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2-(4-Methylphenyl)-5-[({[5-(4-methylphenyl)-1,3,4-thiadiazol-2-yl]sulfanyl}methyl)sulfanyl]-1,3,4-thiadiazole

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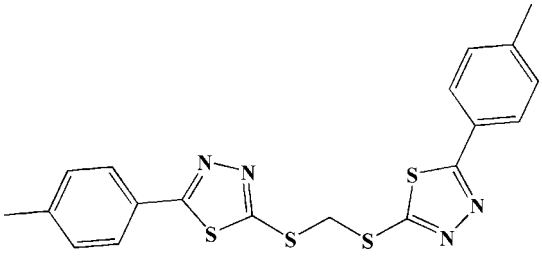
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.005 Å; R factor = 0.057; wR factor = 0.142; data-to-parameter ratio = 18.4.

In the title compound, $C_{19}H_{16}N_4S_4$, the molecules exhibit a butterfly conformation, where the thiadiazole and attached benzene rings in two wings are almost coplanar, with dihedral angles of 0.8 (3) and 0.9 (3)°, respectively, while the two thiadiazole rings form a dihedral angle of 46.3 (3)°.

Related literature

For the biological properties of 1,3,4-thiadiazole derivatives, see: Nakagawa *et al.* (1996); Wang *et al.* (1999); Carvalho *et al.* (2004). For the crystal structures of related compounds, see: Li *et al.* (2011); Wang *et al.* (2010).



Experimental

Crystal data

$C_{19}H_{16}N_4S_4$
M_r = 428.60
Monoclinic, $P2_1/n$
a = 16.8944 (14) Å
b = 4.1959 (5) Å
c = 27.107 (2) Å
β = 96.084 (8)°
V = 1910.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.51 mm^{-1}
T = 113 K
0.50 × 0.04 × 0.04 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.785, T_{max} = 0.980
14969 measured reflections
4535 independent reflections
2927 reflections with $I > 2\sigma(I)$
R_{int} = 0.094

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.142
S = 0.96
4535 reflections
246 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}

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: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5245).

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supporting information

Acta Cryst. (2012). E**68**, o851 [doi:10.1107/S1600536812007520]

2-(4-Methylphenyl)-5-[({[5-(4-methylphenyl)-1,3,4-thiadiazol-2-yl]sulfanyl}methyl)sulfanyl]-1,3,4-thiadiazole

Yong Wang, Wen-ge Zhang, Yu-bo Wang, Jing-wen Yu and Lin Zhou

S1. Comment

1,3,4-Thiadiazole derivatives are important for medicinal chemistry due to their chemical and pharmaceutical properties (Nakagawa *et al.*, 1996; Wang *et al.*, 1999; Carvalho *et al.*, 2004). Similar crystal structures of the 1,3,4-thiadiazole derivatives have been reported . As a part of our research, the title compound (I) has been synthesized, and herewith we present its crystal structure.

In (I) (Fig. 1), all geometric parameters are normal and comparable with those reported for related 1,3,4-thiadiazole derivatives (Li *et al.*, 2011; Wang *et al.*, 2010). Two thiadiazole rings form a dihedral angle of 46.3 (3)%. The dihedral angles between the benzene rings and attached thiadiazole rings are 0.8 (3) and 0.9 (3)° indicating the two rings are almost parallel. The same situation has been observed in the crystal structure of 1,4-bis(5-phenyl-1,3,4-thiadiazol-2-ylsulfanyl)butane (Li *et al.*, 2011).

S2. Experimental

The title compound was synthesized by the reaction of the 1,1-dibromomethane (1.0 mmol) and 5-tolyl-1,3,4-thiadiazol-2-thiol (2.0 mmol) in ethanol (20 ml) at room temperature. Crystals of (I) suitable for single-crystal X-ray analysis were grown by slow evaporation of a solution in chloroform-enthanol (1:1).

S3. Refinement

All H atoms were positioned geometrically and refined as riding (C—H = 0.95 - 0.99 Å), with U_{iso}(H) = 1.2 - 1.5U_{eq} (C).

