

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Bis[2-(1,3-benzothiazol-2-ylsulfanyl)-ethyl] ether

Hui-Guo Chen,^a Xiao-Feng Li,^b Yan An,^b Li-Hui Yao^c and Wei-Sheng Liu^{a*}

^aKey Laboratory of Nonferrous Metal Chemistry and Resources Utilization of Gansu Province, College of Chemistry and Chemical Engineering, Lanzhou University, Lanzhou 730000, People's Republic of China, ^bInstitute of Marine Material and Engineering, Shanghai Maritime University, Shanghai 200135, People's Republic of China, and ^cCollege of Chemistry and Chemical Engineering, Lanzhou University, Lanzhou 730000, People's Republic of China

Correspondence e-mail: liuws@lzu.edu.cn

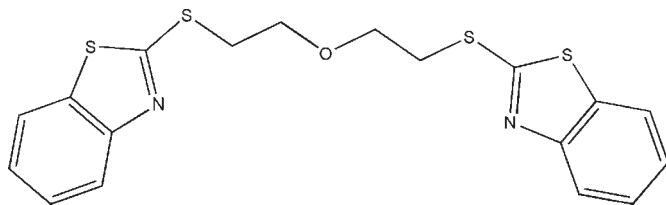
Received 10 November 2009; accepted 5 December 2009

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.043; wR factor = 0.129; data-to-parameter ratio = 14.8.

The complete molecule of title compound, $\text{C}_{18}\text{H}_{16}\text{N}_2\text{OS}_4$, is generated by crystallographic twofold symmetry, with the O atom lying on the rotation axis. The dihedral angle between the ring systems is $80.91(2)^\circ$. In the crystal, adjacent molecules are connected through π - π stacking interactions [centroid-centroid distance = $3.882(2)$ Å], forming a three-dimensional network.

Related literature

For coordination polymers in supramolecular chemistry and crystal engineering, see: Robinson & Zaworotko (1995); Yaghi & Li (1996); Fujita *et al.* (1995); Tong *et al.* (2000); Bu *et al.* (2003); Long *et al.* (2004); Massue *et al.* (2007); Zou *et al.* (2004).



Experimental

Crystal data

$\text{C}_{18}\text{H}_{16}\text{N}_2\text{OS}_4$
 $M_r = 404.57$
 Monoclinic, $C2/c$
 $a = 24.617(3)$ Å

$b = 4.7085(3)$ Å
 $c = 17.7866(15)$ Å
 $\beta = 116.571(13)^\circ$
 $V = 1843.9(3)$ Å³

$Z = 4$
 Cu $K\alpha$ radiation
 $\mu = 4.81$ mm⁻¹

$T = 293$ K
 $0.18 \times 0.15 \times 0.07$ mm

Data collection

Oxford Diffraction Xcalibur
 Sapphire3 diffractometer
 Absorption correction: multi-scan
 (*CrysAlis RED*; Oxford
 Diffraction, 2005)
 $T_{\min} = 0.765$, $T_{\max} = 1.000$

2930 measured reflections
 1682 independent reflections
 1353 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.129$
 $S = 1.05$
 1682 reflections

114 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.27$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

The authors acknowledge the NSFC (Grant Nos. 20771048, 20931003), the Project of Shanghai Municipal Education Commission (2008080, 2008068, 09YZ245, 10YZ111, 10ZZ98), the 'Chen Guang' project supported by Shanghai Municipal Education Commission and Shanghai Education Development Foundation (09 C G52), the Innovative Activities of University Students in Shanghai Maritime University Project (090503) and the State Key Laboratory of Pollution Control and Resource Reuse Foundation (PCRRF09001) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2684).

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supplementary materials

Acta Cryst. (2010). E66, o125 [doi:10.1107/S1600536809052301]

Bis[2-(1,3-benzothiazol-2-ylsulfanyl)ethyl] ether

H.-G. Chen, X.-F. Li, Y. An, L.-H. Yao and W.-S. Liu

Comment

Ligands containing thioether and nitrogenous heterocyclic groups are well established sources for biologically active complexes. In addition, this kind of ligands may form one- or multi-dimensional supramolecular structures *via* the intermolecular interactions such as hydrogen-bond or π - π stacking, attracting intense attention in the field of supramolecular chemistry and crystal engineering (Robinson *et al.*, 1995; Yaghi *et al.*, 1996; Fujita *et al.*, 1995; Tong *et al.*, 2000).

