Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tris(2-hydroxyethyl)ammonium 1,3-benzothiazole-2-thiolate

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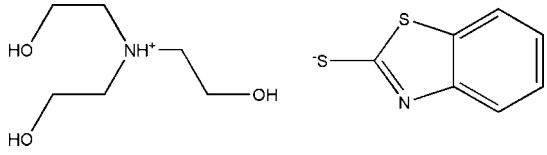
Received 21 May 2009; accepted 12 June 2009

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.092; data-to-parameter ratio = 15.3.

In the title compound, $C_6H_{16}NO_3^+\cdot C_7H_4NS_2^-$, the cations and anions are connected by O—H···N and O—H···S hydrogen bonding. Weak C—H···O hydrogen bonding between adjacent cations helps to stabilize the crystal structure.

Related literature

For related structures, see Bethge *et al.* (2008); Siracusa *et al.* (2008); Solar *et al.* (2008); Varlamov *et al.* (2005).



Experimental

Crystal data

$C_6H_{16}NO_3^+\cdot C_7H_4NS_2^-$
M_r = 316.43
Monoclinic, $P2_1/c$
a = 16.496 (2) Å
b = 5.7184 (8) Å
c = 17.462 (3) Å
β = 111.524 (2)°
V = 1532.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 296 K
0.56 × 0.38 × 0.23 mm

Data collection

Bruker SMART area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.803, T_{max} = 0.921
7572 measured reflections
2827 independent reflections
2185 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.092
S = 1.04
2827 reflections
185 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1^{i}	0.82	1.96	2.770 (2)	168
O2—H2···S2^{i}	0.82	2.43	3.2258 (17)	165
O3—H3···S2	0.82	2.35	3.1621 (16)	169
C8—H8A···O1^{ii}	0.97	2.50	3.385 (3)	151
C10—H10B···O3^{iii}	0.97	2.47	3.425 (3)	167

Symmetry codes: (i) $x, -y+\frac{5}{2}, z+\frac{1}{2}$; (ii) $x, y-1, z$; (iii) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The work was supported by the National Natural Science Foundation of China (No. 20772037) and the NSF of Guangdong Province, China (No. 06025033).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2531).

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supporting information

Acta Cryst. (2009). E**65**, o1640 [doi:10.1107/S160053680902248X]

Tris(2-hydroxyethyl)ammonium 1,3-benzothiazole-2-thiolate

Ji-Qin Zhu, Hua-Cai Fang, Bi-Yun Chen, Mao-Song Feng and Jing-Ning Li

S1. Comment

Some related compounds involving the 2-mercaptobenzothiazole and its derivatives has reported previously (Varlamov *et al.*, 2005; Solar *et al.*, 2008; Siracusa *et al.* 2008; Bethge *et al.*, 2008). The crystal structure of the title compound consists of tris(2-hydroxyethyl)ammonium cations and benzothiazole-2-thiolate anions (Fig. 1). The cations and anions are connected by O—H···N and O—H···S hydrogen bonding (Table 1).

S2. Experimental

A mixture of benzothiazole (335 mg, 2 mmol), triethanolamine (0.4 ml and 3 mmol) in ethyl acetate (20 ml) was refluxed for 20 h. The resultant yellow solution was delaminated into two layers at room temperature and then filtered. Single crystals suitable for X-ray diffraction were obtained in two day by slow diffusion of diethyl ether into a dilute solution of the title complex in ethyl acetate. The elemental analysis; calculated for $C_{13}H_{20}N_2O_3S_2$: C 49.37, H 6.33, N 8.86%; found: C 49.31, H 6.38, N 8.82%.

S3. Refinement

H atoms were placed in idealized positions with C—H = 0.93 or 0.97 Å, O—H = 0.82 Å, N—H = 0.91 Å, and refined in riding-model approximation. U_{iso}(H) = 1.5U_{eq}(O) and 1.2U_{eq}(N,*C*).