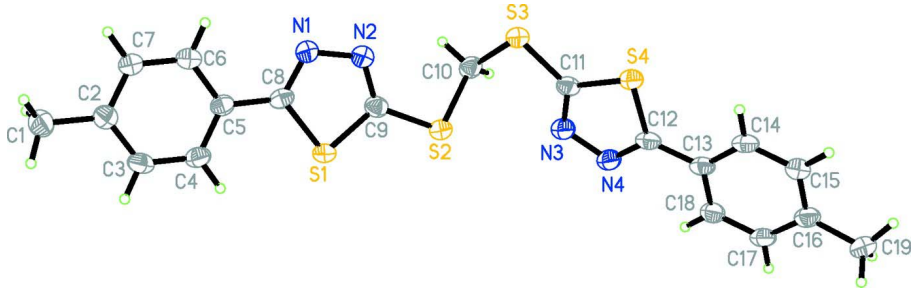

Figure 1

View of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 60% probability level.

2-(4-Methylphenyl)-5-[({[5-(4-methylphenyl)-1,3,4-thiadiazol-2- yl]sulfanyl}methyl)sulfanyl]-1,3,4-thiadiazole

Crystal data

$C_{19}H_{16}N_4S_4$
$M_r = 428.60$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 16.8944$ (14) Å
$b = 4.1959$ (5) Å
$c = 27.107$ (2) Å
$\beta = 96.084$ (8)°
$V = 1910.7$ (3) Å^3
$Z = 4$
$F(000) = 888$
$D_x = 1.490$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5461 reflections
$\theta = 1.4$–27.8°
$\mu = 0.51$ mm^{-1}
$T = 113$ K
Prism, colourless
0.50 × 0.04 × 0.04 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 14.22 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
$T_{min} = 0.785$, $T_{max} = 0.980$
14969 measured reflections
4535 independent reflections
2927 reflections with $I > 2\sigma(I)$
$R_{int} = 0.094$
$\theta_{max} = 27.8°$, $\theta_{min} = 1.4°$
$h = -22\rightarrow22$
$k = -5\rightarrow4$
$l = -35\rightarrow31$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.142$
$S = 0.96$
4535 reflections
246 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
$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.60$ 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	0.41711 (5)	0.77165 (19)	−0.01333 (3)	0.0266 (2)
S2	0.39123 (5)	1.1072 (2)	0.08165 (3)	0.0282 (2)
S3	0.26282 (5)	1.06832 (19)	0.15267 (3)	0.0270 (2)
S4	0.33462 (5)	1.07934 (19)	0.26002 (3)	0.0268 (2)

