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2-Methyl-5-{3-[4-(methylsulfonyl)-phenyl]-1,2,4-oxadiazol-5-ylmethyl-sulfanyl}-1,3,4-thiadiazole

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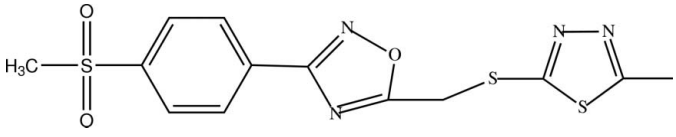
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.051; wR factor = 0.138; data-to-parameter ratio = 14.5.

The title compound, $C_{13}H_{12}N_4O_3S_3$, was synthesized *via* condensation of 1,2,4-oxadiazole chloromethane with 1,3,4-thiadiazolethiol. There are three rings in the molecule. The benzene and oxadiazole rings are coplanar due to the extended aromatic system. The angle between this plane and the thiadiazole plane is 82.2°.

Related literature

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



Experimental

Crystal data

$C_{13}H_{12}N_4O_3S_3$
M_r = 368.45
Monoclinic, $P2_1/c$
a = 12.365 (3) Å
b = 15.956 (3) Å
c = 8.2400 (16) Å
β = 108.26 (3)°
V = 1543.9 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.50 mm^{-1}
T = 293 (2) K
0.40 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.825, T_{max} = 0.907
3239 measured reflections
3019 independent reflections
2349 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.138
S = 1.07
3019 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 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: LW2039).

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supplementary materials

Acta Cryst. (2007). E**63**, o4356 [**doi:10.1107/S1600536807050313**]

2-Methyl-5-{3-[4-(methylsulfonyl)phenyl]-1,2,4-oxadiazol-5-ylmethylsulfanyl}-1,3,4-thiadiazole

P.-L. Wang, H.-S. Zeng, S.-S. Kang, H.-L. Li and H.-B. Wang

Comment

1,2,4-Oxadiazoles represent an important class of five-membered heterocycles. Some derivatives of 1,2,4-oxadiazoles 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 compounds exhibited considerably strong inhibiting activity to Staphylococcus aureus (Talar & Dejai, 1996). We report here the structure of its close analogue with thiadiazole sulfanylether group, (I). The benzene and oxadiazole rings are almost coplanar, howvever, the thiadiazole ring deviates from this plane. There are no classic hydrogen bonds in the molecular structure. The molecular structure of (I) is shown in Fig. 1.

Experimental

5-Mercapto-2-methyl-1,3,4-thiadiazole (20 mmol) was dissolve in ethanol (70 ml) and water (70 mmol). Sodium acetate (20 mmol) was added to this mixture. Then 3-[4-(Methylsulfonyl)phenyl]-5-chloromethyl-1,2,4-oxadiazol (40 mmol) was added. The resulting mixture was refluxed for 8 h. After cooling and filtrating, crude compound (I) was gained. Pure compound (I) was obtained by crystallizing from a mixture of ethyl acetate (8 ml) and petrolum ether (4 ml). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution. ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 7.95–7.99 (m, 2H), 7.75–7.76 (m, 2H), 4.17–4.18 (s, 2H), 2.85–2.86 (s,3*H*), 2.35–2.36 (s,3*H*).

Refinement

All H atoms bonded to the C atoms were placed geometrically at the distances of 0.93–0.96 Å and included in the refinement in riding motion approximation with U_{iso}(H) = 1.2 or 1.5U_{eq} of the carrier atom.

Figures

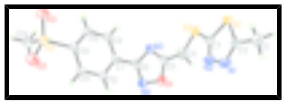


Fig. 1. A view of the molecular structure of (I), showing displacement ellipsoids at the 30% probability level.

2-Methyl-5-{3-[4-(methylsulfonyl)phenyl]-1,2,4-oxadiazol-5-ylmethylsulfanyl}- 1,3,4-thiadiazole

