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

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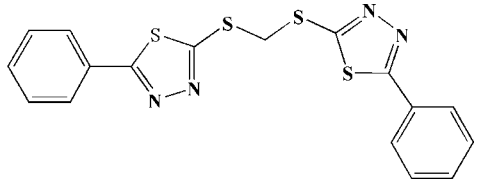
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.004 Å; R factor = 0.029; wR factor = 0.124; data-to-parameter ratio = 12.8.

The asymmetric unit of the title compound, $C_{17}H_{12}N_4S_4$, contains one half-molecule situated on a twofold rotational axis. In the molecule, the thiadiazole and attached phenyl rings are twisted by 5.8 (3)°.

Related literature

For biological activity of 1,3,4-thiadiazole derivatives, see: Nakagawa *et al.* (1996); Wang *et al.* (1999); Carvalho *et al.* (2004); Riente *et al.* (2009); Poorrajab *et al.* (2009).



Experimental

Crystal data

$C_{17}H_{12}N_4S_4$
M_r = 400.55
Orthorhombic, $P2_12_12$
a = 10.805 (2) Å
b = 19.287 (4) Å
c = 4.0738 (8) Å
V = 848.9 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.57 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} = 0.895, T_{max} = 0.945
6754 measured reflections
1477 independent reflections
1421 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.124
S = 1.03
1477 reflections
115 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}
Absolute structure: Flack (1983), 554 Friedel pairs
Flack parameter: 0.16 (14)

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: CV2783).

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supplementary materials

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

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Comment

1,3,4-Thiadiazole derivatives attracted considerable attention due to their broad spectrum of chemical and pharmaceutical properties (Nakagawa *et al.*, 1996; Wang *et al.*, 1999), with particular attention being paid to the anti-trypanosomal activities of Megazol and related compounds (Carvalho *et al.*, 2004; Riente *et al.*, 2009; Poorrajab *et al.*, 2009). Herewith we report the synthesis and crystal structure of the title compound, (I), a new 1,3,4-thiadiazole derivative.

The molecular structure of (I) is shown in Fig.1. In the crystal structure, the molecule is situated on a two-fold rotational axis so asymmetric unit contains a half of the molecule. 1,3,4-Thiadiazole ring is planar with an r.m.s. deviation of 0.0048 (2)Å and maximum deviation of 0.0072 (2)Å for atom C7. The dihedral angle between the thiadiazole and attached phenyl rings is 5.8 (3)°. As a result of π-π conjugation, the C_{sp}^2-S bond length [S2—C8 = 1.751 (3) Å] is significantly shorter than the C_{sp}^3-S bond length [S2—C9 = 1.810 (2) Å].

Experimental

A suspension of 5-diphenyl-1,3,4-thiadiazol-2-thiol (2.0 mmol) and 1,1-dibromomethane (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 95% yield. Crystals of (I) suitable for single-crystal X-ray analysis were grown by slow evaporation of a solution in chloroform-ethanol (1:1).

Refinement

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

Figures

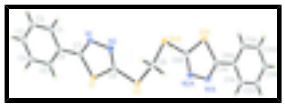


Fig. 1. View of (I) showing the atom-labelling scheme and 35% probability displacement ellipsoids [symmetry code: (A) = -*x*,-*y* + 1,*z*].

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

Crystal data

$C_{17}H_{12}N_4S_4$	$F(000)$ = 412
M_r = 400.55	D_x = 1.567 Mg m^{-3}
Orthorhombic, $P2_12_12$	Mo $K\alpha$ radiation, λ = 0.71073 Å

Hall symbol: P 2 2ab	Cell parameters from 2856 reflections
a = 10.805 (2) Å	θ = 2.1–27.9°
b = 19.287 (4) Å	μ = 0.57 mm^{-1}
c = 4.0738 (8) Å	T = 113 K
V = 848.9 (3) $Å^3$	Prism, colourless
Z = 2	0.20 × 0.18 × 0.10 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer	1477 independent reflections
Radiation source: rotating anode	1421 reflections with $I > 2\sigma(I)$
confocal	R_{int} = 0.028
Detector resolution: 7.31 pixels mm^{-1}	θ_{max} = 25.0°, θ_{min} = 2.1°
φ and ω scans	h = −12→12
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)	k = −22→22
T_{min} = 0.895, T_{max} = 0.945	l = −4→4
6754 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.029	$w = 1/[\sigma^2(F_o^2) + (0.110P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.124	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.02	$\Delta\rho_{max}$ = 0.54 e $Å^{-3}$
1477 reflections	$\Delta\rho_{min}$ = −0.55 e $Å^{-3}$
115 parameters	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
0 restraints	Extinction coefficient: 0.049 (10)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 554 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.16 (14)

