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1,4-Bis[(5-phenyl-1,3,4-thiadiazol-2-yl)-sulfanyl]butane

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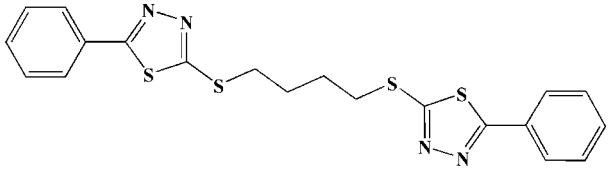
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.088; data-to-parameter ratio = 18.8.

The asymmetric unit of the title compound, $C_{20}H_{18}N_4S_4$, contains one half-molecule situated on a twofold rotation axis, in which the thiadiazole and phenyl rings are twisted by 7.2 (3)°. In the crystal, weak intermolecular C—H···π interactions link the molecules into layers parallel to (103).

Related literature

For the biological activity of 1,3,4-triazole derivatives, see: Nakagawa *et al.* (1996); Wang *et al.* (1999). For the crystal structure of bis(5-phenyl-1,3,4-thiadiazol-2-ylsulfanyl)methane, see: Wang *et al.* (2010).



Experimental

Crystal data

$C_{20}H_{18}N_4S_4$
M_r = 442.62
Monoclinic, $P2_1/c$
a = 5.7976 (7) Å
b = 13.4393 (14) Å
c = 12.9784 (12) Å
β = 99.120 (7)°
V = 998.44 (18) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.49 mm^{-1}
T = 113 K
0.20 × 0.18 × 0.10 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.908, T_{max} = 0.953
9992 measured reflections
2384 independent reflections
1870 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.088
S = 1.06
2384 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

Cg is the centroid of the C1–C6 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9B···Cg^{i}	0.99	2.70	3.540 (2)	144

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: CV5053).

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supporting information

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1,4-Bis[(5-phenyl-1,3,4-thiadiazol-2-yl)sulfanyl]butane

Shao-feng Li, Jing-jing Zhang, Xiao-yu Jia, Yan Gao and Wei Wang

S1. Comment

1,3,4-Thiadiazole derivatives exhibit a wide spectrum of biological activities (Nakagawa *et al.*, 1996; Wang *et al.*, 1999). Recently, we have published the crystal structure of bis(5-phenyl-1,3,4-thiadiazol-2-ylsulfanyl)methane (Wang *et al.*, 2010). Herewith we report the crystal structure of the title compound, (I).

In (I) (Fig. 1), the molecule is situated on a twofold rotational axis so asymmetric unit contains a half of the molecule. The dihedral angle between the thiadiazole and the attached benzene rings is 7.2 (3)° indicating that two rings are almost parallel. As a result of π-π conjugation, the C_{sp}^2-S bond [S2—C8 = 1.742 (2) Å] is significantly shorter than the C_{sp}^3-S bond [S2—C9 = 1.813 (2) Å].

In the crystal structure, weak intermolecular C—H···π interactions (Table 1) link molecules into layers parallel to (103) plane.

S2. Experimental

A suspension of 5-diphenyl-1,3,4-thiadiazol-2-thiol (2.0 mmol) and 1,1-dibromobutane (1.0 mmol) in ethanol (10 ml) was stirred at room temperature. The reaction progress was monitored *via* TLC. The resulting precipitate was filtered off, washed with cold ethanol, dried and purified to give the target product as light yellow solid in 85% yield. Crystals of (I) suitable for single-crystal X-ray analysis were grown by slow evaporation of a solution in chloroform-ethanol (1:1).

S3. Refinement

All H atoms were positioned geometrically (C—H = 0.95–0.99 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C).

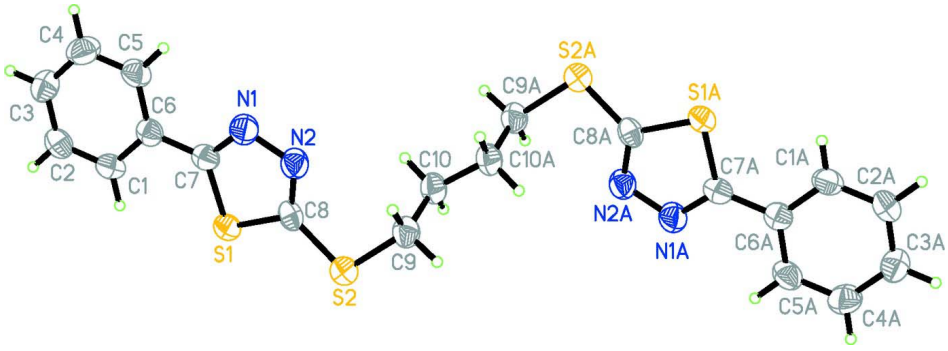

Figure 1

View of the molecule of (I) showing the atom-labelling scheme [symmetry code: (A)-*x* + 1, -*y* + 1, -*z*]. Displacement ellipsoids are drawn at the 85% probability level.

