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# 2-Methyl-5-[3-(2-methylphenyl)-1,2,4-oxadiazol-5-ylmethylsulfanyl]-1,3,4-thiadiazole

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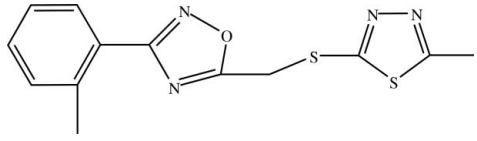
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.007 Å; $R$ factor = 0.062; $wR$ factor = 0.178; data-to-parameter ratio = 15.1.

In the title compound, $C_{13}H_{12}N_4OS_2$, the central oxadiazole ring makes dihedral angles of 8.9 (2) and 89.6 (3)° with the benzene and thiadiazole rings, respectively.

## 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, $P2_1/c$
$a$ = 10.309 (2) Å
$b$ = 12.992 (3) Å
$c$ = 10.600 (2) Å
$\beta$ = 100.63 (3)°
$V$ = 1395.3 (5) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.38 mm$^{-1}$
$T$ = 293 (2) K
0.30 × 0.10 × 0.10 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.894, $T_{max}$ = 0.963
2890 measured reflections
2737 independent reflections
1694 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032
3 standard reflections
every 200 reflections
intensity decay: none

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.178
$S$ = 1.03
2737 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.35 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: HB2618).

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# supplementary materials

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# 2-Methyl-5-[3-(2-methylphenyl)-1,2,4-oxadiazol-5-ylmethylsulfanyl]-1,3,4-thiadiazole

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

## Comment

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

There are three rings in the molecule. The benezene and oxadiazole ring are close to coplanar due to the extended aromatic system [dihedral angle = 8.9 (2)°]. The angle between the oxadiazole plane and the thiadiazole moiety is 89.6 (3)°. The molecular structure of (I) is shown in Fig. 1.

## Experimental

5-Mercapto-2-methyl-1,3,4-thiadiazole (20 mmol) was dissolved in ethanol (70 ml) and water (70 mmol). Sodium acetate (20 mmol) was added to this mixture. Then 3-[2-(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. Pure compound (I) was obstained by crystallizing from a mixture of ethyl acetate (8 ml) and petrolum ether (bp. 333–363 K) (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, 1H), 7.10–7.13(m, 3H), 4.18–4.19 (s, 2H), 2.38–2.39 (s,3*H*), 2.32–2.33 (s,3*H*).

## Refinement

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

## Figures

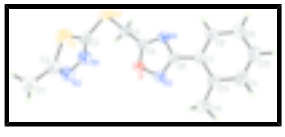

Fig. 1. A view of the molecular structure of (I), showing displacement ellipsoids at the 30% probability level (H atoms represented by arbitrary spheres).

