Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Methyl-5-{[3-(4-methylphenyl)-1,2,4-oxadiazol-5-yl]methylsulfanyl}-1,3,4-thiadiazole

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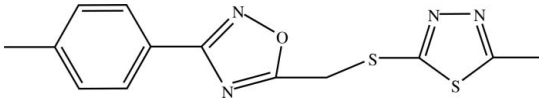
Received 12 November 2007; accepted 20 November 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.010 Å; R factor = 0.077; wR factor = 0.201; data-to-parameter ratio = 16.0.

In the title compound, $C_{13}H_{12}N_4OS_2$, the dihedral angle between the benzene and oxadiazole rings is 4.8 (3)°. The angle between the oxadiazole and thiadiazole rings is 85.6 (3)°.

Related literature

For related literature, see: Nicolaides *et al.* (1998); Romero (2001); Talar & Dejai (1996).



Experimental

Crystal data

$C_{13}H_{12}N_4OS_2$
M_r = 304.39
Monoclinic, $P2_1/c$
a = 16.751 (3) Å
b = 10.686 (2) Å
c = 8.0940 (16) Å
β = 97.12 (3)°
V = 1437.7 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 293 (2) K
0.40 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.866, T_{max} = 0.964
3018 measured reflections
2802 independent reflections
1621 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.202
S = 1.00
2802 reflections
175 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2658).

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  doi:10.1107/S1600536807061338

supplementary materials

Acta Cryst. (2007). E**63**, o4934 [**doi:10.1107/S1600536807061338**]

2-Methyl-5-{[3-(4-methylphenyl)-1,2,4-oxadiazol-5-yl]methylsulfanyl}-1,3,4-thiadiazole

P. Wang, H. Li, S. Kang, H. Zeng and H. Wang

Comment

1,2,4-Oxadiazoles represent an important class of five-membered heterocycles, some of which have anti-inflammatory (Nicolaides *et al.*, 1998) and antipicornaviral (Romero, 2001) properties. We are focusing our synthetic and structural studies on new oxindole derivatives. The sulfurether compound exhibited strong inhibiting activity to Staphylococcus aureus (Talar & Dejai, 1996). We report here the structure of its close analogue containing a thiadiazole sulfanylether group, (I).

There are three rings in the molecule. The benezene and oxadiazole ring close to coplanar [dihedral angle = 4.8 (3)°] due to the extended aromatic system. The angle between the oxadiazole and the thiadiazole planes is 85.6 (3)°.

Experimental

5-Mercapto-2-methyl-1,3,4-thiadiazole (20 mmol) was dissolved in an ethanol (70 ml)/water (70 mmol) mixture. Sodium acetate (20 mmol) and 3-[4-(methyl)phenyl]-5-chloromethyl-1,2,4-oxadiazol (40 mmol) was added. The resulting mixture was refluxed for 8 h. After cooling and filtrating, the crude title compound was obtained and purified by recrystallizing from a mixture of ethyl acetate (6 ml) and petrolum ether (4 ml). Colourless blocks of (I) were obstained by slow evaporation of an ethanol solution. ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 7.35–7.36 (m, 2H), 7.12–7.13(m, 2H), 4.18–4.19 (s, 2H), 2.35–2.36 (s,3*H*), 2.32–2.33 (s,3*H*).

Refinement

All the H atoms were placed geometrically (C—H = 0.93–0.96 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

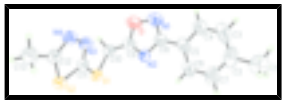

Fig. 1. A view of the molecular structure of (I), showing displacement ellipsoids for the non-hydrogen atoms at the 50% probability level.

2-Methyl-5-{[3-(4-methylphenyl)-1,2,4-oxadiazol-5-yl]methylsulfanyl}- 1,3,4-thiadiazole

Crystal data

$C_{13}H_{12}N_4OS_2$	F_{000} = 632
M_r = 304.39	D_x = 1.406 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å