1,4-Bis[(5-phenyl-1,3,4-thiadiazol-2-yl)sulfanyl]butane

Crystal data

$C_{20}H_{18}N_4S_4$
$M_r = 442.62$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.7976\ (7)$ Å
$b = 13.4393\ (14)$ Å
$c = 12.9784\ (12)$ Å
$\beta = 99.120\ (7)°$
$V = 998.44\ (18)$ Å^3
$Z = 2$
$F(000) = 460$
$D_x = 1.472$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3630 reflections
$\theta = 1.5–27.9°$
$\mu = 0.49$ mm^{-1}
$T = 113$ K
Prism, colorless
0.20 × 0.18 × 0.10 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 14.63 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
$T_{min} = 0.908$, $T_{max} = 0.953$
9992 measured reflections
2384 independent reflections
1870 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.2°$
$h = -7\rightarrow7$
$k = -17\rightarrow17$
$l = -15\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.088$
$S = 1.06$
2384 reflections
127 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
$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.49$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	1.18924 (6)	0.48167 (3)	0.35236 (3)	0.01929 (12)
S2	0.94277 (7)	0.60479 (3)	0.17655 (3)	0.02206 (13)
N1	0.8027 (2)	0.39565 (9)	0.36814 (10)	0.0221 (3)
N2	0.7508 (2)	0.46308 (10)	0.28684 (10)	0.0206 (3)

C1	1.3577 (3)	0.34573 (11)	0.54385 (12)	0.0210 (3)
H1	1.4506	0.3955	0.5185	0.025*
C2	1.4532 (3)	0.28628 (12)	0.62747 (12)	0.0213 (3)
H2	1.6099	0.2969	0.6601	0.026*
C3	1.3224 (3)	0.21206 (12)	0.66341 (12)	0.0237 (4)
H3	1.3889	0.1710	0.7199	0.028*
C4	1.0921 (3)	0.19787 (12)	0.61620 (13)	0.0259 (4)
H4	1.0013	0.1469	0.6409	0.031*
C5	0.9936 (3)	0.25738 (12)	0.53341 (12)	0.0216 (3)
H5	0.8363	0.2469	0.5015	0.026*
C6	1.1262 (3)	0.33270 (11)	0.49711 (11)	0.0173 (3)
C7	1.0225 (3)	0.39663 (11)	0.40992 (12)	0.0171 (3)
C8	0.9342 (3)	0.51325 (11)	0.27088 (12)	0.0175 (3)
C9	0.6507 (3)	0.59299 (11)	0.10328 (12)	0.0211 (3)
H9A	0.5373	0.5895	0.1527	0.025*
H9B	0.6143	0.6531	0.0597	0.025*
C10	0.6215 (2)	0.50122 (11)	0.03322 (12)	0.0205 (3)
H10A	0.6445	0.4405	0.0769	0.025*
H10B	0.7419	0.5018	−0.0130	0.025*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01581 (19)	0.0215 (2)	0.0199 (2)	−0.00349 (15)	0.00081 (16)	0.00081 (15)
S2	0.0213 (2)	0.0229 (2)	0.0209 (2)	−0.00350 (16)	−0.00005 (17)	0.00217 (16)
N1	0.0185 (7)	0.0276 (7)	0.0199 (7)	−0.0024 (6)	0.0025 (6)	0.0024 (6)
N2	0.0171 (6)	0.0260 (7)	0.0185 (7)	−0.0008 (6)	0.0021 (5)	0.0017 (5)
C1	0.0198 (8)	0.0196 (8)	0.0232 (9)	−0.0048 (6)	0.0025 (7)	−0.0012 (6)
C2	0.0160 (7)	0.0247 (8)	0.0224 (9)	−0.0007 (6)	−0.0003 (6)	−0.0022 (7)
C3	0.0249 (8)	0.0251 (9)	0.0208 (8)	0.0021 (7)	0.0027 (7)	0.0039 (7)
C4	0.0235 (8)	0.0267 (9)	0.0286 (9)	−0.0040 (7)	0.0072 (7)	0.0057 (7)
C5	0.0155 (7)	0.0267 (8)	0.0223 (9)	−0.0040 (6)	0.0025 (6)	−0.0008 (7)
C6	0.0189 (7)	0.0175 (7)	0.0159 (8)	−0.0003 (6)	0.0041 (6)	−0.0036 (6)
C7	0.0168 (7)	0.0184 (7)	0.0169 (8)	−0.0024 (6)	0.0052 (6)	−0.0038 (6)
C8	0.0172 (7)	0.0208 (8)	0.0140 (8)	0.0004 (6)	0.0010 (6)	−0.0040 (6)
C9	0.0194 (8)	0.0221 (8)	0.0207 (9)	0.0017 (6)	−0.0008 (7)	0.0015 (6)
C10	0.0184 (8)	0.0224 (8)	0.0194 (8)	0.0023 (6)	−0.0007 (6)	0.0003 (6)

