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Dicyclohexylammonium benzenethiolate

Katarzyna Baranowska, Jaroslaw Chojnacki,* Barbara Becker and Wieslaw Wojnowski

Department of Chemistry, Technical University of Gdańsk 11/12, G. Narutowicz St., 80952-PL Gdańsk, Poland

Correspondence e-mail: jarekch@chem.pg.gda.pl

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Key indicators

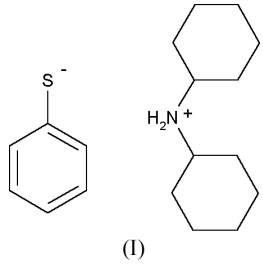
Single-crystal X-ray study
T = 200 K
Mean σ(C–C) = 0.008 Å
R factor = 0.039
wR factor = 0.144
Data-to-parameter ratio = 9.6

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

The title compound, $C_{12}H_{24}N^+\cdot C_6H_5S^-$, has been obtained by the reaction of C_6H_5SH with dicyclohexylamine. The two ionic fragments are linked together in long chains by $\cdots$N—H$\cdots$S$\cdots$ hydrogen bonds. There are two formula units in the asymmetric unit.

Comment

The title compound, (I), has been obtained previously in polycrystalline form (Dance, 1979). It was found to be a good isolable source of the benzenethiolate anion for the preparation of metal thiolates. In addition, there is a need for information in the study of N—H$\cdots$S bonding. Such bonding plays a crucial role in biological systems, *e.g.* in the modification of the redox potential in iron–sulfur proteins (Nakamura & Ueyama, 1988). Detailed research on N—H$\cdots$S hydrogen bonding in simple molecules is, therefore, important.



(I)

The asymmetric unit of (I) consists of two benzenethiolate anions and two dicyclohexylammonium cations. The ions are linked in zigzag chains by S$\cdots$H—N—H$\cdots$ hydrogen bonding, motif $C_2^1(4)$ (Etter, 1990). The donor–acceptor N$\cdots$S distances

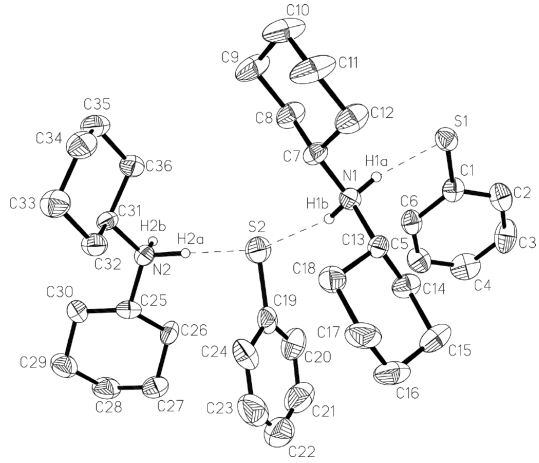


Figure 1
The asymmetric unit of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. All H atoms bonded to C atoms have been omitted.

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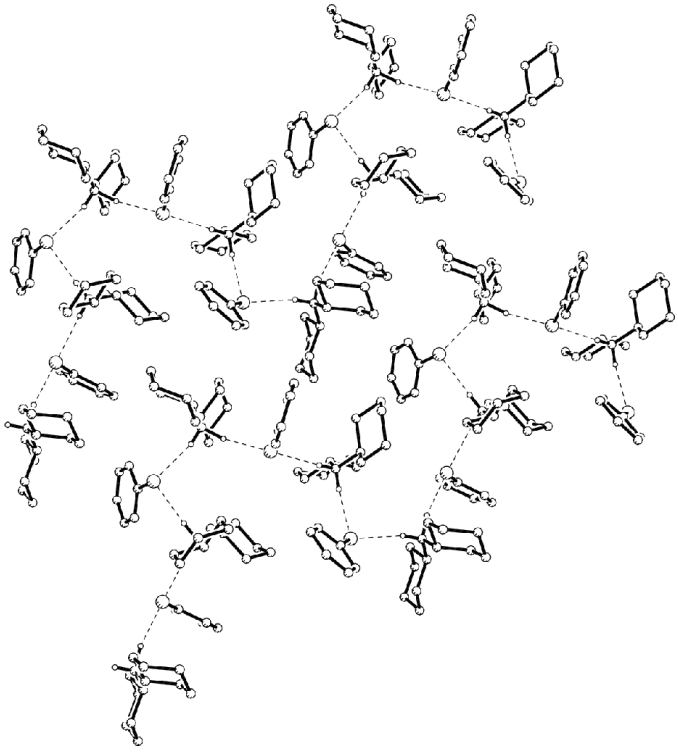

