Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-[(4-Methoxybenzyl)sulfanyl]-2-methyl-1,3,4-thiadiazole

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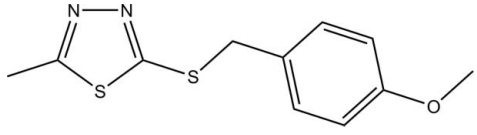
Received 7 December 2010; accepted 10 December 2010

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.052; wR factor = 0.118; data-to-parameter ratio = 19.2.

The title molecule, $C_{11}H_{12}N_2OS_2$, is twisted with a dihedral angle of 83.63 (12)° between the 1,3,4-thiadiazole and benzene rings. The methoxy group deviates slightly from the attached benzene ring, with a C—C—O—C torsion angle of 4.2 (4)°. In the crystal, molecules are linked by weak C—H···N interactions and stacked along the c axis.

Related literature

For bond-length data, see: Allen *et al.* (1987). For a related structure, see: Wang *et al.* (2010). For background to and applications of thiadiazole derivatives, see: Bernard *et al.* (1985); Chandrakantha *et al.* (2010); El-Sabbagh *et al.* (2009); Isloor *et al.* (2010); Kalluraya *et al.* (2004). For the stability of the temperature controller, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_{11}H_{12}N_2OS_2$
M_r = 252.35
Monoclinic, $P2_1/c$
a = 14.7765 (4) Å
b = 8.6916 (3) Å
c = 9.7339 (3) Å
β = 96.477 (1)°
V = 1242.16 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.41 mm^{-1}
T = 296 K
0.25 × 0.19 × 0.03 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.907, T_{max} = 0.987
11429 measured reflections
2828 independent reflections
1660 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.118
S = 1.02
2828 reflections
147 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1B···N1^{i}	0.96	2.59	3.532 (4)	164

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

AMI is thankful to the Director of the National Institute of Technology for providing research facilities and also thanks the Board for Research in Nuclear Sciences, Department of Atomic Energy, Government of India, for the Young Scientist award. SC thanks the Prince of Songkla University for generous support through the Crystal Materials Research Unit. The authors also thank Universiti Sains Malaysia for the Research University grant No. 1001/PFIZIK/811160.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2640).

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‡ Thomson Reuters ResearcherID: A-3561-2009.
§ Thomson Reuters ResearcherID: A-5085-2009.

supporting information

Acta Cryst. (2011). E**67**, o163 [https://doi.org/10.1107/S1600536810051858]

5-[(4-Methoxybenzyl)sulfanyl]-2-methyl-1,3,4-thiadiazole

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S1. Comment

Thiadiazole are a class of heterocyclic compounds having a five membered ring. They occur in nature and are predominant among all types of pharmaceuticals, agrochemicals and veterinary products (El-Sabbagh *et al.*, 2009). The amino and mercapto groups in thiadiazole are readily-accessible nucleophilic centers. 1,3,4-Thiadiazole exhibit a wide spectrum of biological activities (Bernard *et al.*, 1985). Due to the presence of the –N—C—S moiety (Kalluraya *et al.*, 2004), they are found to be used as antibacterial, antimicrobial and anti-inflammatory agents (Chandrakantha *et al.*, 2010). Antibacterial and antifungal (Isloor *et al.*, 2010) activities of the azoles are most widely studied and azoles are also used as antimicrobial agents. Herein we report the crystal structure of the title 1,3,4-thiadiazole derivative, (I).

The molecule of (I) (Fig. 1) is twisted with a dihedral angle between the 1,3,4-thiadiazole and benzene rings being 83.63 (12)°. Atoms C3, S2, C4 and C5 lie nearly on the same plane with *r.m.s*. 0.0517 (5) Å and the torsion angle C3–S2–C4–C5 = 172.25 (18)°. The mean plane through C3/S2/C4/C5 makes the dihedral angles of 9.02 (15) and 75.92 (16)° with the 1,3,4-thiadiazole and benzene rings, respectively. The methoxy group is slightly deviated with respect to the attached benzene ring with the torsion angle C11–O1–C8–C9 = 4.2 (4)°. The bond distances are of normal values (Allen *et al.*, 1987) and are comparable with the related structure (Wang *et al.*, 2010).

