.5
N1—C2—H2C	109.5	H8A—C8—H8B	109.5
H2A—C2—H2C	109.5	N2—C8—H8C	109.5
H2B—C2—H2C	109.5	H8A—C8—H8C	109.5
N1—C3—H3A	109.5	H8B—C8—H8C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H11···O1	0.83 (1)	1.88 (1)	2.706 (3)	174 (3)
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O2*W*—H22···O4*W*	0.84 (2)	1.90 (3)	2.736 (2)	173 (3)
O3*W*—H31···O2^{i}	0.83 (2)	1.93 (2)	2.759 (2)	172 (2)
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Symmetry codes: (i) $-x+3/2, y+1/2, -z+3/2$; (ii) $-x+1, -y+2, -z+1$; (iii) $x-1, y, z$.