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2-Methyl-5-[3-(4-nitrophenyl)-1,2,4-oxadiazol-5-ylmethylenesulfanyl]-1,3,4-thiadiazole

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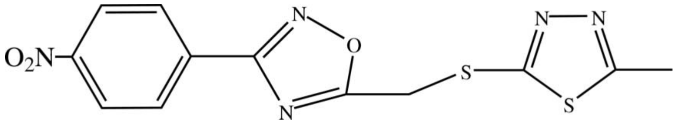
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.064; wR factor = 0.212; data-to-parameter ratio = 13.6.

The title compound, $C_{12}H_9N_5O_3S_2$, was synthesized *via* condensation of 1,2,4-oxadiazole chloromethane with 1,3,4-thiadiazolethiol. The benzene and oxadiazole rings are almost coplanar, making a twist angle of only 4.6 (3)°, but the thiadiazole ring deviates from the molecular plane, making a dihedral angle of 87.9 (3)° with the oxadiazole ring.

Related literature

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



Experimental

Crystal data

$C_{12}H_9N_5O_3S_2$
M_r = 335.36
Monoclinic, $C2/c$
a = 35.939 (7) Å
b = 5.6540 (11) Å
c = 13.714 (3) Å
β = 92.55 (3)°
V = 2783.9 (10) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.40 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} = 0.855, T_{max} = 0.924
2772 measured reflections
2727 independent reflections
1965 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
3 standard reflections every 200 reflections intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.212
S = 1.10
2727 reflections
200 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 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: LW2036).

References

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Nicolaides, D. N., Fylaktakidou, K. C., Litinas, K. E. & Hadjipavlou-Litina, D. (1998). *Eur. J. Med. Chem.* **33**, 715–724.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Romero, J. R. (2001). *Expert Opin. Investig. Drugs*, **10**, 369–379.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Siemens (1996). *SHELXTL*. Version 5.06. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Talar, M. B. & Dejai, S. R. (1996). *Indian J. Heterocycl. Chem.* **5**, 215–218.

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supplementary materials

Acta Cryst. (2007). E**63**, o4236 [**doi:10.1107/S1600536807047812**]

2-Methyl-5-[3-(4-nitrophenyl)-1,2,4-oxadiazol-5-ylmethylenesulfanyl]-1,3,4-thiadiazole

P.-L. Wang, S.-S. Kang, H.-L. Li, H.-S. Zeng 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). This compound crystallizes in the monoclinic system,space group *C*2/*c*. There are three rings in the molecule. The benzene and oxadiazole rings are nearly coplanar, but the thiadiazole ring deviates from the molecular plane. There is no classic hydrogen bond in the molecular structure. The molecular structure of (I) is shown in Fig. 1. The bond lengths and angles are given in Table 1.

Experimental

5-Mercapto-2-methyl-1,3,4-thiadiazole (30 mmol) was dissolve in ethanol (70 ml) and water (70 mmol). Sodium acetate (30 mmol) was added to this mixture. Then 3-[4-(nitro)phenyl]-5-chloromethyl-1,2,4-oxadiazol (50 mmol) was added. The resulting mixture was refluxed for 8 h. After cooling and filtrating, crude compound (I) was gained. Pure compound (I) was obstained by crystallizing from a mixture of ethyl acetate (6 ml) and petrolum ether (6 ml). Crystals of (I) suitable for X-ray diffraction were obstained by slow evaporation of an ethanol solution. ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 8.25–8.29 (m, 2H), 7.72–7.75 (m, 2H), 4.81–4.82 (s, 2H), 2.36–2.38 (s, 3H).

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

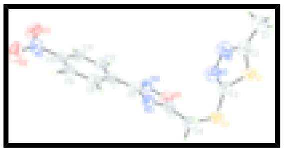

Fig. 1. A view of the molecular structure of (I), showing displacement ellipsoids at the 30% probability level.

2-Methyl-5-[3-(4-nitrophenyl)-1,2,4-oxadiazol-5-ylmethylenesulfanyl]-1,3,4- thiadiazole

Crystal data

$C_{12}H_9N_5O_3S_2$	F_{000} = 1376

$M_r = 335.36$

Monoclinic, $C2/c$

Hall symbol: -C 2yc

$a = 35.939$ (7) Å

$b = 5.6540$ (11) Å

$c = 13.714$ (3) Å

$\beta = 92.55$ (3)°

$V = 2783.9$ (10) Å^3

$Z = 8$

$D_x = 1.600$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 9$–13°

$\mu = 0.40$ mm^{-1}

$T = 293$ (2) K

Block, colourless

0.40 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293(2)$ K

$\omega/2\theta$ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.855$, $T_{\max} = 0.924$