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}	Occ. (<1)
S1	0.19317 (6)	0.33910 (3)	0.3294 (2)	0.0215 (3)	
S2	0.13644 (6)	0.47579 (4)	0.6577 (2)	0.0216 (3)	
N1	−0.0251 (2)	0.29797 (13)	0.4770 (7)	0.0223 (6)	
N2	−0.0161 (2)	0.36481 (13)	0.6002 (7)	0.0231 (6)	
C1	−0.0141 (3)	0.16581 (16)	0.1495 (9)	0.0260 (7)	
H1	−0.0904	0.1795	0.2453	0.031*	
C2	−0.0048 (3)	0.10272 (16)	−0.0079 (9)	0.0289 (8)	
H2	−0.0748	0.0731	−0.0206	0.035*	
C3	0.1073 (3)	0.08236 (15)	−0.1485 (9)	0.0279 (7)	
H3	0.1136	0.0389	−0.2568	0.033*	
C4	0.2085 (3)	0.12546 (15)	−0.1299 (9)	0.0262 (7)	
H4	0.2847	0.1117	−0.2264	0.031*	
C5	0.1999 (3)	0.18852 (16)	0.0284 (8)	0.0234 (7)	
H5	0.2704	0.2178	0.0420	0.028*	
C6	0.0885 (3)	0.20960 (14)	0.1682 (8)	0.0196 (6)	
C7	0.0747 (2)	0.27771 (15)	0.3277 (7)	0.0181 (6)	
C8	0.0919 (3)	0.39217 (14)	0.5395 (8)	0.0192 (7)	
C9	0.0000	0.5000	0.8888 (11)	0.0229 (10)	
H9A	0.0223	0.5394	1.0330	0.027*	0.50
H9B	−0.0223	0.4606	1.0330	0.027*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0163 (4)	0.0229 (4)	0.0252 (5)	−0.0010 (3)	0.0012 (4)	−0.0011 (3)
S2	0.0221 (4)	0.0204 (4)	0.0223 (5)	−0.0011 (3)	−0.0018 (4)	0.0005 (3)
N1	0.0199 (12)	0.0197 (12)	0.0273 (14)	0.0005 (9)	−0.0006 (12)	0.0018 (12)
N2	0.0229 (12)	0.0200 (12)	0.0263 (15)	0.0006 (10)	0.0035 (11)	−0.0007 (12)
C1	0.0198 (14)	0.0279 (15)	0.0303 (18)	0.0002 (11)	0.0039 (16)	0.0029 (17)
C2	0.0258 (14)	0.0254 (15)	0.036 (2)	−0.0019 (12)	−0.0060 (17)	−0.0012 (16)
C3	0.0374 (17)	0.0206 (13)	0.0257 (17)	0.0052 (13)	0.0029 (17)	−0.0019 (15)
C4	0.0244 (14)	0.0254 (14)	0.0289 (18)	0.0079 (12)	0.0013 (15)	0.0031 (16)
C5	0.0208 (14)	0.0232 (14)	0.0260 (17)	0.0012 (12)	−0.0002 (15)	0.0041 (14)
C6	0.0189 (14)	0.0209 (14)	0.0191 (15)	0.0028 (11)	−0.0046 (13)	0.0043 (14)
C7	0.0162 (13)	0.0216 (13)	0.0166 (14)	0.0000 (11)	−0.0015 (13)	0.0033 (13)
C8	0.0206 (14)	0.0198 (12)	0.0170 (15)	0.0038 (11)	−0.0010 (13)	−0.0001 (12)
C9	0.029 (2)	0.0235 (19)	0.016 (2)	−0.0003 (17)	0.000	0.000