## 2-Methyl-5-[3-(2-methylphenyl)-1,2,4-oxadiazol-5-ylmethylsulfanyl]- 1,3,4-thiadiazole

*Crystal data*

$C_{13}H_{12}N_4OS_2$ $F_{000}$ = 632

| | |
|---|---|
| $M_r$ = 304.39 | $D_x$ = 1.449 Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation<br>$\lambda$ = 0.71073 Å |
| Hall symbol: -P 2ybc | Cell parameters from 25 reflections |
| $a$ = 10.309 (2) Å | $\theta$ = 9–13º |
| $b$ = 12.992 (3) Å | $\mu$ = 0.38 mm$^{-1}$ |
| $c$ = 10.600 (2) Å | $T$ = 293 (2) K |
| $\beta$ = 100.63 (3)º | Block, colourless |
| $V$ = 1395.3 (5) Å$^3$ | 0.30 × 0.10 × 0.10 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Nonius CAD-4 diffractometer | $R_{int}$ = 0.032 |
| Radiation source: fine-focus sealed tube | $\theta_{max}$ = 26.0º |
| Monochromator: graphite | $\theta_{min}$ = 2.0º |
| $T$ = 293(2) K | $h$ = −12→12 |
| $\omega/2\theta$ scans | $k$ = 0→16 |
| Absorption correction: $\psi$ scan (North *et al.*, 1968) | $l$ = 0→13 |
| $T_{min}$ = 0.894, $T_{max}$ = 0.963 | 3 standard reflections |
| 2890 measured reflections | every 200 reflections |
| 2737 independent reflections | intensity decay: none |
| 1694 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 |
| $R[F^2 > 2\sigma(F^2)]$ = 0.062 | H-atom parameters constrained |
| $wR(F^2)$ = 0.178 | $w = 1/[\sigma^2(F_o^2) + (0.060P)^2 + 2.3P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 1.03 | $(\Delta/\sigma)_{max} < 0.001$ |
| 2737 reflections | $\Delta\rho_{max}$ = 0.29 e Å$^{-3}$ |
| 181 parameters | $\Delta\rho_{min}$ = −0.35 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O | 0.1434 (4) | 0.1828 (2) | 0.0705 (3) | 0.0684 (10) |
| S1 | 0.53469 (12) | 0.38060 (11) | 0.12681 (13) | 0.0669 (4) |
| N1 | 0.2889 (4) | 0.3910 (3) | 0.0430 (4) | 0.0695 (12) |
| C1 | 0.4913 (6) | 0.5355 (4) | 0.3034 (5) | 0.0754 (15) |
| H1B | 0.4209 | 0.5770 | 0.3239 | 0.113* |
| H1C | 0.5582 | 0.5793 | 0.2804 | 0.113* |
| H1D | 0.5286 | 0.4947 | 0.3766 | 0.113* |
| S2 | 0.39670 (13) | 0.24762 (10) | −0.09151 (13) | 0.0618 (4) |
| N2 | 0.3157 (4) | 0.4621 (3) | 0.1412 (4) | 0.0640 (11) |
| C2 | 0.4386 (4) | 0.4665 (3) | 0.1935 (4) | 0.0492 (10) |
| N3 | 0.1246 (5) | 0.0994 (3) | 0.1516 (4) | 0.0687 (12) |
| C3 | 0.3944 (4) | 0.3437 (3) | 0.0250 (4) | 0.0506 (11) |
| N4 | 0.2023 (3) | 0.0434 (3) | −0.0210 (3) | 0.0462 (8) |
| C4 | 0.2240 (4) | 0.2126 (4) | −0.1264 (4) | 0.0526 (11) |
| H4B | 0.1706 | 0.2743 | −0.1294 | 0.063* |
| H4C | 0.2041 | 0.1801 | −0.2101 | 0.063* |
| C5 | 0.1892 (4) | 0.1415 (3) | −0.0289 (4) | 0.0436 (9) |
| C6 | 0.1615 (4) | 0.0195 (3) | 0.0926 (4) | 0.0423 (9) |
| C7 | 0.1625 (4) | −0.0868 (3) | 0.1392 (4) | 0.0421 (9) |
| C8 | 0.2228 (5) | −0.1601 (3) | 0.0731 (4) | 0.0520 (11) |
| H8A | 0.2607 | −0.1397 | 0.0041 | 0.062* |
| C9 | 0.2273 (5) | −0.2620 (4) | 0.1082 (5) | 0.0612 (13) |
| H9A | 0.2672 | −0.3102 | 0.0628 | 0.073* |
| C10 | 0.1719 (5) | −0.2925 (4) | 0.2116 (5) | 0.0622 (13) |
| H10A | 0.1757 | −0.3611 | 0.2367 | 0.075* |
| C11 | 0.1115 (5) | −0.2215 (4) | 0.2769 (5) | 0.0592 (12) |
| H11A | 0.0727 | −0.2438 | 0.3446 | 0.071* |
| C12 | 0.1061 (4) | −0.1176 (3) | 0.2458 (4) | 0.0447 (10) |
| C13 | 0.0405 (5) | −0.0452 (4) | 0.3241 (5) | 0.0636 (13) |