N1	0.26848 (16)	0.6479 (6)	−0.02748 (10)	0.0285 (6)
N2	0.27897 (16)	0.8187 (6)	0.01681 (10)	0.0288 (6)
N3	0.39960 (17)	1.3525 (6)	0.18983 (10)	0.0292 (6)
N4	0.44931 (16)	1.4137 (6)	0.23312 (10)	0.0289 (6)
C1	0.3546 (2)	−0.0838 (8)	−0.23078 (12)	0.0349 (8)
H1A	0.3223	−0.2782	−0.2313	0.052*
H1B	0.4101	−0.1403	−0.2341	0.052*
H1C	0.3343	0.0547	−0.2584	0.052*
C2	0.3500 (2)	0.0891 (7)	−0.18231 (12)	0.0263 (7)
C3	0.4179 (2)	0.2240 (8)	−0.15616 (12)	0.0296 (8)
H3	0.4680	0.2007	−0.1687	0.036*
C4	0.4135 (2)	0.3902 (7)	−0.11251 (12)	0.0283 (7)
H4	0.4602	0.4807	−0.0955	0.034*
C5	0.34068 (19)	0.4251 (7)	−0.09335 (11)	0.0237 (7)
C6	0.27246 (19)	0.2897 (7)	−0.11872 (12)	0.0268 (7)
H6	0.2225	0.3126	−0.1060	0.032*
C7	0.2776 (2)	0.1227 (7)	−0.16224 (12)	0.0275 (7)
H7	0.2309	0.0289	−0.1788	0.033*
C8	0.33463 (19)	0.6019 (7)	−0.04729 (11)	0.0242 (7)
C9	0.35275 (19)	0.8994 (7)	0.02832 (11)	0.0255 (7)
C10	0.2996 (2)	1.2661 (7)	0.10072 (12)	0.0277 (7)
H10A	0.3080	1.4939	0.1092	0.033*
H10B	0.2581	1.2551	0.0721	0.033*
C11	0.33781 (19)	1.1814 (7)	0.19861 (12)	0.0247 (7)
C12	0.42392 (18)	1.2861 (7)	0.27270 (12)	0.0258 (7)
C13	0.46511 (19)	1.3186 (7)	0.32295 (12)	0.0262 (7)
C14	0.43531 (19)	1.1764 (8)	0.36363 (12)	0.0278 (7)
H14	0.3872	1.0580	0.3591	0.033*
C15	0.4755 (2)	1.2068 (8)	0.41067 (12)	0.0292 (7)
H15	0.4540	1.1096	0.4380	0.035*
C16	0.54653 (19)	1.3758 (7)	0.41906 (12)	0.0285 (7)
C17	0.5753 (2)	1.5225 (8)	0.37799 (12)	0.0286 (7)
H17	0.6231	1.6434	0.3826	0.034*
C18	0.53548 (19)	1.4947 (8)	0.33077 (12)	0.0279 (7)
H18	0.5562	1.5961	0.3035	0.033*
C19	0.5928 (2)	1.3981 (9)	0.46959 (12)	0.0346 (8)
H19A	0.6398	1.2602	0.4708	0.052*
H19B	0.5590	1.3294	0.4949	0.052*
H19C	0.6096	1.6190	0.4760	0.052*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0174 (4)	0.0334 (5)	0.0289 (4)	−0.0015 (3)	0.0025 (3)	−0.0022 (4)
S2	0.0207 (4)	0.0359 (5)	0.0279 (4)	−0.0021 (3)	0.0019 (3)	−0.0029 (4)
S3	0.0204 (4)	0.0320 (5)	0.0285 (4)	−0.0002 (3)	0.0022 (3)	−0.0018 (3)
S4	0.0197 (4)	0.0329 (5)	0.0279 (4)	−0.0018 (3)	0.0022 (3)	0.0007 (3)
N1	0.0230 (15)	0.0343 (16)	0.0279 (14)	0.0008 (12)	0.0014 (12)	−0.0048 (12)

N2	0.0242 (15)	0.0342 (16)	0.0282 (14)	0.0000 (12)	0.0035 (12)	−0.0044 (12)
N3	0.0228 (15)	0.0352 (16)	0.0296 (14)	−0.0016 (12)	0.0026 (12)	0.0011 (12)
N4	0.0211 (15)	0.0357 (16)	0.0297 (15)	−0.0025 (12)	0.0013 (12)	0.0020 (12)
C1	0.034 (2)	0.037 (2)	0.0336 (19)	0.0041 (16)	0.0044 (16)	−0.0033 (16)
C2	0.0266 (18)	0.0262 (17)	0.0261 (16)	0.0032 (14)	0.0024 (14)	0.0034 (13)
C3	0.0204 (17)	0.0360 (19)	0.0332 (18)	0.0014 (14)	0.0064 (15)	0.0030 (15)
C4	0.0199 (17)	0.0312 (18)	0.0332 (18)	−0.0029 (14)	0.0003 (14)	−0.0010 (14)
C5	0.0209 (17)	0.0231 (16)	0.0262 (16)	0.0013 (13)	−0.0009 (13)	0.0034 (13)
C6	0.0203 (17)	0.0292 (17)	0.0311 (17)	0.0002 (13)	0.0035 (14)	0.0023 (14)
C7	0.0214 (17)	0.0306 (18)	0.0303 (17)	−0.0017 (14)	0.0012 (14)	−0.0005 (14)
C8	0.0196 (16)	0.0231 (16)	0.0290 (16)	0.0002 (13)	−0.0020 (13)	0.0030 (13)
C9	0.0230 (17)	0.0279 (17)	0.0261 (16)	0.0001 (14)	0.0045 (14)	0.0043 (14)
C10	0.0249 (18)	0.0283 (17)	0.0290 (17)	0.0051 (14)	−0.0019 (14)	−0.0024 (14)
C11	0.0185 (16)	0.0265 (17)	0.0291 (16)	0.0049 (13)	0.0034 (13)	0.0005 (13)
C12	0.0151 (16)	0.0272 (17)	0.0355 (18)	0.0012 (13)	0.0041 (14)	−0.0025 (14)
C13	0.0169 (16)	0.0276 (17)	0.0342 (18)	0.0044 (13)	0.0029 (14)	−0.0012 (14)
C14	0.0156 (16)	0.0309 (18)	0.0371 (18)	0.0004 (13)	0.0028 (14)	−0.0020 (15)
C15	0.0217 (17)	0.0355 (19)	0.0308 (17)	−0.0002 (14)	0.0044 (14)	−0.0010 (15)
C16	0.0193 (17)	0.0306 (18)	0.0350 (18)	0.0046 (14)	0.0001 (14)	−0.0035 (15)
C17	0.0169 (16)	0.0317 (18)	0.0373 (19)	0.0002 (14)	0.0027 (14)	−0.0037 (15)
C18	0.0210 (17)	0.0300 (17)	0.0329 (17)	0.0012 (14)	0.0043 (14)	−0.0028 (15)
C19	0.0221 (18)	0.046 (2)	0.0344 (18)	0.0029 (15)	−0.0023 (15)	−0.0011 (16)