Herein, we report the synthesis and structure of the title compound, namely bis[2-(benzothiazol-2-ylthio)ethyl]ether (Fig. 1). As shown in Fig. 2, a two-dimensional supramolecular network was formed by hydrogen bonds (Table 2) [Symmetry codes (i): $x, -y + 1, z + 1/2$] and S—S bonds of 3.575 (2) Å [Symmetry codes (ii): $-x + 1/2, -y + 3/2, -z + 2$], and there are also weak π - π stacking interactions between the phenyl rings and the thiazolyl rings of adjacent molecules with a centroid-centroid distances of 3.882 Å along b direction.

Experimental

Bis(2-chloroethyl)ether (0.02 mol, 2.86 g) was added dropwise to a hot mixture solution (353 K) of 2-mercaptobenzothiazole (0.04 mol, 6.69 g), KOH (0.04 mol, 2.24 g) in ethanol (100 ml), and the mixture was further stirred at 353 K for 15 h. After cooling, the precipitate was filtered, washed with ethanol and water, and recrystallized from ethanol to obtain white powder. Yield: 56% (Bu *et al.*, 2003; Massue *et al.*, 2007; Long *et al.*, 2004). ^1H NMR (CDCl_3 , 400 MHz): 3.56 (t, 4H), 3.89 (t, 4H), 7.25 (m, 2H), 7.39 (m, 2H), 7.70 (d, 2H), 7.82 (t, 2H). MS (ESI) m/z (%): 405.0 (M^{+1}).

Refinement

The H atoms were placed at calculated positions in the riding model approximation (C—H 0.93 Å), with their temperature factors were set to 1.2 times those of the equivalent isotropic temperature factors of the parent atoms.

Figures

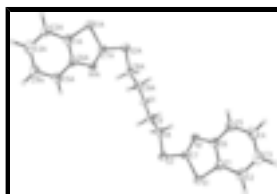


Fig. 1. The structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.



Fig. 2. The three-dimensional structure by molecular packing, showing the hydrogen bonds as blue dashed lines [Symmetry codes: (i) $x, -y + 1, z + 1/2$], S—S bonds as green dashed lines and π - π stacking interactions as red dashed lines.

Bis[2-(1,3-benzothiazol-2-ylsulfanyl)ethyl] ether

Crystal data

$C_{18}H_{16}N_2OS_4$

$M_r = 404.57$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 24.617\ (3)\ \text{\AA}$

$b = 4.7085\ (3)\ \text{\AA}$

$c = 17.7866\ (15)\ \text{\AA}$

$\beta = 116.571\ (13)^\circ$

$V = 1843.9\ (3)\ \text{\AA}^3$

$Z = 4$

$F(000) = 840$

$D_x = 1.457\ \text{Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54178\ \text{\AA}$

Cell parameters from 1595 reflections

$\theta = 2.8\text{--}68.1^\circ$

$\mu = 4.81\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, colourless

$0.18 \times 0.15 \times 0.07\ \text{mm}$

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer

Radiation source: Enhance (Cu) X-ray Source graphite

Detector resolution: $16.0855\ \text{pixels mm}^{-1}$

ω scans

Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2005)

$T_{\min} = 0.765$, $T_{\max} = 1.000$

2930 measured reflections

1682 independent reflections

1353 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\max} = 68.1^\circ$, $\theta_{\min} = 2.8^\circ$

$h = -29 \rightarrow 25$

$k = -5 \rightarrow 3$

$l = -21 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.129$

$S = 1.05$

1682 reflections

114 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0876P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.27\ \text{e \AA}^{-3}$