In the crystal packing (Fig. 2), the molecules are linked by C1—H1B···N1 weak interactions (Table 1) and stacked along the *c* axis. S···N [3.340 (2) Å] short contacts (symmetry codes: *x*, 1/2 - *y*, 1/2 + *z* and *x*, 1/2 - *y*, -1/2 + *z*) are presented in the crystal.

S2. Experimental

The title compound was synthesized by adding 4-methoxybenzylbromide (3.02 g, 0.0151 mol) dropwise to a stirred solution of 5-methyl-1,3,4-thiadiazole-2-thiol (2.00 g, 0.0151 mol) and anhydrous potassiumcarbonate (4.16 g, 0.03 mol) in dry acetonitrile (50 ml) at room temperature and the reaction mixture was stirred at room temperature for 5 h. After the completion of reaction, the reaction mixture was filtered and the filtrate was concentrated. The crude product was recrystallized with hot ethanol to afford the title compound as yellow solid (2.00 g, yield 57%). Yellow plate-shaped single crystals of the title compound suitable for *x*-ray structure determination were recrystalized from ethanol by the slow evaporation of the solvent at room temperature after several days (m.p. 413–415 K).

S3. Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with *d*(C—H) = 0.93 Å for aromatic, 0.97 Å for CH_2 and 0.96 Å for CH_3 atoms. The U_{iso}(H) values were constrained to be 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for the remaining H atoms. A rotating group model was used for the methyl groups.

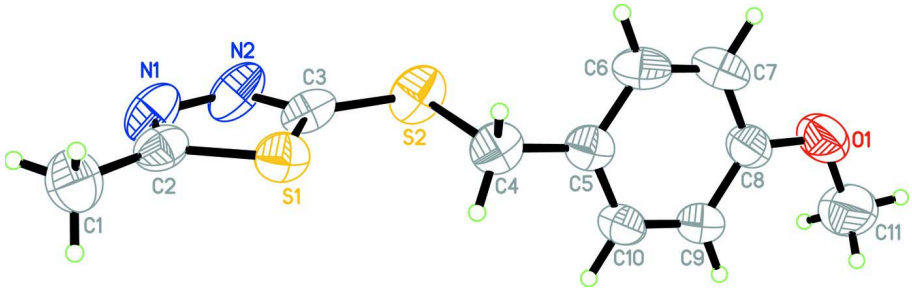

Figure 1

The molecular structure of the title compound, showing 40% probability displacement ellipsoids and the atom-numbering scheme.