Hall symbol: -P 2ybc	Cell parameters from 25 reflections
a = 16.751 (3) Å	θ = 9–12º
b = 10.686 (2) Å	μ = 0.37 mm^{-1}
c = 8.0940 (16) Å	T = 293 (2) K
β = 97.12 (3)º	Block, colourless
V = 1437.7 (5) $Å^3$	0.40 × 0.10 × 0.10 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.024
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 1.2º
T = 293(2) K	h = −20→20
ω/2θ scans	k = 0→13
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→9
T_{min} = 0.866, T_{max} = 0.964	3 standard reflections
3018 measured reflections	every 200 reflections
2802 independent reflections	intensity decay: none
1621 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.077	H-atom parameters constrained
$wR(F^2)$ = 0.202	$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 4.P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max} < 0.001$
2802 reflections	$\Delta\rho_{max}$ = 0.43 e $Å^{-3}$
175 parameters	$\Delta\rho_{min}$ = −0.29 e $Å^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.38091 (8)	0.27665 (13)	0.02659 (18)	0.0545 (4)
S2	0.54227 (9)	0.27974 (13)	0.2425 (2)	0.0595 (4)
O	0.2715 (3)	−0.0502 (4)	0.0223 (6)	0.0794 (13)
N1	0.2244 (4)	−0.0948 (5)	0.1441 (8)	0.0834 (17)
C1	0.0502 (5)	−0.0087 (8)	0.8173 (10)	0.102
H1B	0.0358	−0.0933	0.8408	0.154*
H1C	0.0025	0.0420	0.8016	0.154*
H1D	0.0862	0.0234	0.9089	0.154*
N2	0.2485 (3)	0.1100 (4)	0.1756 (6)	0.0549 (12)
C2	0.0910 (4)	−0.0061 (9)	0.6610 (9)	0.084 (2)
N3	0.4642 (3)	0.0792 (4)	0.1651 (6)	0.0604 (13)
C3	0.1020 (4)	−0.1118 (9)	0.5731 (11)	0.092 (2)
H3B	0.0819	−0.1870	0.6084	0.110*
N4	0.5348 (3)	0.0428 (4)	0.2610 (6)	0.0601 (13)
C4	0.1418 (4)	−0.1121 (6)	0.4343 (8)	0.0706 (17)
H4B	0.1498	−0.1867	0.3795	0.085*
C5	0.1703 (3)	0.0013 (6)	0.3761 (7)	0.0598 (15)
C6	0.1591 (3)	0.1093 (6)	0.4617 (8)	0.0650 (16)
H6A	0.1781	0.1850	0.4255	0.078*
C7	0.1197 (4)	0.1055 (8)	0.6020 (9)	0.084 (2)
H7A	0.1122	0.1795	0.6585	0.101*
C8	0.2138 (3)	0.0047 (5)	0.2277 (8)	0.0580 (15)
C9	0.2828 (3)	0.0735 (5)	0.0515 (7)	0.0513 (13)
C10	0.3277 (3)	0.1415 (5)	−0.0646 (7)	0.0531 (13)
H10A	0.2904	0.1684	−0.1593	0.064*
H10B	0.3661	0.0849	−0.1054	0.064*
C11	0.4594 (3)	0.1977 (5)	0.1446 (7)	0.0510 (13)
C12	0.5812 (3)	0.1350 (5)	0.3068 (8)	0.0633 (16)
C13	0.6623 (4)	0.1207 (6)	0.4094 (10)	0.083 (2)
H13A	0.6741	0.0334	0.4266	0.124*
H13B	0.6615	0.1611	0.5151	0.124*
H13C	0.7029	0.1584	0.3517	0.124*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0524 (8)	0.0498 (8)	0.0629 (9)	0.0081 (7)	0.0134 (6)	0.0052 (7)
S2	0.0516 (8)	0.0439 (8)	0.0832 (11)	−0.0021 (7)	0.0092 (7)	−0.0008 (7)
O	0.101 (4)	0.052 (3)	0.088 (3)	−0.007 (2)	0.023 (3)	−0.005 (2)
N1	0.099 (4)	0.059 (3)	0.096 (4)	−0.015 (3)	0.026 (4)	−0.014 (3)
C1	0.102	0.102	0.102	0.000	0.013	0.000
N2	0.049 (3)	0.054 (3)	0.063 (3)	−0.002 (2)	0.013 (2)	−0.008 (2)
C2	0.047 (4)	0.132 (7)	0.073 (5)	−0.002 (4)	0.009 (3)	0.008 (5)
N3	0.053 (3)	0.044 (3)	0.083 (3)	0.003 (2)	0.006 (2)	−0.009 (2)

C3	0.058 (4)	0.108 (6)	0.109 (6)	−0.011 (4)	0.011 (4)	0.038 (5)
N4	0.052 (3)	0.040 (3)	0.086 (3)	0.003 (2)	−0.003 (2)	0.005 (2)
C4	0.063 (4)	0.068 (4)	0.082 (5)	−0.007 (3)	0.012 (3)	0.008 (4)
C5	0.042 (3)	0.069 (4)	0.066 (4)	−0.010 (3)	−0.003 (3)	0.002 (3)
C6	0.053 (4)	0.068 (4)	0.075 (4)	−0.014 (3)	0.014 (3)	0.002 (3)
C7	0.061 (4)	0.110 (6)	0.084 (5)	−0.017 (4)	0.017 (4)	−0.020 (4)
C8	0.046 (3)	0.049 (3)	0.077 (4)	0.000 (3)	0.002 (3)	−0.003 (3)
C9	0.050 (3)	0.052 (3)	0.049 (3)	0.002 (3)	−0.005 (3)	0.000 (3)
C10	0.054 (3)	0.051 (3)	0.054 (3)	0.006 (3)	0.003 (3)	0.000 (3)
C11	0.045 (3)	0.050 (3)	0.061 (3)	0.001 (3)	0.016 (2)	−0.003 (3)
C12	0.052 (3)	0.047 (3)	0.093 (4)	0.007 (3)	0.016 (3)	−0.002 (3)
C13	0.053 (4)	0.061 (4)	0.130 (6)	0.005 (3)	−0.003 (4)	−0.005 (4)

Geometric parameters (Å, °)