2772 measured reflections

2727 independent reflections

1965 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.033$

$\theta_{\max} = 26.0$°

$\theta_{\min} = 1.1$°

$h = -44 \rightarrow 44$

$k = 0 \rightarrow 6$

$l = 0 \rightarrow 16$

3 standard reflections

every 200 reflections

intensity decay: none

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.064$

$wR(F^2) = 0.212$

$S = 1.10$

2727 reflections

200 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 1P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.30$ e Å^{-3}

$\Delta\rho_{\min} = -0.31$ e Å^{-3}

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	0.48917 (3)	0.5272 (2)	0.62377 (9)	0.0525 (4)
S2	0.56945 (4)	0.6940 (2)	0.64667 (10)	0.0565 (4)
O1	0.61049 (9)	0.4474 (6)	0.4886 (2)	0.0542 (9)
O2	0.75292 (12)	−0.7027 (8)	0.3772 (3)	0.0766 (12)
O3	0.72006 (12)	−0.6820 (7)	0.2417 (3)	0.0709 (11)
N1	0.51318 (12)	0.1027 (8)	0.6228 (3)	0.0598 (11)
N2	0.54470 (11)	0.2462 (8)	0.6310 (3)	0.0554 (10)
N3	0.64182 (10)	0.1824 (7)	0.5757 (3)	0.0465 (9)
N4	0.62608 (11)	0.2924 (7)	0.4218 (3)	0.0516 (10)
N5	0.72802 (12)	−0.6150 (8)	0.3256 (3)	0.0541 (10)
C1	0.44496 (15)	0.1124 (11)	0.6123 (4)	0.0652 (14)
H1B	0.4420	0.0103	0.6673	0.098*
H1C	0.4425	0.0218	0.5532	0.098*
H1D	0.4262	0.2334	0.6115	0.098*
C2	0.48254 (14)	0.2238 (9)	0.6198 (3)	0.0522 (12)
C3	0.53603 (13)	0.4704 (8)	0.6337 (3)	0.0465 (11)
C4	0.61043 (14)	0.5081 (10)	0.6631 (4)	0.0594 (13)
H4B	0.6314	0.6079	0.6830	0.071*
H4C	0.6064	0.3991	0.7162	0.071*
C5	0.62069 (12)	0.3713 (8)	0.5783 (3)	0.0447 (10)
C6	0.64432 (12)	0.1382 (8)	0.4777 (3)	0.0447 (10)
C7	0.66602 (11)	−0.0549 (7)	0.4390 (3)	0.0401 (9)
C8	0.68800 (12)	−0.2013 (8)	0.5007 (3)	0.0462 (11)
H8A	0.6887	−0.1742	0.5677	0.055*
C9	0.70838 (12)	−0.3833 (9)	0.4645 (3)	0.0488 (11)
H9A	0.7227	−0.4804	0.5060	0.059*
C10	0.70704 (12)	−0.4188 (8)	0.3649 (3)	0.0445 (10)
C11	0.68665 (13)	−0.2755 (8)	0.3014 (3)	0.0482 (11)
H11A	0.6870	−0.3000	0.2344	0.058*
C12	0.66587 (12)	−0.0965 (9)	0.3382 (3)	0.0481 (11)
H12A	0.6515	−0.0015	0.2959	0.058*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0564 (7)	0.0482 (7)	0.0516 (7)	0.0156 (6)	−0.0111 (5)	−0.0032 (5)
S2	0.0608 (8)	0.0420 (7)	0.0656 (8)	0.0118 (6)	−0.0108 (6)	−0.0096 (6)
O1	0.0568 (19)	0.0386 (18)	0.065 (2)	0.0108 (15)	−0.0173 (16)	0.0030 (16)
O2	0.082 (3)	0.070 (3)	0.078 (3)	0.031 (2)	0.000 (2)	0.006 (2)

