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2-Hydroxypropanammonium benzenethiolate

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The asymmetric unit of the title compund, $C_3H_{10}NO^+\cdot C_6H_5S^-$, contains two benzenothiolate anions and two 2-hydroxypropanammonium cations. The ions are linked into sheets of $R_2^4(8)$, $R_2^4(11)$ and $R_2^4(14)$ rings by a combination of N—H···S, O—H···S and N—H···O hydrogen bonds.

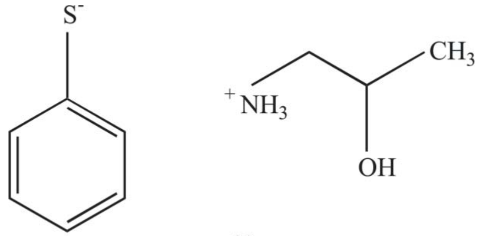
Key indicators

Single-crystal X-ray study
T = 200 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.084
Data-to-parameter ratio = 15.4

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

Comment

Hydrogen bonds of the N^+—H···S^- type have attracted little attention, because the transfer of a proton is seldom observed in such systems (Jetti *et al.*, 2004). An exception is found for silanethiolate compounds, where ionization of the S—H group is facilitated by the neighbouring Si atom (Baranowska, Chojnacki, Wojnowski & Becker, 2003). Reactions of alkoxysilanethiols with primary amines lead to tetrameric salts with a cubane-like core of a hydrogen-bonding net (Becker *et al.*, 2004). Reactions of tri-*tert*-butoxysilanethiol with secondary amines give derivatives with discrete dimeric units in the solid state (Baranowska, Chojnacki, Becker & Wojnowski, 2003; Baranowska *et al.*, 2006). In order to obtain further insight into these complex intermolecular interactions, the study of a bifunctional amine, namely 2-hydroxypropanamine, was undertaken, and we present here the crystal structure of the title compound, (I), containing this amine.



(I)

The asymmetric unit of (I) consists of two benzenothiolate anions and two 2-hydroxypropanammonium cations (Fig. 1). Each independent cation is linked to its centrosymmetric equivalent by N—H···S hydrogen bonds involving one of the independent symmetry-equivalent benzenothiolate anions (that with atom S1), forming an eight-membered ring leading to the graph-set motif $R_2^4(8)$ (Etter, 1990; Bernstein *et al.*, 1995). Similar (thiol–amine)$_2$ ring formation has been observed in other ammonium salts (Baranowska, Chojnacki, Wojnowski & Becker, 2003; Baranowska, Chojnacki, Becker & Wojnowski, 2003; Baranowska *et al.*, 2006).

These rings are further linked by N—H···S hydrogen bonds involving the other independent benzenothiolate anion (that

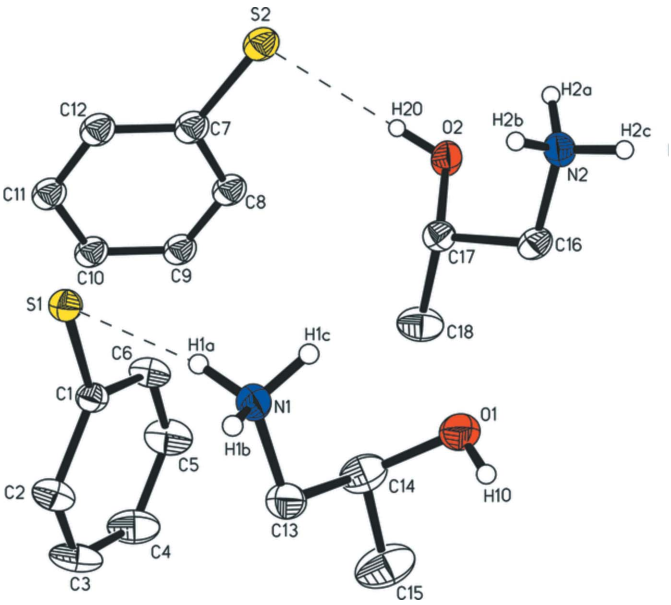


Figure 1
The asymmetric unit of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. C-bound H atoms have been omitted for clarity. Dashed lines indicate hydrogen bonds.