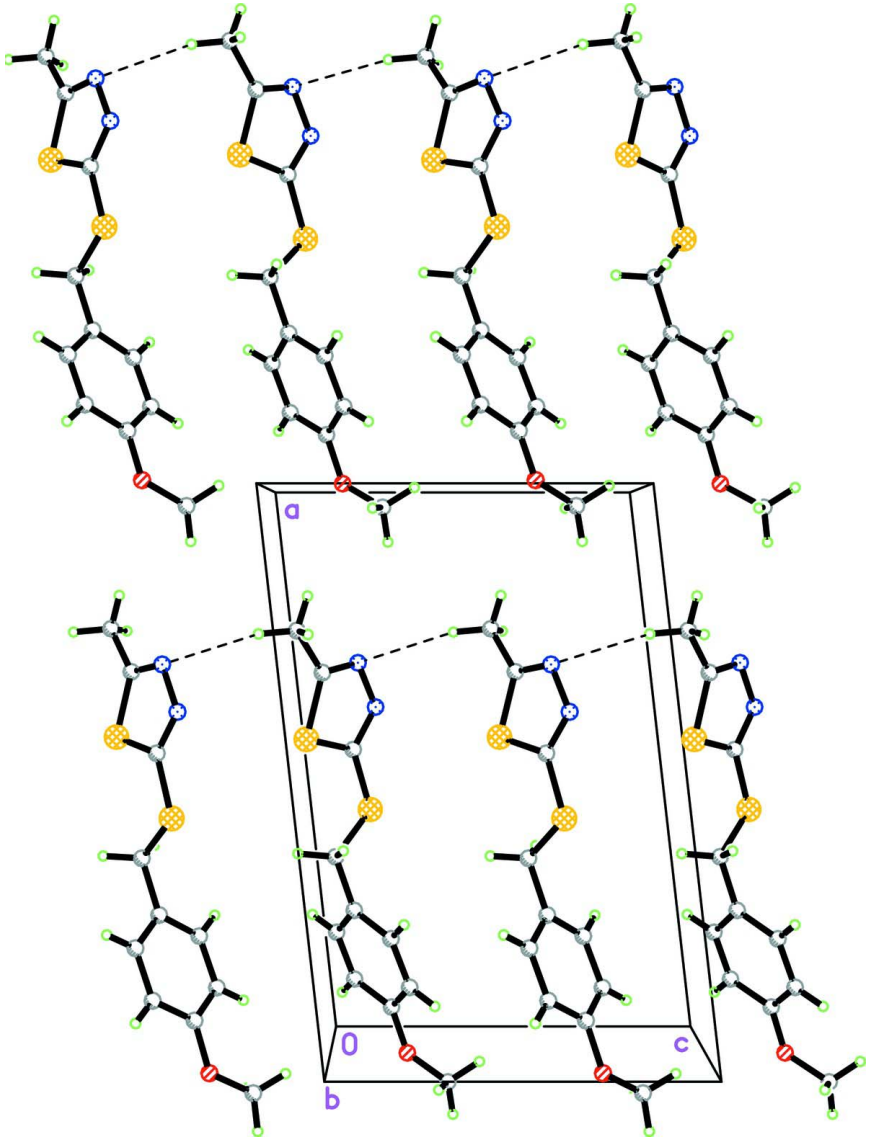

Figure 2

The crystal packing of the title compound viewed along the *b* axis. C—H···N weak interactions are shown as dashed lines.

5-[(4-Methoxybenzyl)sulfanyl]-2-methyl-1,3,4-thiadiazole

Crystal data

$C_{11}H_{12}N_2OS_2$
M_r = 252.35
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 14.7765 (4) Å
b = 8.6916 (3) Å
c = 9.7339 (3) Å
β = 96.477 (1)°
V = 1242.16 (7) Å^3
Z = 4
$F(000)$ = 528
D_x = 1.349 Mg m^{-3}
Melting point = 413–415 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2828 reflections
θ = 2.7–27.5°
μ = 0.41 mm^{-1}
T = 296 K
Plate, yellow
0.25 × 0.19 × 0.03 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.907, T_{max} = 0.987
11429 measured reflections
2828 independent reflections
1660 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 27.5°, θ_{min} = 2.7°
$h = -19 \rightarrow 19$
$k = -11 \rightarrow 11$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.118
S = 1.02
2828 reflections
147 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
$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.2899P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 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	0.43447 (5)	0.19481 (8)	0.95596 (7)	0.0631 (2)
S2	0.56474 (5)	0.40517 (9)	0.81756 (8)	0.0805 (3)
O1	0.99330 (14)	0.4254 (2)	0.7932 (2)	0.0899 (7)
N1	0.30610 (16)	0.3239 (3)	0.8041 (2)	0.0770 (7)

