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2,5-Bis[(3-chlorobenzyl)sulfanyl]-1,3,4-thiadiazole

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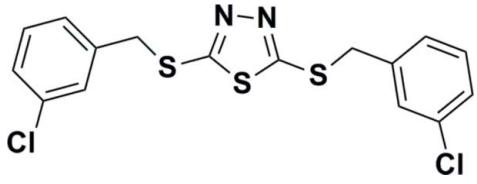
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.024; wR factor = 0.063; data-to-parameter ratio = 14.2.

The complete molecule of the title compound, $C_{16}H_{12}Cl_2N_2S_3$, is generated by crystallographic twofold symmetry, with the S atom of the thiadiazole ring lying on the rotation axis. The dihedral angle between the mean planes of the 1,3,4-thiadiazole and benzene rings is 87.19 (7)°. In the crystal, molecules are linked by C—H···N interactions and short S···S contacts [3.3389 (9) Å] occur.

Related literature

For details of the synthesis, see: Liu *et al.* (2012); Tan *et al.* (2012). For a related structure, see: Liu & Liu (2011. For the biological activity of related compounds, see: Liu *et al.* (2011*a*,*b*).



Experimental

Crystal data

$C_{16}H_{12}Cl_2N_2S_3$
M_r = 399.36
Monoclinic, $C2/c$
a = 17.200 (3) Å
b = 5.6604 (11) Å
c = 17.524 (4) Å
β = 92.56 (3)°
V = 1704.4 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.75 mm^{-1}
T = 113 K
0.12 × 0.10 × 0.06 mm

Data collection

Rigaku Saturn CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.916, T_{max} = 0.957
5343 measured reflections
1491 independent reflections
1382 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.063
S = 1.17
1491 reflections
105 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···N1^{i}	0.93	2.59	3.514 (2)	172

Symmetry code: (i) $x+\frac{1}{2}, y+\frac{1}{2}, z$.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear* (Rigaku/MSC, 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6758).

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supporting information

Acta Cryst. (2012). E**68**, o1620 [doi:10.1107/S1600536812019150]

2,5-Bis[(3-chlorobenzyl)sulfanyl]-1,3,4-thiadiazole

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S1. Experimental

To a stirred solution of 1,3,4-thiadiazole-2,5-dithiol (5.1 mmol) and potassium carbonate (5.6 mmol) in DMF (15 ml), 1-chloro-3-(chloromethyl)benzene (5.6 mmol) was added dropwise. The resulting mixture was stirred at room temperature overnight. The mixture was poured into water, the precipitate formed was filtered off and recrystallized from acetone to give title compound in good yields. Compound was dissolved in hot alcohol and the resulting solution was allowed to stand in air at room temperature to colourless blocks of the title compound.

S2. Refinement

All the H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

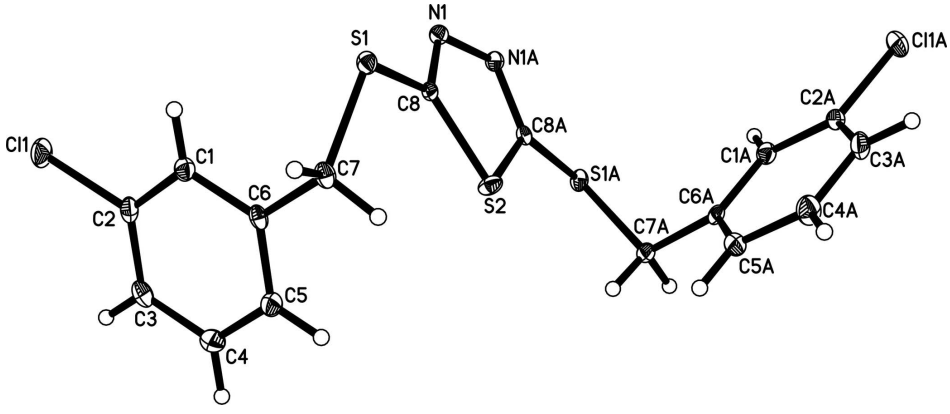

Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

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Crystal data

$C_{16}H_{12}Cl_2N_2S_3$
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Monoclinic, *C*2/*c*
a = 17.200 (3) Å
b = 5.6604 (11) Å
c = 17.524 (4) Å
β = 92.56 (3)°
V = 1704.4 (6) Å^3
Z = 4
F(000) = 816
D_x = 1.556 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 2634 reflections
θ = 3.3–27.9°
μ = 0.75 mm^{-1}
T = 113 K
Block, colorless
0.12 × 0.10 × 0.06 mm

Data collection

Rigaku Saturn CCD diffractometer
Radiation source: rotating anode
Confocal monochromator
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.916, T_{max} = 0.957
5343 measured reflections
1491 independent reflections
1382 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 25.0°, θ_{min} = 3.3°
h = −20→20
k = −6→6
l = −14→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.063
S = 1.17
1491 reflections
105 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
$w = 1/[\sigma^2(F_o^2) + (0.0254P)^2 + 1.1171P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
S1	0.03445 (2)	−0.05340 (7)	0.41412 (2)	0.01769 (13)
S2	0.0000	0.13382 (10)	0.2500	0.01979 (16)
Cl1	0.36343 (2)	−0.14225 (8)	0.42335 (3)	0.03039 (14)
N1	0.01164 (7)	−0.3031 (2)	0.28829 (7)	0.0161 (3)
C1	0.22023 (9)	0.0583 (3)	0.41728 (9)	0.0198 (3)
H1	0.2061	−0.0599	0.4508	0.024*
C2	0.29495 (9)	0.0682 (3)	0.39189 (9)	0.0206 (4)
C3	0.31795 (9)	0.2422 (3)	0.34242 (9)	0.0255 (4)
H3	0.3687	0.2464	0.3263	0.031*
C4	0.26429 (10)	0.4097 (3)	0.31736 (10)	0.0284 (4)
H4	0.2789	0.5278	0.2840	0.034*
C5	0.18844 (10)	0.4023 (3)	0.34188 (9)	0.0232 (4)
H5	0.1525	0.5150	0.3245	0.028*
C6	0.16605 (9)	0.2277 (3)	0.39205 (8)	0.0171 (3)
C7	0.08449 (9)	0.2289 (3)	0.42062 (9)	0.0190 (3)
H7A	0.0540	0.3448	0.3916	0.023*