0 restraints

$$\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.17154 (13)	0.1689 (5)	1.11310 (16)	0.0505 (6)
C2	0.18584 (15)	-0.0249 (6)	1.17798 (17)	0.0608 (7)
H2	0.2256	-0.0860	1.2098	0.073*
C3	0.13924 (18)	-0.1235 (7)	1.1936 (2)	0.0701 (8)
H3	0.1474	-0.2568	1.2359	0.084*
C4	0.08011 (16)	-0.0269 (8)	1.1470 (2)	0.0702 (8)
H4	0.0494	-0.0954	1.1591	0.084*
C5	0.06625 (15)	0.1676 (6)	1.0837 (2)	0.0614 (7)
H5	0.0266	0.2328	1.0534	0.074*
C6	0.11236 (12)	0.2665 (6)	1.06519 (15)	0.0489 (6)
C7	0.15597 (12)	0.4948 (6)	0.99910 (14)	0.0483 (6)
C8	0.09486 (13)	0.8627 (6)	0.86818 (16)	0.0532 (6)
H8C	0.0791	0.9199	0.9070	0.064*
H8B	0.0995	1.0323	0.8406	0.064*
C9	0.05001 (13)	0.6678 (5)	0.80325 (17)	0.0552 (6)
H9A	0.0683	0.5769	0.7712	0.066*
H9B	0.0370	0.5215	0.8300	0.066*
N1	0.10483 (10)	0.4522 (5)	1.00023 (13)	0.0507 (5)
S1	0.21965 (3)	0.32443 (16)	1.07717 (4)	0.0560 (2)
S2	0.16852 (3)	0.69930 (17)	0.92661 (4)	0.0592 (3)
O1	0.0000	0.8363 (5)	0.7500	0.0501 (6)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0535 (14)	0.0516 (13)	0.0422 (12)	-0.0026 (11)	0.0176 (11)	-0.0037 (10)
C2	0.0671 (18)	0.0625 (16)	0.0465 (14)	0.0052 (13)	0.0197 (13)	0.0058 (12)
C3	0.094 (2)	0.0659 (17)	0.0546 (16)	-0.0017 (16)	0.0364 (17)	0.0077 (13)
C4	0.081 (2)	0.076 (2)	0.0661 (18)	-0.0114 (16)	0.0442 (17)	-0.0024 (15)
C5	0.0579 (16)	0.0686 (18)	0.0606 (17)	-0.0045 (13)	0.0290 (14)	-0.0061 (13)
C6	0.0530 (15)	0.0504 (13)	0.0395 (12)	-0.0021 (11)	0.0171 (11)	-0.0063 (10)

supplementary materials

C7	0.0454 (13)	0.0524 (13)	0.0387 (11)	-0.0023 (10)	0.0112 (10)	-0.0007 (10)
C8	0.0561 (15)	0.0484 (13)	0.0442 (13)	0.0012 (11)	0.0127 (11)	0.0024 (10)
C9	0.0528 (15)	0.0482 (13)	0.0482 (13)	0.0051 (11)	0.0078 (12)	-0.0003 (11)
N1	0.0460 (11)	0.0559 (12)	0.0427 (10)	-0.0030 (9)	0.0132 (9)	-0.0023 (9)
S1	0.0438 (4)	0.0679 (5)	0.0484 (4)	0.0033 (3)	0.0136 (3)	0.0090 (3)
S2	0.0450 (4)	0.0713 (5)	0.0519 (4)	-0.0050 (3)	0.0132 (3)	0.0131 (3)
O1	0.0485 (14)	0.0462 (13)	0.0436 (12)	0.000	0.0099 (11)	0.000

Geometric parameters (Å, °)