| H13A | 0.0105 | −0.0827 | 0.3913 | 0.095* |
| H13B | −0.0335 | −0.0131 | 0.2701 | 0.095* |
| H13C | 0.1023 | 0.0067 | 0.3610 | 0.095* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O | 0.110 (3) | 0.0366 (16) | 0.074 (2) | 0.0038 (17) | 0.058 (2) | −0.0004 (15) |
| S1 | 0.0509 (7) | 0.0809 (10) | 0.0723 (8) | 0.0051 (6) | 0.0203 (6) | −0.0044 (7) |
| N1 | 0.062 (3) | 0.055 (3) | 0.097 (3) | 0.003 (2) | 0.028 (2) | −0.012 (2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C1 | 0.086 (4) | 0.072 (4) | 0.071 (3) | −0.008 (3) | 0.021 (3) | −0.014 (3) |
| S2 | 0.0675 (8) | 0.0563 (7) | 0.0718 (8) | −0.0028 (6) | 0.0393 (6) | −0.0030 (6) |
| N2 | 0.062 (3) | 0.054 (2) | 0.079 (3) | 0.006 (2) | 0.021 (2) | −0.010 (2) |
| C2 | 0.046 (2) | 0.055 (3) | 0.048 (2) | 0.000 (2) | 0.0133 (19) | 0.008 (2) |
| N3 | 0.110 (3) | 0.040 (2) | 0.072 (3) | −0.001 (2) | 0.059 (3) | 0.000 (2) |
| C3 | 0.049 (2) | 0.049 (3) | 0.060 (3) | 0.003 (2) | 0.025 (2) | 0.014 (2) |
| N4 | 0.064 (2) | 0.0383 (19) | 0.0408 (19) | 0.0034 (17) | 0.0222 (16) | −0.0005 (15) |
| C4 | 0.067 (3) | 0.049 (3) | 0.044 (2) | 0.002 (2) | 0.018 (2) | 0.003 (2) |
| C5 | 0.046 (2) | 0.048 (2) | 0.040 (2) | 0.0010 (19) | 0.0138 (17) | −0.0027 (19) |
| C6 | 0.049 (2) | 0.039 (2) | 0.043 (2) | 0.0028 (18) | 0.0196 (18) | 0.0031 (18) |
| C7 | 0.047 (2) | 0.039 (2) | 0.043 (2) | −0.0015 (18) | 0.0151 (18) | 0.0009 (18) |
| C8 | 0.068 (3) | 0.042 (2) | 0.054 (3) | −0.003 (2) | 0.031 (2) | −0.003 (2) |
| C9 | 0.077 (3) | 0.042 (3) | 0.070 (3) | 0.008 (2) | 0.029 (3) | 0.002 (2) |
| C10 | 0.070 (3) | 0.037 (2) | 0.083 (4) | −0.001 (2) | 0.021 (3) | 0.011 (2) |
| C11 | 0.056 (3) | 0.061 (3) | 0.066 (3) | −0.005 (2) | 0.024 (2) | 0.017 (2) |
| C12 | 0.041 (2) | 0.050 (2) | 0.045 (2) | 0.0001 (19) | 0.0127 (17) | 0.0076 (19) |
| C13 | 0.073 (3) | 0.064 (3) | 0.064 (3) | −0.003 (3) | 0.039 (3) | 0.003 (2) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O—C5 | 1.343 (5) | C4—H4B | 0.9700 |
| O—N3 | 1.418 (5) | C4—H4C | 0.9700 |
| S1—C3 | 1.706 (5) | C6—C7 | 1.467 (5) |
| S1—C2 | 1.727 (5) | C7—C8 | 1.394 (6) |
| N1—C3 | 1.293 (5) | C7—C12 | 1.420 (5) |
| N1—N2 | 1.381 (6) | C8—C9 | 1.373 (6) |
| C1—C2 | 1.491 (6) | C8—H8A | 0.9300 |
| C1—H1B | 0.9600 | C9—C10 | 1.383 (6) |
| C1—H1C | 0.9600 | C9—H9A | 0.9300 |
| C1—H1D | 0.9600 | C10—C11 | 1.370 (7) |
| S2—C3 | 1.759 (5) | C10—H10A | 0.9300 |
| S2—C4 | 1.809 (5) | C11—C12 | 1.389 (6) |
| N2—C2 | 1.287 (6) | C11—H11A | 0.9300 |
| N3—C6 | 1.304 (5) | C12—C13 | 1.496 (6) |
| N4—C5 | 1.283 (5) | C13—H13A | 0.9600 |
| N4—C6 | 1.382 (5) | C13—H13B | 0.9600 |
| C4—C5 | 1.479 (6) | C13—H13C | 0.9600 |
| C5—O—N3 | 106.1 (3) | N3—C6—N4 | 113.6 (4) |
| C3—S1—C2 | 87.5 (2) | N3—C6—C7 | 125.0 (4) |
| C3—N1—N2 | 111.8 (4) | N4—C6—C7 | 121.4 (3) |
| C2—C1—H1B | 109.5 | C8—C7—C12 | 119.6 (4) |
| C2—C1—H1C | 109.5 | C8—C7—C6 | 116.8 (4) |
| H1B—C1—H1C | 109.5 | C12—C7—C6 | 123.7 (4) |
| C2—C1—H1D | 109.5 | C9—C8—C7 | 121.2 (4) |
| H1B—C1—H1D | 109.5 | C9—C8—H8A | 119.4 |
| H1C—C1—H1D | 109.5 | C7—C8—H8A | 119.4 |
| C3—S2—C4 | 100.5 (2) | C8—C9—C10 | 119.5 (4) |
| C2—N2—N1 | 113.5 (4) | C8—C9—H9A | 120.3 |
| N2—C2—C1 | 123.4 (4) | C10—C9—H9A | 120.3 |