H7B	0.0869	0.2793	0.4736	0.023*
C8	0.01873 (8)	−0.0905 (3)	0.31589 (8)	0.0136 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0171 (2)	0.0229 (2)	0.0129 (2)	−0.00199 (16)	−0.00114 (15)	0.00075 (15)
S2	0.0314 (3)	0.0127 (3)	0.0149 (3)	0.000	−0.0042 (2)	0.000
Cl1	0.0197 (2)	0.0303 (3)	0.0404 (3)	0.00735 (18)	−0.00680 (18)	0.00062 (19)
N1	0.0158 (6)	0.0171 (7)	0.0153 (6)	−0.0004 (6)	0.0002 (5)	0.0007 (5)
C1	0.0191 (8)	0.0213 (8)	0.0187 (8)	−0.0008 (7)	−0.0024 (6)	−0.0009 (7)
C2	0.0169 (8)	0.0213 (8)	0.0230 (8)	0.0029 (7)	−0.0063 (6)	−0.0050 (7)
C3	0.0143 (8)	0.0314 (10)	0.0308 (9)	−0.0037 (7)	0.0006 (7)	−0.0027 (8)
C4	0.0230 (10)	0.0274 (10)	0.0348 (10)	−0.0044 (8)	0.0018 (7)	0.0074 (8)
C5	0.0198 (9)	0.0204 (9)	0.0289 (9)	0.0020 (7)	−0.0031 (7)	0.0004 (7)
C6	0.0153 (8)	0.0201 (8)	0.0156 (7)	−0.0020 (7)	−0.0030 (6)	−0.0070 (6)
C7	0.0166 (8)	0.0214 (8)	0.0190 (8)	−0.0002 (7)	−0.0009 (6)	−0.0061 (7)
C8	0.0096 (7)	0.0164 (8)	0.0149 (8)	0.0001 (6)	0.0006 (6)	0.0018 (6)

Geometric parameters (Å, º)

S1—C8	1.7432 (15)	C2—C3	1.382 (2)
S1—C7	1.8161 (17)	C3—C4	1.381 (2)
S2—C8^{i}	1.7367 (15)	C3—H3	0.9300
S2—C8	1.7368 (15)	C4—C5	1.392 (2)
Cl1—C2	1.7471 (17)	C4—H4	0.9300
N1—C8	1.301 (2)	C5—C6	1.389 (2)
N1—N1^{i}	1.383 (2)	C5—H5	0.9300
C1—C2	1.380 (2)	C6—C7	1.511 (2)
C1—C6	1.395 (2)	C7—H7A	0.9700
C1—H1	0.9300	C7—H7B	0.9700
C8—S1—C7	102.72 (7)	C6—C5—C4	120.47 (16)
C8^{i}—S2—C8	86.03 (10)	C6—C5—H5	119.8
C8—N1—N1^{i}	112.28 (8)	C4—C5—H5	119.8
C2—C1—C6	119.25 (15)	C5—C6—C1	119.31 (15)
C2—C1—H1	120.4	C5—C6—C7	119.64 (15)
C6—C1—H1	120.4	C1—C6—C7	121.02 (14)
C1—C2—C3	121.89 (15)	C6—C7—S1	114.82 (11)
C1—C2—Cl1	119.68 (13)	C6—C7—H7A	108.6
C3—C2—Cl1	118.43 (13)	S1—C7—H7A	108.6
C4—C3—C2	118.87 (15)	C6—C7—H7B	108.6
C4—C3—H3	120.6	S1—C7—H7B	108.6
C2—C3—H3	120.6	H7A—C7—H7B	107.5
C3—C4—C5	120.22 (16)	N1—C8—S2	114.69 (11)
C3—C4—H4	119.9	N1—C8—S1	119.12 (11)
C5—C4—H4	119.9	S2—C8—S1	125.76 (9)

C6—C1—C2—C3	0.4 (2)	C5—C6—C7—S1	131.52 (14)
C6—C1—C2—Cl1	179.77 (12)	C1—C6—C7—S1	−50.67 (18)
C1—C2—C3—C4	−0.5 (3)	C8—S1—C7—C6	−69.17 (13)
Cl1—C2—C3—C4	−179.85 (13)	N1^{i}—N1—C8—S2	−1.62 (19)
C2—C3—C4—C5	0.1 (3)	N1^{i}—N1—C8—S1	171.24 (12)
C3—C4—C5—C6	0.4 (3)	C8^{i}—S2—C8—N1	0.62 (7)
C4—C5—C6—C1	−0.5 (2)	C8^{i}—S2—C8—S1	−171.70 (14)
C4—C5—C6—C7	177.37 (15)	C7—S1—C8—N1	154.09 (12)
C2—C1—C6—C5	0.1 (2)	C7—S1—C8—S2	−33.91 (11)
C2—C1—C6—C7	−177.73 (14)		

Symmetry code: (i) $-x, y, -z+1/2$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···N1^{ii}	0.93	2.59	3.514 (2)	172

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