C1—C2	1.387 (4)	C7—N1	1.284 (4)
C1—C6	1.396 (4)	C7—S2	1.744 (3)
C1—S1	1.739 (3)	C7—S1	1.755 (3)
C2—C3	1.376 (5)	C8—C9	1.501 (4)
C2—H2	0.9300	C8—S2	1.810 (3)
C3—C4	1.390 (5)	C8—H8C	0.9700
C3—H3	0.9300	C8—H8B	0.9700
C4—C5	1.372 (5)	C9—O1	1.414 (3)
C4—H4	0.9300	C9—H9A	0.9700
C5—C6	1.395 (4)	C9—H9B	0.9700
C5—H5	0.9300	O1—C9 ⁱ	1.414 (3)
C6—N1	1.394 (4)		
C2—C1—C6	122.1 (3)	N1—C7—S1	116.8 (2)
C2—C1—S1	128.4 (2)	S2—C7—S1	116.50 (15)
C6—C1—S1	109.5 (2)	C9—C8—S2	112.65 (19)
C3—C2—C1	117.7 (3)	C9—C8—H8C	109.1
C3—C2—H2	121.1	S2—C8—H8C	109.1
C1—C2—H2	121.1	C9—C8—H8B	109.1
C2—C3—C4	121.0 (3)	S2—C8—H8B	109.1
C2—C3—H3	119.5	H8C—C8—H8B	107.8
C4—C3—H3	119.5	O1—C9—C8	107.0 (2)
C5—C4—C3	121.1 (3)	O1—C9—H9A	110.3
C5—C4—H4	119.4	C8—C9—H9A	110.3
C3—C4—H4	119.4	O1—C9—H9B	110.3
C4—C5—C6	119.1 (3)	C8—C9—H9B	110.3
C4—C5—H5	120.4	H9A—C9—H9B	108.6
C6—C5—H5	120.4	C7—N1—C6	110.1 (2)
N1—C6—C5	125.7 (2)	C1—S1—C7	88.23 (13)
N1—C6—C1	115.3 (2)	C7—S2—C8	101.30 (13)
C5—C6—C1	118.9 (3)	C9—O1—C9 ⁱ	111.7 (3)
N1—C7—S2	126.7 (2)		

Symmetry codes: (i) $-x, y, -z+3/2$.

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4—H4 ⁱⁱ —O1 ⁱⁱ	0.93	2.71	3.358 (3)	128.

Symmetry codes: (ii) $x, -y+1, z+1/2$.

Fig. 1

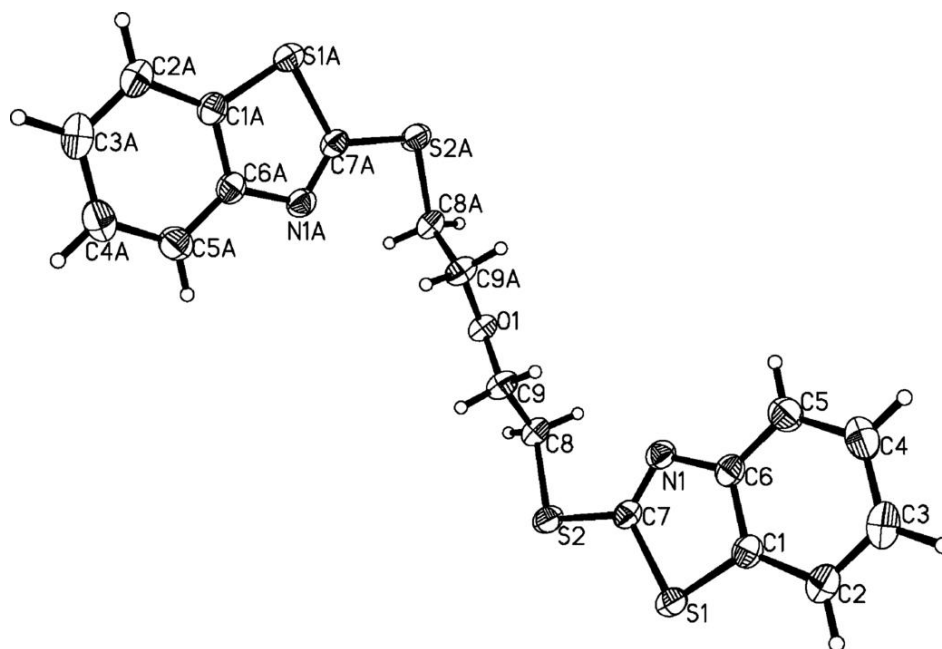


Fig. 2