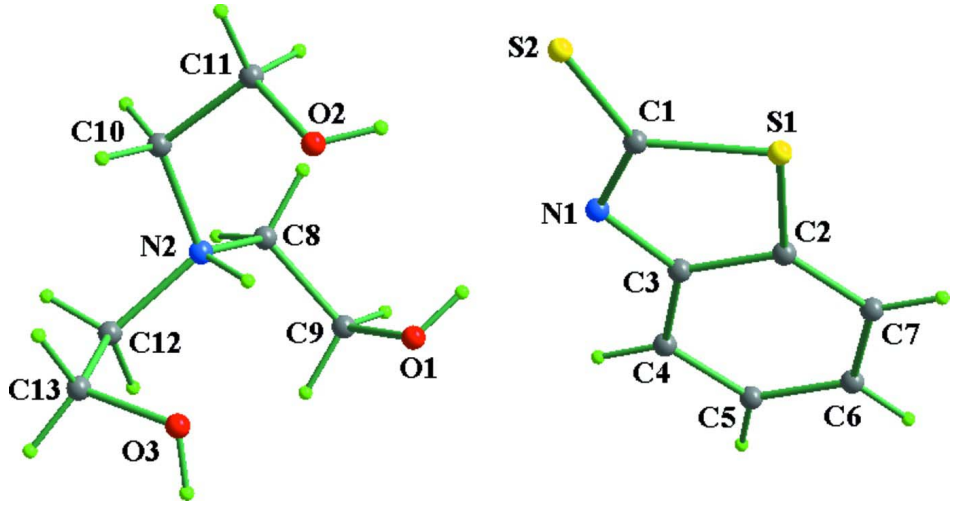

Figure 1

The structure of the title compound with 50% probability displacement ellipsoids.

Tris(2-hydroxyethyl)ammonium 1,3-benzothiazole-2-thiolate

Crystal data

$C_6H_{16}NO_3^+ \cdot C_7H_4NS_2^-$
$M_r = 316.43$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 16.496$ (2) Å
$b = 5.7184$ (8) Å
$c = 17.462$ (3) Å
$\beta = 111.524$ (2)°
$V = 1532.3$ (4) Å^3
$Z = 4$
$F(000) = 672$
$D_x = 1.372$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 236 reflections
$\theta = 2.4$–25.5°
$\mu = 0.36$ mm^{-1}
$T = 296$ K
Block, colorless
0.56 × 0.38 × 0.23 mm

Data collection

Bruker SMART area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.803$, $T_{max} = 0.921$
7572 measured reflections
2827 independent reflections
2185 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.4°$
$h = -19 \rightarrow 19$
$k = -6 \rightarrow 6$
$l = -20 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.092$
$S = 1.04$
2827 reflections
185 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2 + 0.2793P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.26675 (13)	1.1324 (4)	0.12815 (11)	0.0361 (5)
C2	0.39268 (13)	0.8624 (4)	0.15930 (11)	0.0347 (5)
C3	0.39516 (12)	1.0658 (3)	0.11586 (11)	0.0347 (5)
C4	0.46629 (14)	1.1048 (4)	0.09259 (13)	0.0459 (5)

H4	0.4692	1.2382	0.0632	0.055*
C5	0.53229 (15)	0.9413 (5)	0.11398 (14)	0.0521 (6)
H5	0.5803	0.9667	0.0992	0.062*
C6	0.52890 (15)	0.7410 (4)	0.15671 (13)	0.0498 (6)
H6	0.5743	0.6335	0.1699	0.060*
C7	0.45902 (14)	0.6983 (4)	0.18000 (12)	0.0445 (5)
H7	0.4565	0.5634	0.2088	0.053*
C8	0.25776 (13)	0.6974 (4)	0.44442 (12)	0.0412 (5)
H8A	0.2820	0.5472	0.4383	0.049*
H8B	0.2541	0.7025	0.4986	0.049*
C9	0.31648 (14)	0.8890 (4)	0.43727 (13)	0.0451 (5)
H9A	0.3726	0.8760	0.4819	0.054*
H9B	0.3257	0.8727	0.3858	0.054*
C10	0.09961 (15)	0.6154 (4)	0.40603 (14)	0.0501 (6)
H10A	0.1175	0.4593	0.4272	0.060*
H10B	0.0458	0.6019	0.3585	0.060*
C11	0.08354 (14)	0.7592 (4)	0.47092 (14)	0.0485 (6)
H11A	0.0323	0.7022	0.4798	0.058*
H11B	0.1330	0.7479	0.5224	0.058*
C12	0.16366 (15)	0.6302 (4)	0.29797 (12)	0.0477 (6)
H12A	0.1524	0.4633	0.2951	0.057*
H12B	0.2190	0.6556	0.2916	0.057*
C13	0.09228 (15)	0.7522 (5)	0.22968 (13)	0.0537 (6)
H13A	0.0969	0.7155	0.1772	0.064*
H13B	0.0359	0.6984	0.2282	0.064*
N1	0.32350 (11)	1.2134 (3)	0.09787 (10)	0.0371 (4)
N2	0.16844 (9)	0.7219 (3)	0.38008 (9)	0.0318 (4)
H2A	0.1569	0.8778	0.3736	0.038*
O1	0.28024 (11)	1.1097 (3)	0.44019 (9)	0.0515 (4)
H1	0.2898	1.1449	0.4882	0.077*
O2	0.07100 (11)	0.9936 (3)	0.44415 (9)	0.0581 (4)
H2	0.0927	1.0808	0.4837	0.087*
O3	0.09930 (10)	0.9968 (3)	0.24292 (9)	0.0533 (4)
H3	0.1244	1.0551	0.2148	0.080*
S1	0.29720 (3)	0.86166 (10)	0.17920 (3)	0.04177 (18)
S2	0.16918 (4)	1.25406 (11)	0.11908 (4)	0.04881 (19)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0443 (12)	0.0320 (12)	0.0297 (10)	−0.0048 (9)	0.0107 (9)	−0.0028 (9)
C2	0.0382 (11)	0.0340 (12)	0.0270 (9)	−0.0052 (9)	0.0063 (8)	−0.0007 (9)
C3	0.0405 (11)	0.0326 (11)	0.0289 (9)	−0.0054 (9)	0.0103 (8)	−0.0021 (9)
C4	0.0496 (13)	0.0463 (15)	0.0453 (12)	−0.0018 (11)	0.0215 (10)	0.0073 (11)
C5	0.0451 (13)	0.0620 (17)	0.0543 (14)	−0.0008 (11)	0.0245 (11)	−0.0012 (12)
C6	0.0465 (13)	0.0513 (16)	0.0478 (13)	0.0115 (11)	0.0128 (11)	−0.0004 (11)
C7	0.0509 (13)	0.0388 (13)	0.0379 (11)	0.0017 (10)	0.0094 (10)	0.0031 (10)
C8	0.0392 (12)	0.0404 (13)	0.0411 (11)	0.0054 (9)	0.0114 (9)	0.0034 (10)