N2	0.38487 (18)	0.3952 (3)	0.7726 (2)	0.0795 (7)
C1	0.24763 (19)	0.1218 (4)	0.9455 (3)	0.0849 (9)
H1A	0.1893	0.1624	0.9098	0.127*
H1B	0.2534	0.1234	1.0447	0.127*
H1C	0.2529	0.0178	0.9141	0.127*
C2	0.32087 (18)	0.2174 (3)	0.8958 (2)	0.0616 (7)
C3	0.45726 (18)	0.3383 (3)	0.8427 (2)	0.0609 (7)
C4	0.63995 (18)	0.2718 (3)	0.9180 (3)	0.0672 (7)
H4A	0.6353	0.2848	1.0159	0.081*
H4B	0.6239	0.1665	0.8925	0.081*
C5	0.73488 (17)	0.3071 (3)	0.8867 (2)	0.0581 (7)
C6	0.7820 (2)	0.4335 (3)	0.9444 (3)	0.0697 (8)
H6A	0.7551	0.4956	1.0062	0.084*
C7	0.8673 (2)	0.4688 (3)	0.9122 (3)	0.0738 (8)
H7A	0.8977	0.5539	0.9528	0.089*
C8	0.90874 (18)	0.3794 (3)	0.8200 (3)	0.0624 (7)
C9	0.86360 (19)	0.2522 (3)	0.7635 (3)	0.0678 (7)
H9A	0.8909	0.1892	0.7029	0.081*
C10	0.77752 (18)	0.2183 (3)	0.7970 (3)	0.0653 (7)
H10A	0.7475	0.1324	0.7573	0.078*
C11	1.0358 (2)	0.3427 (5)	0.6936 (4)	0.1167 (14)
H11A	1.0964	0.3813	0.6905	0.175*
H11B	1.0014	0.3548	0.6046	0.175*
H11C	1.0385	0.2356	0.7180	0.175*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0756 (5)	0.0557 (4)	0.0557 (4)	0.0085 (3)	−0.0024 (3)	0.0090 (3)
S2	0.0880 (6)	0.0761 (6)	0.0793 (5)	0.0158 (4)	0.0181 (4)	0.0313 (4)
O1	0.0779 (14)	0.0861 (16)	0.1076 (16)	−0.0229 (11)	0.0186 (12)	−0.0254 (12)
N1	0.0787 (17)	0.0942 (19)	0.0594 (14)	0.0339 (14)	0.0139 (12)	0.0159 (14)
N2	0.0849 (17)	0.0896 (18)	0.0665 (15)	0.0385 (15)	0.0200 (13)	0.0277 (14)
C1	0.079 (2)	0.094 (2)	0.079 (2)	−0.0025 (18)	−0.0034 (16)	0.0020 (18)
C2	0.0744 (18)	0.0651 (18)	0.0449 (14)	0.0152 (14)	0.0056 (13)	−0.0067 (13)
C3	0.0806 (18)	0.0570 (17)	0.0466 (14)	0.0229 (14)	0.0137 (13)	0.0045 (12)
C4	0.0779 (19)	0.0640 (18)	0.0595 (16)	0.0065 (14)	0.0061 (14)	0.0151 (14)
C5	0.0708 (17)	0.0520 (16)	0.0507 (14)	0.0007 (13)	0.0035 (13)	0.0073 (13)
C6	0.099 (2)	0.0566 (18)	0.0563 (16)	−0.0032 (16)	0.0208 (15)	−0.0093 (14)
C7	0.099 (2)	0.0593 (18)	0.0631 (17)	−0.0209 (16)	0.0091 (16)	−0.0141 (15)
C8	0.0659 (17)	0.0571 (17)	0.0626 (17)	−0.0049 (14)	0.0002 (13)	−0.0044 (14)
C9	0.0701 (18)	0.0569 (17)	0.0758 (18)	0.0007 (14)	0.0062 (14)	−0.0168 (15)
C10	0.0701 (18)	0.0512 (17)	0.0726 (18)	−0.0057 (13)	−0.0011 (14)	−0.0122 (14)
C11	0.092 (2)	0.105 (3)	0.162 (4)	−0.014 (2)	0.051 (3)	−0.037 (3)

Geometric parameters (Å, º)