Geometric parameters (Å, º)

S1—C9	1.733 (3)	C4—H4	0.9500
S1—C8	1.739 (3)	C5—C6	1.398 (4)
S2—C9	1.753 (3)	C5—C8	1.465 (4)
S2—C10	1.810 (3)	C6—C7	1.383 (4)
S3—C11	1.745 (3)	C6—H6	0.9500
S3—C10	1.801 (3)	C7—H7	0.9500
S4—C11	1.725 (3)	C10—H10A	0.9900
S4—C12	1.743 (3)	C10—H10B	0.9900
N1—C8	1.304 (4)	C12—C13	1.469 (4)
N1—N2	1.393 (4)	C13—C14	1.394 (4)
N2—C9	1.297 (4)	C13—C18	1.397 (4)
N3—C11	1.310 (4)	C14—C15	1.386 (4)
N3—N4	1.393 (4)	C14—H14	0.9500
N4—C12	1.311 (4)	C15—C16	1.391 (4)
C1—C2	1.510 (4)	C15—H15	0.9500
C1—H1A	0.9800	C16—C17	1.403 (5)
C1—H1B	0.9800	C16—C19	1.506 (4)
C1—H1C	0.9800	C17—C18	1.386 (5)
C2—C7	1.398 (4)	C17—H17	0.9500
C2—C3	1.403 (4)	C18—H18	0.9500
C3—C4	1.382 (4)	C19—H19A	0.9800
C3—H3	0.9500	C19—H19B	0.9800
C4—C5	1.393 (4)	C19—H19C	0.9800