Crystal data

$C_{13}H_{12}N_4O_3S_3$	F_{000} = 760
M_r = 368.45	D_x = 1.585 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 = 12.365 (3) Å	θ = 10–13º
b = 15.956 (3) Å	μ = 0.50 mm^{-1}
c = 8.2400 (16) Å	T = 293 (2) K
β = 108.26 (3)º	Block, colourless
V = 1543.9 (6) Å^3	0.40 × 0.30 × 0.20 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.032
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 1.7º
T = 293(2) K	h = −15→14
ω/2θ scans	k = 0→19
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→10
T_{min} = 0.825, T_{max} = 0.907	3 standard reflections
3239 measured reflections	every 200 reflections
3019 independent reflections	intensity decay: none
2349 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
$R[F^2 > 2\sigma(F^2)]$ = 0.051	H-atom parameters constrained
$wR(F^2)$ = 0.138	$w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 0.5P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{max} < 0.001$
3019 reflections	$\Delta\rho_{max}$ = 0.37 e Å^{-3}
208 parameters	$\Delta\rho_{min}$ = −0.57 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	0.72521 (7)	0.34658 (5)	0.36586 (11)	0.0417 (2)
O1	0.4584 (2)	0.04592 (14)	0.1689 (3)	0.0481 (6)
N1	0.7921 (2)	0.19730 (17)	0.4507 (4)	0.0441 (7)
C1	0.9215 (3)	0.3017 (2)	0.6335 (5)	0.0467 (8)
H1B	0.9637	0.2532	0.6874	0.070*
H1C	0.8978	0.3329	0.7159	0.070*
H1D	0.9690	0.3363	0.5886	0.070*
S2	0.52374 (7)	0.27954 (6)	0.09389 (12)	0.0481 (3)
O2	0.1594 (2)	0.01484 (14)	0.9643 (3)	0.0488 (6)
N2	0.6927 (2)	0.18906 (16)	0.3141 (4)	0.0450 (7)
C2	0.8194 (3)	0.27461 (18)	0.4915 (4)	0.0372 (7)
S3	0.10797 (6)	0.07761 (4)	0.83848 (10)	0.0334 (2)
O3	0.1006 (2)	0.16209 (13)	0.8943 (3)	0.0482 (6)
N3	0.3686 (2)	0.15372 (15)	0.2358 (3)	0.0376 (6)
C3	0.6486 (3)	0.26122 (18)	0.2582 (4)	0.0363 (7)
N4	0.4067 (2)	0.01607 (16)	0.2870 (4)	0.0433 (6)
C4	0.4766 (3)	0.1743 (2)	0.0265 (4)	0.0487 (9)
H4B	0.4181	0.1771	−0.0840	0.058*
H4C	0.5401	0.1430	0.0120	0.058*
C5	0.4305 (3)	0.1279 (2)	0.1470 (4)	0.0399 (7)
C6	0.3552 (2)	0.08168 (17)	0.3201 (4)	0.0338 (6)
C7	0.2925 (2)	0.07993 (17)	0.4449 (4)	0.0340 (7)
C8	0.2290 (3)	0.14939 (19)	0.4627 (4)	0.0426 (8)
H8A	0.2247	0.1960	0.3933	0.051*
C9	0.1727 (3)	0.14945 (18)	0.5821 (4)	0.0394 (7)
H9A	0.1297	0.1956	0.5938	0.047*
C10	0.1810 (2)	0.07929 (17)	0.6852 (4)	0.0323 (6)
C11	0.2437 (3)	0.00997 (18)	0.6702 (4)	0.0374 (7)
H11A	0.2484	−0.0363	0.7407	0.045*
C13	−0.0297 (3)	0.0440 (2)	0.7251 (4)	0.0442 (8)
H13A	−0.0750	0.0415	0.8008	0.066*
H13B	−0.0262	−0.0106	0.6782	0.066*
H13C	−0.0636	0.0827	0.6342	0.066*
C12	0.2993 (2)	0.01041 (18)	0.5485 (4)	0.0376 (7)
H12A	0.3414	−0.0361	0.5361	0.045*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0426 (4)	0.0254 (4)	0.0617 (5)	0.0052 (3)	0.0227 (4)	0.0095 (3)
O1	0.0512 (14)	0.0434 (13)	0.0611 (15)	−0.0006 (11)	0.0341 (12)	−0.0035 (11)
N1	0.0510 (16)	0.0318 (14)	0.0530 (16)	0.0119 (12)	0.0215 (14)	0.0076 (12)
C1	0.0410 (18)	0.0450 (19)	0.056 (2)	0.0067 (15)	0.0188 (15)	0.0063 (16)
S2	0.0403 (5)	0.0470 (5)	0.0606 (5)	0.0017 (4)	0.0208 (4)	0.0177 (4)