S1—C3	1.723 (3)	C4—H4B	0.9700
S1—C2	1.725 (3)	C5—C10	1.371 (3)
S2—C3	1.734 (3)	C5—C6	1.386 (4)
S2—C4	1.814 (3)	C6—C7	1.367 (4)
O1—C8	1.365 (3)	C6—H6A	0.9300
O1—C11	1.410 (3)	C7—C8	1.382 (4)
N1—C2	1.288 (3)	C7—H7A	0.9300
N1—N2	1.383 (3)	C8—C9	1.373 (4)
N2—C3	1.300 (3)	C9—C10	1.380 (3)
C1—C2	1.488 (4)	C9—H9A	0.9300
C1—H1A	0.9600	C10—H10A	0.9300
C1—H1B	0.9600	C11—H11A	0.9600
C1—H1C	0.9600	C11—H11B	0.9600
C4—C5	1.500 (3)	C11—H11C	0.9600
C4—H4A	0.9700		
C3—S1—C2	87.33 (13)	C10—C5—C4	121.5 (2)
C3—S2—C4	102.99 (12)	C6—C5—C4	121.2 (2)
C8—O1—C11	118.1 (2)	C7—C6—C5	121.3 (2)
C2—N1—N2	113.2 (2)	C7—C6—H6A	119.4
C3—N2—N1	112.1 (2)	C5—C6—H6A	119.4
C2—C1—H1A	109.5	C6—C7—C8	120.7 (3)
C2—C1—H1B	109.5	C6—C7—H7A	119.7
H1A—C1—H1B	109.5	C8—C7—H7A	119.7
C2—C1—H1C	109.5	O1—C8—C9	125.0 (2)
H1A—C1—H1C	109.5	O1—C8—C7	116.2 (2)
H1B—C1—H1C	109.5	C9—C8—C7	118.8 (3)
N1—C2—C1	123.7 (3)	C8—C9—C10	119.8 (3)
N1—C2—S1	113.5 (2)	C8—C9—H9A	120.1
C1—C2—S1	122.8 (2)	C10—C9—H9A	120.1
N2—C3—S1	113.7 (2)	C5—C10—C9	122.2 (2)
N2—C3—S2	120.7 (2)	C5—C10—H10A	118.9
S1—C3—S2	125.53 (16)	C9—C10—H10A	118.9
C5—C4—S2	106.83 (17)	O1—C11—H11A	109.5
C5—C4—H4A	110.4	O1—C11—H11B	109.5
S2—C4—H4A	110.4	H11A—C11—H11B	109.5
C5—C4—H4B	110.4	O1—C11—H11C	109.5
S2—C4—H4B	110.4	H11A—C11—H11C	109.5
H4A—C4—H4B	108.6	H11B—C11—H11C	109.5
C10—C5—C6	117.3 (2)		
C2—N1—N2—C3	0.2 (3)	S2—C4—C5—C6	76.6 (3)
N2—N1—C2—C1	−178.9 (2)	C10—C5—C6—C7	0.5 (4)
N2—N1—C2—S1	0.9 (3)	C4—C5—C6—C7	−177.7 (2)
C3—S1—C2—N1	−1.3 (2)	C5—C6—C7—C8	0.4 (4)
C3—S1—C2—C1	178.5 (2)	C11—O1—C8—C9	4.2 (4)

N1—N2—C3—S1	−1.3 (3)	C11—O1—C8—C7	−176.3 (3)
N1—N2—C3—S2	178.12 (18)	C6—C7—C8—O1	179.1 (2)
C2—S1—C3—N2	1.5 (2)	C6—C7—C8—C9	−1.4 (4)
C2—S1—C3—S2	−177.88 (18)	O1—C8—C9—C10	−179.1 (3)
C4—S2—C3—N2	−171.8 (2)	C7—C8—C9—C10	1.5 (4)
C4—S2—C3—S1	7.5 (2)	C6—C5—C10—C9	−0.4 (4)
C3—S2—C4—C5	172.25 (18)	C4—C5—C10—C9	177.8 (2)
S2—C4—C5—C10	−101.5 (3)	C8—C9—C10—C5	−0.6 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*B*···N1^{i}	0.96	2.59	3.532 (4)	164

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