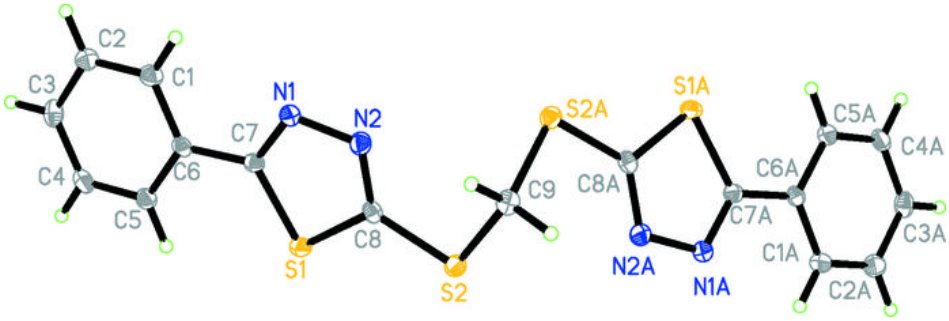
Geometric parameters (Å, °)

S1—C8	1.726 (3)	C2—H2	0.9500
S1—C7	1.744 (3)	C3—C4	1.376 (5)
S2—C8	1.751 (3)	C3—H3	0.9500
S2—C9	1.810 (2)	C4—C5	1.380 (5)
N1—C7	1.298 (4)	C4—H4	0.9500

N1—N2	1.387 (4)	C5—C6	1.391 (4)
N2—C8	1.304 (4)	C5—H5	0.9500
C1—C2	1.379 (5)	C6—C7	1.473 (4)
C1—C6	1.396 (4)	C9—S2^{i}	1.810 (2)
C1—H1	0.9500	C9—H9A	0.9900
C2—C3	1.396 (4)	C9—H9B	0.9900
C8—S1—C7	86.51 (14)	C4—C5—H5	119.8
C8—S2—C9	99.02 (10)	C6—C5—H5	119.8
C7—N1—N2	113.0 (2)	C5—C6—C1	119.2 (3)
C8—N2—N1	111.8 (2)	C5—C6—C7	121.9 (3)
C2—C1—C6	120.1 (3)	C1—C6—C7	118.9 (3)
C2—C1—H1	119.9	N1—C7—C6	124.0 (3)
C6—C1—H1	119.9	N1—C7—S1	113.8 (2)
C1—C2—C3	120.1 (3)	C6—C7—S1	122.2 (2)
C1—C2—H2	119.9	N2—C8—S1	114.9 (2)
C3—C2—H2	119.9	N2—C8—S2	124.5 (2)
C4—C3—C2	119.8 (3)	S1—C8—S2	120.57 (17)
C4—C3—H3	120.1	S2—C9—S2^{i}	117.3 (2)
C2—C3—H3	120.1	S2—C9—H9A	108.0
C3—C4—C5	120.3 (3)	S2^{i}—C9—H9A	108.0
C3—C4—H4	119.9	S2—C9—H9B	108.0
C5—C4—H4	119.9	S2^{i}—C9—H9B	108.0
C4—C5—C6	120.5 (3)	H9A—C9—H9B	107.2
C7—N1—N2—C8	−0.5 (4)	C1—C6—C7—N1	5.7 (5)
C6—C1—C2—C3	0.1 (5)	C5—C6—C7—S1	4.3 (4)
C1—C2—C3—C4	0.0 (5)	C1—C6—C7—S1	−173.9 (3)
C2—C3—C4—C5	0.3 (6)	C8—S1—C7—N1	−1.1 (2)
C3—C4—C5—C6	−0.6 (5)	C8—S1—C7—C6	178.6 (3)
C4—C5—C6—C1	0.7 (5)	N1—N2—C8—S1	−0.4 (3)
C4—C5—C6—C7	−177.6 (3)	N1—N2—C8—S2	179.9 (2)
C2—C1—C6—C5	−0.4 (5)	C7—S1—C8—N2	0.8 (3)
C2—C1—C6—C7	177.9 (3)	C7—S1—C8—S2	−179.5 (2)
N2—N1—C7—C6	−178.6 (2)	C9—S2—C8—N2	4.3 (3)
N2—N1—C7—S1	1.1 (3)	C9—S2—C8—S1	−175.34 (19)
C5—C6—C7—N1	−176.0 (3)	C8—S2—C9—S2^{i}	−76.74 (11)

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

Fig. 1


C2
C1
C3
N1
N2
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S1
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S2A
S1A
C8A
N2A
N1A
C7A
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C4A
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