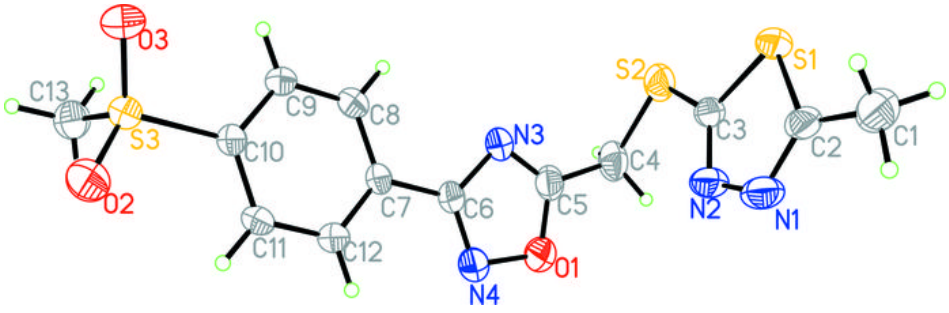
O2	0.0559 (14)	0.0431 (13)	0.0513 (13)	0.0099 (11)	0.0222 (11)	0.0146 (11)
N2	0.0509 (16)	0.0290 (13)	0.0609 (17)	0.0083 (12)	0.0258 (14)	0.0082 (13)
C2	0.0400 (16)	0.0305 (16)	0.0501 (18)	0.0080 (13)	0.0273 (14)	0.0082 (13)
S3	0.0354 (4)	0.0268 (4)	0.0415 (4)	0.0008 (3)	0.0172 (3)	0.0019 (3)
O3	0.0613 (15)	0.0302 (12)	0.0605 (14)	0.0002 (10)	0.0296 (12)	−0.0083 (10)
N3	0.0371 (14)	0.0346 (13)	0.0462 (15)	−0.0045 (11)	0.0205 (12)	0.0019 (11)
C3	0.0412 (16)	0.0275 (14)	0.0499 (18)	0.0026 (12)	0.0284 (14)	0.0057 (13)
N4	0.0460 (15)	0.0361 (14)	0.0564 (17)	−0.0041 (12)	0.0281 (13)	−0.0012 (12)
C4	0.048 (2)	0.058 (2)	0.0481 (19)	−0.0096 (17)	0.0268 (16)	−0.0005 (16)
C5	0.0350 (16)	0.0419 (18)	0.0459 (18)	−0.0076 (13)	0.0170 (14)	−0.0034 (14)
C6	0.0282 (14)	0.0314 (15)	0.0444 (16)	−0.0058 (12)	0.0153 (12)	−0.0019 (12)
C7	0.0269 (14)	0.0271 (15)	0.0519 (18)	−0.0045 (11)	0.0180 (13)	0.0013 (13)
C8	0.0475 (18)	0.0276 (15)	0.062 (2)	0.0043 (13)	0.0304 (16)	0.0120 (14)
C9	0.0408 (17)	0.0253 (14)	0.0605 (19)	0.0077 (13)	0.0280 (15)	0.0090 (14)
C10	0.0285 (14)	0.0266 (14)	0.0460 (16)	−0.0008 (11)	0.0178 (12)	0.0010 (12)
C11	0.0375 (15)	0.0229 (14)	0.0564 (19)	0.0032 (12)	0.0214 (14)	0.0085 (13)
C13	0.0377 (17)	0.0413 (18)	0.057 (2)	−0.0043 (14)	0.0192 (15)	−0.0019 (15)
C12	0.0354 (15)	0.0270 (15)	0.0555 (19)	0.0032 (12)	0.0215 (14)	0.0035 (13)

Geometric parameters (Å, °)

S1—C2	1.729 (3)	N4—C6	1.298 (4)
S1—C3	1.736 (3)	C4—C5	1.488 (4)
O1—C5	1.350 (4)	C4—H4B	0.9700
O1—N4	1.405 (3)	C4—H4C	0.9700
N1—C2	1.295 (4)	C6—C7	1.468 (4)
N1—N2	1.389 (4)	C7—C12	1.386 (4)
C1—C2	1.492 (5)	C7—C8	1.393 (4)
C1—H1B	0.9600	C8—C9	1.372 (4)
C1—H1C	0.9600	C8—H8A	0.9300
C1—H1D	0.9600	C9—C10	1.389 (4)
S2—C3	1.730 (3)	C9—H9A	0.9300
S2—C4	1.807 (4)	C10—C11	1.378 (4)
O2—S3	1.439 (2)	C11—C12	1.383 (4)
N2—C3	1.295 (4)	C11—H11A	0.9300
S3—O3	1.436 (2)	C13—H13A	0.9600
S3—C13	1.750 (3)	C13—H13B	0.9600
S3—C10	1.768 (3)	C13—H13C	0.9600
N3—C5	1.281 (4)	C12—H12A	0.9300
N3—C6	1.380 (4)		
C2—S1—C3	86.69 (15)	N3—C5—O1	114.1 (3)
C5—O1—N4	105.6 (2)	N3—C5—C4	130.0 (3)
C2—N1—N2	113.1 (3)	O1—C5—C4	115.9 (3)
C2—C1—H1B	109.5	N4—C6—N3	114.9 (3)
C2—C1—H1C	109.5	N4—C6—C7	122.2 (3)
H1B—C1—H1C	109.5	N3—C6—C7	122.8 (2)
C2—C1—H1D	109.5	C12—C7—C8	119.9 (3)
H1B—C1—H1D	109.5	C12—C7—C6	120.2 (3)
H1C—C1—H1D	109.5	C8—C7—C6	119.8 (3)