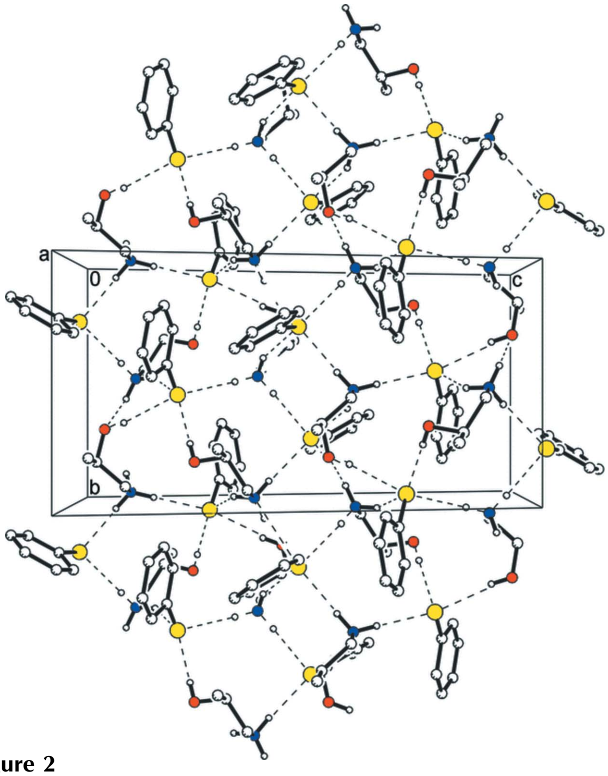


Figure 2
The crystal packing of (I), viewed approximately down the *a* axis. Hydrogen bonds are shown as dashed lines. C-bound H atoms have been omitted.

with atom S2), and by O—H···S and N—H···O hydrogen bonds, forming a sheet parallel to the $(10\overline{1})$ plane (Fig. 2). Application of graph theory to the sheet results in a variety of possible hydrogen-bonding patterns: the N1—H1*A*···S1, N2—H2*B*···S1^{ii}, O2—H2O···S2 and N1—H1*B*···S2^{ii} hydrogen bonds form an $R_2^4(11)$ ring, the N1—H1*B*···S2^{ii}, N2—H2*A*···S2^{iii}, O2—H2O···S2 and O1—H1O···S2^{i} hydrogen bonds form an $R_2^4(14)$ ring, and the N2—H2*C*···O1^{v}, N2—H2*C*···S1^{iv} and N1—H1*C*···S1^{ii} hydrogen bonds form an $R_2^2(7)$ ring, in which atom N2 acts as a bifurcated donor.

The N···S distances in (I) lie in the range 3.222 (2)–3.397 (2) Å, comparable with values observed in zinc and cobalt benzenethiolate complexes (Chung *et al.*, 1991*a*,*b*; Walters *et al.*, 1991), aromatic thiolates (Baranowska, Chojnacki, Becker & Wojnowski, 2003; Jetti *et al.*, 2004) or silanethiolates (Dołęga *et al.*, 2006).

Experimental

The synthesis was carried out using standard vacuum–nitrogen line and Schlenk techniques. The solvent and amine were purified and dried by standard methods (Perrin & Armarego, 1988). To a solution of benzenethiol (2 mmol) in 2-propanol (8 ml), 2-hydroxypropanamine (2 mmol) was added. Immediately, a white precipitate formed and this was dissolved by the addition of a portion of the solvent. The mixture was kept at 277 K for a few days, and colourless crystals of (I) suitable for X-ray diffraction were obtained. Elemental analysis for $C_9H_{16}N_1O_1S_1$ (185.28 g mol^{-1}), calculated: C 58.34, H 8.16, N 7.56, S 17.31%; found: C 58.25, H 8.23, N 7.56, S 17.14%.

Crystal data

$C_3H_{10}NO^+ \cdot C_6H_5S^-$
M_r = 185.28
Monoclinic, $P2_1/n$
a = 13.226 (3) Å
b = 9.182 (2) Å
c = 18.256 (4) Å
β = 106.68 (3)°
V = 2123.7 (9) Å^3
Z = 8
Mo *Kα* radiation
μ = 0.26 mm^{-1}
T = 200 (2) K
0.21 × 0.18 × 0.15 mm

Data collection

Stoe IPDS diffractometer
Absorption correction: none
14929 measured reflections
3762 independent reflections
2836 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.085
S = 0.97
3762 reflections
245 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O···S2^{i}	0.84	2.42	3.2379 (18)	166
N1—H1*A*···S1	0.91 (1)	2.34 (1)	3.2401 (18)	170 (2)
N1—H1*B*···S2^{ii}	0.91 (1)	2.31 (1)	3.2200 (19)	174 (2)
N1—H1*C*···S1^{ii}	0.90 (1)	2.46 (1)	3.2920 (18)	154 (2)
O2—H2O···S2	0.84	2.31	3.1530 (14)	178
N2—H2*A*···S2^{iii}	0.90 (1)	2.62 (1)	3.397 (2)	145 (2)
N2—H2*B*···S1^{ii}	0.90 (1)	2.35 (1)	3.2218 (17)	163 (2)
N2—H2*C*···S1^{iv}	0.90 (1)	2.47 (1)	3.2730 (17)	148 (2)
N2—H2*C*···O1^{v}	0.90 (1)	2.57 (2)	3.187 (2)	126 (2)

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

The ammonium H atoms were located in a difference map and refined with the N—H distances restrained to 0.90 (1) Å. The remaining H atoms were placed in idealized positions and allowed to ride on their parent atoms, with O—H = 0.84 Å, and C—H = 0.95–1.00 Å, and with $U_{iso}(H) = 1.2–1.5\ U_{eq}(C,O)$.

Data collection: *IPDS Software* (Stoe & Cie, 1999); cell refinement: *IPDS Software*; data reduction: *X-RED32* (Stoe & Cie, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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