C9—S1—C8	87.06 (15)	S1—C9—S2	119.18 (19)
C9—S2—C10	99.51 (15)	S3—C10—S2	115.46 (17)
C11—S3—C10	98.52 (15)	S3—C10—H10A	108.4
C11—S4—C12	87.24 (15)	S2—C10—H10A	108.4
C8—N1—N2	113.2 (3)	S3—C10—H10B	108.4
C9—N2—N1	111.9 (3)	S2—C10—H10B	108.4
C11—N3—N4	111.6 (3)	H10A—C10—H10B	107.5
C12—N4—N3	113.2 (3)	N3—C11—S4	114.7 (2)
C2—C1—H1A	109.5	N3—C11—S3	123.5 (2)
C2—C1—H1B	109.5	S4—C11—S3	121.77 (18)
H1A—C1—H1B	109.5	N4—C12—C13	123.9 (3)
C2—C1—H1C	109.5	N4—C12—S4	113.2 (2)
H1A—C1—H1C	109.5	C13—C12—S4	123.0 (2)
H1B—C1—H1C	109.5	C14—C13—C18	118.6 (3)
C7—C2—C3	117.7 (3)	C14—C13—C12	121.2 (3)
C7—C2—C1	121.0 (3)	C18—C13—C12	120.1 (3)
C3—C2—C1	121.3 (3)	C15—C14—C13	120.3 (3)
C4—C3—C2	121.4 (3)	C15—C14—H14	119.8
C4—C3—H3	119.3	C13—C14—H14	119.8
C2—C3—H3	119.3	C14—C15—C16	121.9 (3)
C3—C4—C5	120.2 (3)	C14—C15—H15	119.1
C3—C4—H4	119.9	C16—C15—H15	119.1
C5—C4—H4	119.9	C15—C16—C17	117.3 (3)
C4—C5—C6	119.2 (3)	C15—C16—C19	122.4 (3)
C4—C5—C8	121.0 (3)	C17—C16—C19	120.3 (3)
C6—C5—C8	119.8 (3)	C18—C17—C16	121.4 (3)
C7—C6—C5	120.2 (3)	C18—C17—H17	119.3
C7—C6—H6	119.9	C16—C17—H17	119.3
C5—C6—H6	119.9	C17—C18—C13	120.4 (3)
C6—C7—C2	121.3 (3)	C17—C18—H18	119.8
C6—C7—H7	119.3	C13—C18—H18	119.8
C2—C7—H7	119.3	C16—C19—H19A	109.5
N1—C8—C5	124.5 (3)	C16—C19—H19B	109.5
N1—C8—S1	113.2 (2)	H19A—C19—H19B	109.5
C5—C8—S1	122.3 (2)	C16—C19—H19C	109.5
N2—C9—S1	114.6 (2)	H19A—C19—H19C	109.5
N2—C9—S2	126.2 (3)	H19B—C19—H19C	109.5

C8—N1—N2—C9	1.0 (4)	C11—S3—C10—S2	67.8 (2)
C11—N3—N4—C12	0.4 (4)	C9—S2—C10—S3	104.05 (19)
C7—C2—C3—C4	−1.3 (5)	N4—N3—C11—S4	−0.1 (3)
C1—C2—C3—C4	178.2 (3)	N4—N3—C11—S3	178.8 (2)
C2—C3—C4—C5	0.4 (5)	C12—S4—C11—N3	−0.2 (3)
C3—C4—C5—C6	0.1 (5)	C12—S4—C11—S3	−179.1 (2)
C3—C4—C5—C8	−179.6 (3)	C10—S3—C11—N3	−2.0 (3)
C4—C5—C6—C7	0.2 (5)	C10—S3—C11—S4	176.80 (19)
C8—C5—C6—C7	179.9 (3)	N3—N4—C12—C13	−179.3 (3)

C5—C6—C7—C2	−1.0 (5)	N3—N4—C12—S4	−0.6 (3)
C3—C2—C7—C6	1.6 (5)	C11—S4—C12—N4	0.4 (2)
C1—C2—C7—C6	−177.9 (3)	C11—S4—C12—C13	179.2 (3)
N2—N1—C8—C5	178.9 (3)	N4—C12—C13—C14	−179.8 (3)
N2—N1—C8—S1	−0.9 (3)	S4—C12—C13—C14	1.6 (4)
C4—C5—C8—N1	179.1 (3)	N4—C12—C13—C18	0.3 (5)
C6—C5—C8—N1	−0.7 (5)	S4—C12—C13—C18	−178.3 (2)
C4—C5—C8—S1	−1.2 (4)	C18—C13—C14—C15	−0.8 (5)
C6—C5—C8—S1	179.1 (2)	C12—C13—C14—C15	179.3 (3)
C9—S1—C8—N1	0.4 (2)	C13—C14—C15—C16	−0.5 (5)
C9—S1—C8—C5	−179.4 (3)	C14—C15—C16—C17	1.5 (5)
N1—N2—C9—S1	−0.7 (3)	C14—C15—C16—C19	−177.1 (3)
N1—N2—C9—S2	−178.4 (2)	C15—C16—C17—C18	−1.3 (5)
C8—S1—C9—N2	0.2 (3)	C19—C16—C17—C18	177.4 (3)
C8—S1—C9—S2	178.1 (2)	C16—C17—C18—C13	0.0 (5)
C10—S2—C9—N2	−16.9 (3)	C14—C13—C18—C17	1.0 (5)
C10—S2—C9—S1	165.53 (19)	C12—C13—C18—C17	−179.1 (3)