Figure 2
Diagram of the hydrogen-bond network.

[range 3.194 (5)–3.266 (5) Å] are comparable with the values observed in zinc and cobalt benzenethiolate complexes, $[(C_6H_{11})_2NH_2]_2[Zn_2(SC_6H_5)_6]$ (Chung *et al.*, 1991*a*) and $[(C_6H_{11})_2NH_2]_2[Co(SC_6H_5)_4]$ (Chung *et al.*, 1991*b*), and in $[(CH_3)_3NCH_2CONH_2][SC_6H_5]$ (Walters *et al.*, 1991).

Experimental

All procedures were carried out under argon, using standard Schlenk techniques. The solvent and the amine were dried by standard methods, and distilled under argon prior to use. To a solution of benzenethiol (0.22 g, 2 mmol) in acetonitrile, freshly prepared dicyclohexylamine (0.36 g, 2 mmol) was added. Immediately a white precipitate formed. Recrystallization from hot acetonitrile afforded colourless crystals of (I) suitable for X-ray diffraction analysis.

Crystal data

$C_{12}H_{24}N^+\cdot C_6H_5S^-$
M_r = 291.50
Orthorhombic, $Pca2_1$
a = 21.208 (4) Å
b = 11.678 (2) Å
c = 13.974 (3) Å
V = 3460.9 (12) Å^3
Z = 8
D_x = 1.119 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 40 reflections
θ = 2.8–18.7°
μ = 0.18 mm^{-1}
T = 200 (2) K
Plate, colourless
0.5 × 0.4 × 0.1 mm

Data collection

Oxford Diffraction KM-4 diffractometer
w-2θ scans
3593 measured reflections
3593 independent reflections
2159 reflections with $I > 2\sigma(I)$
θ_{max} = 26.4°
$h = -26 \rightarrow 0$
$k = -14 \rightarrow 0$
$l = 0 \rightarrow 17$
3 standard reflections
every 200 reflections
intensity decay: 0.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.144
S = 1.04
3593 reflections
373 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.079P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.032
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Absolute structure: Flack (1983), 33 Friedel pairs
Flack parameter = −0.06 (13)

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.762 (5)	N1—C7	1.497 (6)
S2—C19	1.755 (6)	N2—C25	1.490 (6)
N1—C13	1.498 (6)	N2—C31	1.507 (6)
C13—N1—C7	117.6 (4)	C13—N1—H1*B*	103 (3)
C13—N1—H1*A*	113 (3)	C7—N1—H1*B*	113 (3)
C7—N1—H1*A*	102 (3)	H1*A*—N1—H1*B*	109 (4)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···S1^{i}	0.92 (5)	2.29 (6)	3.200 (4)	170 (4)
N1—H1*B*···S2	0.93 (6)	2.37 (6)	3.266 (5)	163 (4)
N2—H2*B*···S1^{ii}	0.87 (6)	2.34 (6)	3.194 (5)	167 (5)
N2—H2*A*···S2^{iii}	1.11 (5)	2.15 (6)	3.246 (5)	166 (4)

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

H atoms bonded to C atoms were treated as riding, with fixed isotropic displacement parameters. The coordinates of the H atoms bonded to N were refined.

Data collection: *KM-4 System* (Gałdecki, Kowalski *et al.*, 1996); cell refinement: *KM-4 System*; data reduction: *DATAPROC* (Gałdecki, Kowalski & Uszyński, 1996); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *RESVIEW* (Schwenk, 1998); software used to prepare material for publication: *SHELXL*97.

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