C9	0.0449 (12)	0.0458 (14)	0.0457 (12)	−0.0063 (10)	0.0181 (10)	−0.0091 (11)
C10	0.0506 (13)	0.0500 (15)	0.0526 (13)	−0.0124 (11)	0.0225 (11)	−0.0010 (11)
C11	0.0449 (13)	0.0574 (16)	0.0485 (13)	−0.0021 (11)	0.0234 (11)	0.0049 (12)
C12	0.0649 (15)	0.0422 (14)	0.0375 (11)	0.0001 (11)	0.0204 (11)	−0.0070 (10)
C13	0.0533 (14)	0.0671 (18)	0.0342 (11)	−0.0093 (12)	0.0082 (10)	0.0001 (11)
N1	0.0436 (10)	0.0307 (10)	0.0375 (9)	−0.0019 (8)	0.0154 (8)	0.0030 (7)
N2	0.0351 (9)	0.0275 (9)	0.0319 (8)	0.0001 (7)	0.0110 (7)	0.0011 (7)
O1	0.0690 (10)	0.0380 (10)	0.0454 (8)	−0.0041 (8)	0.0186 (8)	−0.0074 (7)
O2	0.0705 (11)	0.0570 (12)	0.0490 (9)	0.0164 (9)	0.0244 (8)	0.0038 (8)
O3	0.0591 (10)	0.0590 (12)	0.0462 (9)	0.0122 (8)	0.0244 (7)	0.0144 (8)
S1	0.0457 (3)	0.0401 (3)	0.0402 (3)	−0.0032 (2)	0.0166 (2)	0.0092 (2)
S2	0.0518 (4)	0.0470 (4)	0.0536 (4)	0.0074 (3)	0.0263 (3)	0.0074 (3)

Geometric parameters (Å, º)

C1—N1	1.317 (2)	C9—H9A	0.9700
C1—S2	1.707 (2)	C9—H9B	0.9700
C1—S1	1.765 (2)	C10—N2	1.497 (2)
C2—C7	1.385 (3)	C10—C11	1.500 (3)
C2—C3	1.398 (3)	C10—H10A	0.9700
C2—S1	1.733 (2)	C10—H10B	0.9700
C3—N1	1.391 (2)	C11—O2	1.410 (3)
C3—C4	1.393 (3)	C11—H11A	0.9700
C4—C5	1.379 (3)	C11—H11B	0.9700
C4—H4	0.9300	C12—N2	1.501 (2)
C5—C6	1.379 (3)	C12—C13	1.505 (3)
C5—H5	0.9300	C12—H12A	0.9700
C6—C7	1.378 (3)	C12—H12B	0.9700
C6—H6	0.9300	C13—O3	1.415 (3)
C7—H7	0.9300	C13—H13A	0.9700
C8—N2	1.496 (2)	C13—H13B	0.9700
C8—C9	1.497 (3)	N2—H2A	0.9100
C8—H8A	0.9700	O1—H1	0.8200
C8—H8B	0.9700	O2—H2	0.8200
C9—O1	1.405 (3)	O3—H3	0.8200
N1—C1—S2	127.19 (16)	N2—C10—H10A	109.3
N1—C1—S1	113.51 (15)	C11—C10—H10A	109.3
S2—C1—S1	119.24 (11)	N2—C10—H10B	109.3
C7—C2—C3	121.86 (19)	C11—C10—H10B	109.3
C7—C2—S1	129.44 (16)	H10A—C10—H10B	107.9
C3—C2—S1	108.70 (15)	O2—C11—C10	108.40 (17)
N1—C3—C4	125.02 (18)	O2—C11—H11A	110.0
N1—C3—C2	115.84 (17)	C10—C11—H11A	110.0
C4—C3—C2	119.13 (19)	O2—C11—H11B	110.0
C5—C4—C3	118.6 (2)	C10—C11—H11B	110.0
C5—C4—H4	120.7	H11A—C11—H11B	108.4
C3—C4—H4	120.7	N2—C12—C13	110.28 (18)