C3—S2—C4	101.93 (16)	C9—C8—C7	120.4 (3)
C3—N2—N1	111.8 (3)	C9—C8—H8A	119.8
N1—C2—C1	124.5 (3)	C7—C8—H8A	119.8
N1—C2—S1	113.9 (3)	C8—C9—C10	118.7 (3)
C1—C2—S1	121.6 (2)	C8—C9—H9A	120.6
O3—S3—O2	118.73 (15)	C10—C9—H9A	120.6
O3—S3—C13	108.04 (16)	C11—C10—C9	121.9 (3)
O2—S3—C13	107.96 (16)	C11—C10—S3	118.6 (2)
O3—S3—C10	108.20 (14)	C9—C10—S3	119.5 (2)
O2—S3—C10	108.40 (13)	C10—C11—C12	118.9 (3)
C13—S3—C10	104.63 (15)	C10—C11—H11A	120.6
C5—N3—C6	102.0 (3)	C12—C11—H11A	120.6
N2—C3—S2	127.0 (3)	S3—C13—H13A	109.5
N2—C3—S1	114.5 (3)	S3—C13—H13B	109.5
S2—C3—S1	118.54 (17)	H13A—C13—H13B	109.5
C6—N4—O1	103.4 (2)	S3—C13—H13C	109.5
C5—C4—S2	114.3 (2)	H13A—C13—H13C	109.5
C5—C4—H4B	108.7	H13B—C13—H13C	109.5
S2—C4—H4B	108.7	C11—C12—C7	120.2 (3)
C5—C4—H4C	108.7	C11—C12—H12A	119.9
S2—C4—H4C	108.7	C7—C12—H12A	119.9
H4B—C4—H4C	107.6		
C2—N1—N2—C3	−0.3 (4)	C5—N3—C6—C7	177.7 (3)
N2—N1—C2—C1	179.7 (3)	N4—C6—C7—C12	8.7 (4)
N2—N1—C2—S1	0.0 (3)	N3—C6—C7—C12	−168.0 (3)
C3—S1—C2—N1	0.3 (2)	N4—C6—C7—C8	−173.3 (3)
C3—S1—C2—C1	−179.5 (3)	N3—C6—C7—C8	10.0 (4)
N1—N2—C3—S2	−178.4 (2)	C12—C7—C8—C9	−0.1 (5)
N1—N2—C3—S1	0.6 (3)	C6—C7—C8—C9	−178.2 (3)
C4—S2—C3—N2	−0.4 (3)	C7—C8—C9—C10	0.4 (5)
C4—S2—C3—S1	−179.38 (17)	C8—C9—C10—C11	−0.2 (5)
C2—S1—C3—N2	−0.5 (2)	C8—C9—C10—S3	−179.4 (3)
C2—S1—C3—S2	178.59 (19)	O3—S3—C10—C11	150.6 (2)
C5—O1—N4—C6	0.3 (3)	O2—S3—C10—C11	20.6 (3)
C3—S2—C4—C5	73.2 (3)	C13—S3—C10—C11	−94.4 (3)
C6—N3—C5—O1	−0.6 (4)	O3—S3—C10—C9	−30.3 (3)
C6—N3—C5—C4	−179.9 (3)	O2—S3—C10—C9	−160.3 (2)
N4—O1—C5—N3	0.2 (4)	C13—S3—C10—C9	84.7 (3)
N4—O1—C5—C4	179.7 (3)	C9—C10—C11—C12	−0.2 (5)
S2—C4—C5—N3	38.5 (5)	S3—C10—C11—C12	178.9 (2)
S2—C4—C5—O1	−140.8 (3)	C10—C11—C12—C7	0.5 (5)
O1—N4—C6—N3	−0.6 (4)	C8—C7—C12—C11	−0.4 (5)
O1—N4—C6—C7	−177.6 (3)	C6—C7—C12—C11	177.7 (3)
C5—N3—C6—N4	0.8 (4)		

Fig. 1


O3
C13
S3
O2
C10
C9
C8
C7
C11
C12
N3
C6
C5
N4
O1
S2
C4
C3
S1
C2
C1
N2
N1