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2,2′-(1,2-Ethanediyldithio)bis(1,3-benzothiazole)

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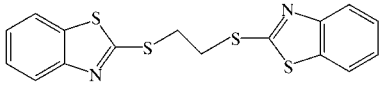
In the title compound, $C_{16}H_{12}N_2S_4$, which is the result of the S-alkylation reaction of 2-mercaptobenzothiazole with ethylene dibromide, the planes of the two benzothiazole moieties form a dihedral angle of 3.84 (14)°. The bridging chain moiety, $-SCH_2CH_2S-$, adopts an antiperiplanar conformation. There are intermolecular S···S non-bonded contacts of 3.6471 (9) Å, which stabilize the crystal packing.

Comment

Acyclic crown ethers with N-heterocycle end groups that will coordinate and transport metals have been of interest for several years (Vögtle & Weber, 1979; Meth-Cohn & Smith, 1982; Liu *et al.*, 1992; Matthews *et al.*, 1996). Bis(benzothiazole) compounds have been studied as potential mimics for metalloproteins (Thompson *et al.*, 1982) and bleomycin (Kane *et al.*, 1995). We have recently reported the synthesis and characterization of acyclic bis(benzothiazole) crown ethers (Liu *et al.*, 2001). We report here the X-ray crystal structure of the title compound, (I).



(I)

In the molecular structure of (I) (Fig. 1), the two benzothiazole moieties form a dihedral angle of 3.84 (14)°. The two thiazole moieties, like those of bis(benzothiazole-2-ylsulfanyl)methane (Matthews *et al.*, 1996) and bis(2-benzothiazolyl)disulfane (Zingaro & Meyers, 1980), are *anti* to each other. The bridging chain moiety, $-SCH_2CH_2S-$, adopts an antiperiplanar conformation. The molecule is approximately centrosymmetric. The bond lengths and angles in (I) show normal values (Table 1).

The crystal packing of (I) is shown in Fig. 2. As seen in bis(benzothiazole-2-ylsulfanyl)methane (Matthews *et al.*, 1996) and bis(2-benzothiazolyl)disulfane (Zingaro & Meyers, 1980), there are non-bonded intermolecular S1···S4($\frac{1}{2}+x$, $\frac{1}{2}-y$, $z-\frac{1}{2}$) contacts of 3.6471 (9) Å. These S···S contacts and crosslinking interactions stabilize the crystal packing.

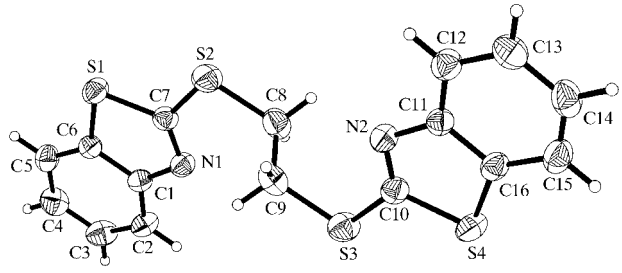


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms are shown as small spheres of arbitrary radii.

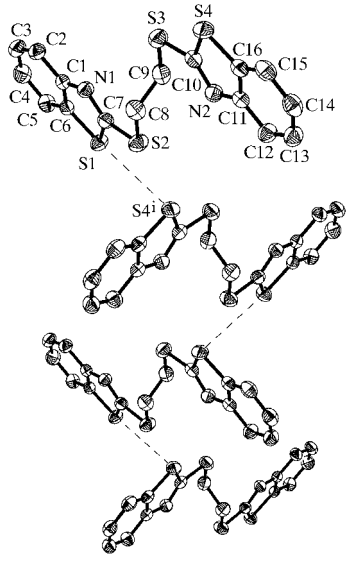


Figure 2
The two-dimensional crystal packing of (I). [Symmetry code: (i) $\frac{1}{2}+x$, $\frac{3}{2}-y$, $-\frac{1}{2}+z$.]

Experimental

The title compound was prepared *via* the S-alkylation reaction of 2-mercaptobenzothiazole with ethylene dibromide in acetone solution using potassium carbonate as base. Single crystals of (I) (m.p. 422–423 K) suitable for X-ray diffraction were obtained by slow evaporation of a solution in acetone.

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Crystal data

$C_{16}H_{12}N_2S_4$
M_r = 360.52
Monoclinic, $P2_1/n$
a = 11.513 (2) Å
b = 10.711 (2) Å
c = 13.998 (2) Å
β = 114.10 (1)°
V = 1575.7 (5) Å^3
Z = 4
D_x = 1.520 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 29 reflections
θ = 2.7–14.8°
μ = 0.60 mm^{-1}
T = 296 (2) K
Plate, colourless
0.48 × 0.44 × 0.40 mm

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: empirical (*SHELXS*86; Sheldrick, 1990)
T_{min} = 0.748, T_{max} = 0.787
3203 measured reflections
2779 independent reflections
2095 reflections with $I > 2\sigma(I)$
R_{int} = 0.010
θ_{max} = 25°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 12$
$l = -16 \rightarrow 15$
3 standard reflections
every 97 reflections
intensity decay: 6.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.080
S = 1.06
2779 reflections
200 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0419P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 1997)
Extinction coefficient: 0.0095 (9)

The positions of all H atoms were fixed geometrically, with C—H distances in the range 0.93–0.97 Å.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Table 1
Selected geometric parameters (Å, °).

S1—C6	1.734 (2)	S4—C16	1.730 (2)
S1—C7	1.753 (2)	S4—C10	1.750 (2)
S2—C7	1.742 (2)	N1—C7	1.297 (2)
S2—C8	1.818 (2)	N1—C1	1.396 (2)
S3—C10	1.744 (2)	N2—C10	1.294 (3)
S3—C9	1.822 (2)	N2—C11	1.398 (2)
C7—S2—C8	100.75 (10)	C9—C8—S2	112.22 (16)
C10—S3—C9	101.14 (10)	C8—C9—S3	112.23 (15)
C8—S2—C7—N1	−0.8 (2)	C10—S3—C9—C8	84.38 (17)
C8—S2—C7—S1	−179.38 (12)	C9—S3—C10—N2	−3.7 (2)
C7—S2—C8—C9	−82.90 (18)	C9—S3—C10—S4	175.63 (12)
S2—C8—C9—S3	178.53 (11)		

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OB1097). Services for accessing these data are described at the back of the journal.

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