C4—C5—C6	121.7 (2)	N2—C12—H12A	109.6
C4—C5—H5	119.2	C13—C12—H12A	109.6
C6—C5—H5	119.2	N2—C12—H12B	109.6
C7—C6—C5	120.7 (2)	C13—C12—H12B	109.6
C7—C6—H6	119.6	H12A—C12—H12B	108.1
C5—C6—H6	119.6	O3—C13—C12	109.59 (18)
C6—C7—C2	118.0 (2)	O3—C13—H13A	109.8
C6—C7—H7	121.0	C12—C13—H13A	109.8
C2—C7—H7	121.0	O3—C13—H13B	109.8
N2—C8—C9	110.98 (17)	C12—C13—H13B	109.8
N2—C8—H8A	109.4	H13A—C13—H13B	108.2
C9—C8—H8A	109.4	C1—N1—C3	111.49 (17)
N2—C8—H8B	109.4	C8—N2—C10	112.46 (15)
C9—C8—H8B	109.4	C8—N2—C12	112.13 (15)
H8A—C8—H8B	108.0	C10—N2—C12	111.54 (16)
O1—C9—C8	110.92 (17)	C8—N2—H2A	106.8
O1—C9—H9A	109.5	C10—N2—H2A	106.8
C8—C9—H9A	109.5	C12—N2—H2A	106.8
O1—C9—H9B	109.5	C9—O1—H1	109.5
C8—C9—H9B	109.5	C11—O2—H2	109.5
H9A—C9—H9B	108.0	C13—O3—H3	109.5
N2—C10—C11	111.68 (18)	C2—S1—C1	90.44 (9)

C7—C2—C3—N1	178.88 (18)	S2—C1—N1—C3	−178.71 (15)
S1—C2—C3—N1	−1.0 (2)	S1—C1—N1—C3	−1.5 (2)
C7—C2—C3—C4	0.1 (3)	C4—C3—N1—C1	−179.62 (19)
S1—C2—C3—C4	−179.79 (15)	C2—C3—N1—C1	1.7 (2)
N1—C3—C4—C5	−179.2 (2)	C9—C8—N2—C10	−153.34 (17)
C2—C3—C4—C5	−0.5 (3)	C9—C8—N2—C12	80.0 (2)
C3—C4—C5—C6	0.7 (3)	C11—C10—N2—C8	72.5 (2)
C4—C5—C6—C7	−0.4 (3)	C11—C10—N2—C12	−160.57 (18)
C5—C6—C7—C2	−0.1 (3)	C13—C12—N2—C8	−153.34 (18)
C3—C2—C7—C6	0.2 (3)	C13—C12—N2—C10	79.5 (2)
S1—C2—C7—C6	−179.94 (16)	C7—C2—S1—C1	−179.76 (19)
N2—C8—C9—O1	55.1 (2)	C3—C2—S1—C1	0.09 (14)
N2—C10—C11—O2	49.9 (2)	N1—C1—S1—C2	0.85 (15)
N2—C12—C13—O3	48.2 (2)	S2—C1—S1—C2	178.27 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1^{i}	0.82	1.96	2.770 (2)	168
O2—H2···S2^{i}	0.82	2.43	3.2258 (17)	165
O3—H3···S2	0.82	2.35	3.1621 (16)	169
C8—H8*A*···O1^{ii}	0.97	2.50	3.385 (3)	151
C10—H10*B*···O3^{iii}	0.97	2.47	3.425 (3)	167

Symmetry codes: (i) *x*, −*y*+5/2, *z*+1/2; (ii) *x*, *y*−1, *z*; (iii) −*x*, *y*−1/2, −*z*+1/2.