S1—C11	1.744 (6)	C3—C4	1.376 (10)
S1—C10	1.806 (5)	C3—H3B	0.9300
S2—C12	1.733 (6)	N4—C12	1.282 (7)
S2—C11	1.747 (5)	C4—C5	1.405 (8)
O—C9	1.353 (7)	C4—H4B	0.9300
O—N1	1.420 (7)	C5—C6	1.370 (8)
N1—C8	1.284 (7)	C5—C8	1.481 (8)
C1—C2	1.511 (10)	C6—C7	1.383 (9)
C1—H1B	0.9600	C6—H6A	0.9300
C1—H1C	0.9600	C7—H7A	0.9300
C1—H1D	0.9600	C9—C10	1.467 (7)
N2—C9	1.278 (6)	C10—H10A	0.9700
N2—C8	1.358 (7)	C10—H10B	0.9700
C2—C3	1.359 (11)	C12—C13	1.509 (8)
C2—C7	1.392 (10)	C13—H13A	0.9600
N3—C11	1.278 (6)	C13—H13B	0.9600
N3—N4	1.387 (6)	C13—H13C	0.9600
C11—S1—C10	97.9 (3)	C6—C7—C2	121.8 (7)
C12—S2—C11	86.4 (3)	C6—C7—H7A	119.1
C9—O—N1	106.5 (4)	C2—C7—H7A	119.1
C8—N1—O	102.3 (5)	N1—C8—N2	115.4 (5)
C2—C1—H1B	109.5	N1—C8—C5	121.5 (6)
C2—C1—H1C	109.5	N2—C8—C5	123.0 (5)
H1B—C1—H1C	109.5	N2—C9—O	111.6 (5)
C2—C1—H1D	109.5	N2—C9—C10	132.0 (5)
H1B—C1—H1D	109.5	O—C9—C10	116.3 (5)
H1C—C1—H1D	109.5	C9—C10—S1	113.6 (4)
C9—N2—C8	104.1 (5)	C9—C10—H10A	108.8
C3—C2—C7	117.2 (7)	S1—C10—H10A	108.8
C3—C2—C1	121.8 (8)	C9—C10—H10B	108.8
C7—C2—C1	121.0 (8)	S1—C10—H10B	108.8
C11—N3—N4	112.7 (5)	H10A—C10—H10B	107.7
C2—C3—C4	122.7 (7)	N3—C11—S1	125.5 (4)
C2—C3—H3B	118.6	N3—C11—S2	113.9 (4)

C4—C3—H3B	118.6	S1—C11—S2	120.6 (3)
C12—N4—N3	113.0 (5)	N4—C12—C13	123.6 (5)
C3—C4—C5	119.4 (7)	N4—C12—S2	114.0 (4)
C3—C4—H4B	120.3	C13—C12—S2	122.4 (5)
C5—C4—H4B	120.3	C12—C13—H13A	109.5
C6—C5—C4	118.9 (6)	C12—C13—H13B	109.5
C6—C5—C8	120.1 (5)	H13A—C13—H13B	109.5
C4—C5—C8	120.9 (6)	C12—C13—H13C	109.5
C5—C6—C7	119.9 (6)	H13A—C13—H13C	109.5
C5—C6—H6A	120.0	H13B—C13—H13C	109.5
C7—C6—H6A	120.0		
C9—O—N1—C8	0.1 (6)	C4—C5—C8—N2	−174.0 (5)
C7—C2—C3—C4	1.9 (11)	C8—N2—C9—O	0.0 (6)
C1—C2—C3—C4	−177.4 (6)	C8—N2—C9—C10	177.0 (5)
C11—N3—N4—C12	−1.1 (7)	N1—O—C9—N2	−0.1 (6)
C2—C3—C4—C5	−2.1 (11)	N1—O—C9—C10	−177.6 (5)
C3—C4—C5—C6	1.4 (9)	N2—C9—C10—S1	30.9 (8)
C3—C4—C5—C8	179.6 (6)	O—C9—C10—S1	−152.3 (4)
C4—C5—C6—C7	−0.6 (9)	C11—S1—C10—C9	75.0 (4)
C8—C5—C6—C7	−178.8 (5)	N4—N3—C11—S1	178.0 (4)
C5—C6—C7—C2	0.4 (10)	N4—N3—C11—S2	−0.1 (6)
C3—C2—C7—C6	−1.0 (11)	C10—S1—C11—N3	−7.6 (6)
C1—C2—C7—C6	178.2 (6)	C10—S1—C11—S2	170.5 (3)
O—N1—C8—N2	−0.1 (7)	C12—S2—C11—N3	0.9 (5)
O—N1—C8—C5	−176.9 (5)	C12—S2—C11—S1	−177.3 (4)
C9—N2—C8—N1	0.1 (7)	N3—N4—C12—C13	−178.9 (6)
C9—N2—C8—C5	176.8 (5)	N3—N4—C12—S2	1.8 (7)
C6—C5—C8—N1	−179.3 (6)	C11—S2—C12—N4	−1.6 (5)
C4—C5—C8—N1	2.5 (9)	C11—S2—C12—C13	179.1 (6)
C6—C5—C8—N2	4.2 (8)		

Fig. 1