| | | | |
|---|---|---|---|
| N2—C2—S1 | 112.8 (3) | C11—C10—C9 | 120.0 (4) |
| C1—C2—S1 | 123.7 (4) | C11—C10—H10A | 120.0 |
| C6—N3—O | 103.5 (3) | C9—C10—H10A | 120.0 |
| N1—C3—S1 | 114.3 (4) | C10—C11—C12 | 122.5 (4) |
| N1—C3—S2 | 124.1 (4) | C10—C11—H11A | 118.8 |
| S1—C3—S2 | 121.6 (3) | C12—C11—H11A | 118.8 |
| C5—N4—C6 | 103.6 (3) | C11—C12—C7 | 117.2 (4) |
| C5—C4—S2 | 111.8 (3) | C11—C12—C13 | 118.8 (4) |
| C5—C4—H4B | 109.3 | C7—C12—C13 | 123.9 (4) |
| S2—C4—H4B | 109.3 | C12—C13—H13A | 109.5 |
| C5—C4—H4C | 109.3 | C12—C13—H13B | 109.5 |
| S2—C4—H4C | 109.3 | H13A—C13—H13B | 109.5 |
| H4B—C4—H4C | 107.9 | C12—C13—H13C | 109.5 |
| N4—C5—O | 113.2 (4) | H13A—C13—H13C | 109.5 |
| N4—C5—C4 | 129.0 (4) | H13B—C13—H13C | 109.5 |
| O—C5—C4 | 117.6 (4) | | |
| C3—N1—N2—C2 | 0.1 (6) | O—N3—C6—N4 | −0.4 (5) |
| N1—N2—C2—C1 | −178.5 (4) | O—N3—C6—C7 | 179.1 (4) |
| N1—N2—C2—S1 | −0.8 (5) | C5—N4—C6—N3 | 0.2 (5) |
| C3—S1—C2—N2 | 1.0 (4) | C5—N4—C6—C7 | −179.3 (4) |
| C3—S1—C2—C1 | 178.6 (4) | N3—C6—C7—C8 | −171.0 (5) |
| C5—O—N3—C6 | 0.4 (5) | N4—C6—C7—C8 | 8.4 (6) |
| N2—N1—C3—S1 | 0.6 (5) | N3—C6—C7—C12 | 9.0 (7) |
| N2—N1—C3—S2 | 179.6 (3) | N4—C6—C7—C12 | −171.6 (4) |
| C2—S1—C3—N1 | −0.9 (4) | C12—C7—C8—C9 | 0.9 (7) |
| C2—S1—C3—S2 | −179.9 (3) | C6—C7—C8—C9 | −179.1 (4) |
| C4—S2—C3—N1 | −17.8 (4) | C7—C8—C9—C10 | −0.5 (8) |
| C4—S2—C3—S1 | 161.1 (3) | C8—C9—C10—C11 | 1.0 (8) |
| C3—S2—C4—C5 | −80.5 (3) | C9—C10—C11—C12 | −1.8 (8) |
| C6—N4—C5—O | 0.1 (5) | C10—C11—C12—C7 | 2.1 (7) |
| C6—N4—C5—C4 | 176.4 (4) | C10—C11—C12—C13 | −178.8 (5) |
| N3—O—C5—N4 | −0.4 (5) | C8—C7—C12—C11 | −1.6 (6) |
| N3—O—C5—C4 | −177.1 (4) | C6—C7—C12—C11 | 178.4 (4) |
| S2—C4—C5—N4 | −83.2 (5) | C8—C7—C12—C13 | 179.3 (4) |
| S2—C4—C5—O | 92.9 (4) | C6—C7—C12—C13 | −0.7 (7) |

**Fig. 1**