Geometric parameters (Å, º)

S1—C8	1.7289 (15)	C3—H3	0.9500
S1—C7	1.7400 (15)	C4—C5	1.388 (2)
S2—C8	1.7417 (16)	C4—H4	0.9500
S2—C9	1.8126 (15)	C5—C6	1.397 (2)
N1—C7	1.303 (2)	C5—H5	0.9500
N1—N2	1.3872 (17)	C6—C7	1.471 (2)
N2—C8	1.303 (2)	C9—C10	1.526 (2)
C1—C2	1.390 (2)	C9—H9A	0.9900

C1—C6	1.393 (2)	C9—H9B	0.9900
C1—H1	0.9500	C10—C10^{i}	1.531 (3)
C2—C3	1.379 (2)	C10—H10A	0.9900
C2—H2	0.9500	C10—H10B	0.9900
C3—C4	1.391 (2)		

C8—S1—C7	86.82 (7)	C1—C6—C7	120.63 (14)
C8—S2—C9	100.29 (7)	C5—C6—C7	120.18 (13)
C7—N1—N2	112.95 (13)	N1—C7—C6	124.58 (14)
C8—N2—N1	112.01 (12)	N1—C7—S1	113.64 (12)
C2—C1—C6	120.32 (15)	C6—C7—S1	121.78 (11)
C2—C1—H1	119.8	N2—C8—S1	114.58 (12)
C6—C1—H1	119.8	N2—C8—S2	126.30 (12)
C3—C2—C1	120.49 (14)	S1—C8—S2	119.11 (9)
C3—C2—H2	119.8	C10—C9—S2	112.94 (11)
C1—C2—H2	119.8	C10—C9—H9A	109.0
C2—C3—C4	119.44 (15)	S2—C9—H9A	109.0
C2—C3—H3	120.3	C10—C9—H9B	109.0
C4—C3—H3	120.3	S2—C9—H9B	109.0
C5—C4—C3	120.70 (15)	H9A—C9—H9B	107.8
C5—C4—H4	119.6	C9—C10—C10^{i}	111.00 (16)
C3—C4—H4	119.6	C9—C10—H10A	109.4
C4—C5—C6	119.84 (15)	C10^{i}—C10—H10A	109.4
C4—C5—H5	120.1	C9—C10—H10B	109.4
C6—C5—H5	120.1	C10^{i}—C10—H10B	109.4
C1—C6—C5	119.19 (14)	H10A—C10—H10B	108.0

C7—N1—N2—C8	−0.54 (19)	C1—C6—C7—S1	6.9 (2)
C6—C1—C2—C3	1.7 (2)	C5—C6—C7—S1	−172.53 (12)
C1—C2—C3—C4	−0.9 (2)	C8—S1—C7—N1	0.48 (12)
C2—C3—C4—C5	0.2 (3)	C8—S1—C7—C6	−179.80 (13)
C3—C4—C5—C6	−0.2 (3)	N1—N2—C8—S1	0.93 (17)
C2—C1—C6—C5	−1.8 (2)	N1—N2—C8—S2	−179.93 (11)
C2—C1—C6—C7	178.80 (14)	C7—S1—C8—N2	−0.81 (13)
C4—C5—C6—C1	1.1 (2)	C7—S1—C8—S2	179.98 (10)
C4—C5—C6—C7	−179.52 (15)	C9—S2—C8—N2	−7.85 (16)
N2—N1—C7—C6	−179.79 (13)	C9—S2—C8—S1	171.26 (9)
N2—N1—C7—S1	−0.08 (17)	C8—S2—C9—C10	−75.00 (13)
C1—C6—C7—N1	−173.44 (15)	S2—C9—C10—C10^{i}	−175.61 (14)
C5—C6—C7—N1	7.2 (2)		

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

C9—H9*B*···*Cg*^{ii}	0.99	2.70	3.540 (2)	144

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