O3	0.091 (3)	0.055 (2)	0.066 (2)	0.003 (2)	−0.002 (2)	−0.0173 (19)
N1	0.058 (3)	0.044 (2)	0.077 (3)	0.009 (2)	−0.005 (2)	0.007 (2)
N2	0.048 (2)	0.047 (2)	0.070 (3)	0.0111 (19)	−0.0053 (19)	0.006 (2)
N3	0.045 (2)	0.046 (2)	0.047 (2)	0.0083 (17)	−0.0105 (16)	−0.0058 (17)
N4	0.055 (2)	0.044 (2)	0.055 (2)	0.0066 (19)	−0.0124 (17)	0.0054 (19)
N5	0.055 (2)	0.043 (2)	0.064 (3)	0.0014 (19)	0.001 (2)	−0.003 (2)
C1	0.061 (3)	0.061 (3)	0.073 (3)	0.006 (3)	−0.010 (3)	0.014 (3)
C2	0.055 (3)	0.054 (3)	0.046 (2)	0.016 (2)	−0.010 (2)	0.003 (2)
C3	0.051 (3)	0.048 (3)	0.040 (2)	0.011 (2)	−0.0060 (18)	−0.002 (2)
C4	0.052 (3)	0.056 (3)	0.068 (3)	0.014 (2)	−0.017 (2)	−0.018 (3)
C5	0.041 (2)	0.041 (2)	0.051 (2)	−0.003 (2)	−0.0118 (18)	−0.002 (2)
C6	0.045 (2)	0.039 (2)	0.049 (2)	−0.012 (2)	−0.0130 (18)	0.0035 (19)
C7	0.039 (2)	0.035 (2)	0.045 (2)	−0.0041 (18)	−0.0117 (17)	−0.0029 (18)
C8	0.050 (2)	0.049 (3)	0.038 (2)	−0.007 (2)	−0.0134 (18)	0.008 (2)
C9	0.045 (2)	0.045 (3)	0.055 (3)	0.003 (2)	−0.017 (2)	0.007 (2)
C10	0.041 (2)	0.039 (2)	0.053 (2)	−0.0056 (19)	−0.0114 (18)	0.002 (2)
C11	0.053 (3)	0.045 (3)	0.046 (2)	−0.003 (2)	−0.011 (2)	0.001 (2)
C12	0.047 (2)	0.054 (3)	0.042 (2)	0.003 (2)	−0.0132 (18)	0.010 (2)

Geometric parameters (Å, °)

S1—C3	1.714 (5)	C1—H1C	0.9600
S1—C2	1.732 (6)	C1—H1D	0.9600
S2—C3	1.747 (5)	C4—C5	1.457 (7)
S2—C4	1.815 (5)	C4—H4B	0.9700
O1—C5	1.340 (5)	C4—H4C	0.9700
O1—N4	1.402 (5)	C6—C7	1.456 (6)
O2—N5	1.221 (5)	C7—C8	1.402 (6)
O3—N5	1.232 (5)	C7—C12	1.402 (6)
N1—C2	1.296 (6)	C8—C9	1.369 (7)
N1—N2	1.394 (6)	C8—H8A	0.9300
N2—C3	1.307 (6)	C9—C10	1.380 (6)
N3—C5	1.312 (6)	C9—H9A	0.9300
N3—C6	1.374 (6)	C10—C11	1.376 (6)
N4—C6	1.316 (6)	C11—C12	1.367 (7)
N5—C10	1.458 (6)	C11—H11A	0.9300
C1—C2	1.490 (7)	C12—H12A	0.9300
C1—H1B	0.9600		

C3—S1—C2	87.2 (2)	S2—C4—H4C	108.2
C3—S2—C4	98.3 (2)	H4B—C4—H4C	107.4
C5—O1—N4	107.4 (3)	N3—C5—O1	111.8 (4)
C2—N1—N2	112.4 (4)	N3—C5—C4	128.4 (4)
C3—N2—N1	111.9 (4)	O1—C5—C4	119.6 (4)
C5—N3—C6	103.8 (4)	N4—C6—N3	113.4 (4)
C6—N4—O1	103.7 (4)	N4—C6—C7	123.1 (4)
O2—N5—O3	123.3 (4)	N3—C6—C7	123.5 (4)
O2—N5—C10	118.2 (4)	C8—C7—C12	118.3 (4)
O3—N5—C10	118.4 (4)	C8—C7—C6	121.3 (4)
C2—C1—H1B	109.5	C12—C7—C6	120.4 (4)

C2—C1—H1C	109.5	C9—C8—C7	121.4 (4)
H1B—C1—H1C	109.5	C9—C8—H8A	119.3
C2—C1—H1D	109.5	C7—C8—H8A	119.3
H1B—C1—H1D	109.5	C8—C9—C10	118.2 (4)
H1C—C1—H1D	109.5	C8—C9—H9A	120.9
N1—C2—C1	123.0 (5)	C10—C9—H9A	120.9
N1—C2—S1	114.0 (4)	C11—C10—C9	122.3 (4)
C1—C2—S1	123.0 (4)	C11—C10—N5	118.9 (4)
N2—C3—S1	114.5 (4)	C9—C10—N5	118.7 (4)
N2—C3—S2	122.8 (4)	C12—C11—C10	119.1 (4)
S1—C3—S2	122.8 (3)	C12—C11—H11A	120.4
C5—C4—S2	116.2 (4)	C10—C11—H11A	120.4
C5—C4—H4B	108.2	C11—C12—C7	120.6 (4)
S2—C4—H4B	108.2	C11—C12—H12A	119.7
C5—C4—H4C	108.2	C7—C12—H12A	119.7

C2—N1—N2—C3	0.0 (6)	C5—N3—C6—N4	0.7 (5)
C5—O1—N4—C6	−0.2 (4)	C5—N3—C6—C7	178.6 (4)
N2—N1—C2—C1	−179.3 (4)	N4—C6—C7—C8	173.6 (4)
N2—N1—C2—S1	1.5 (6)	N3—C6—C7—C8	−4.1 (7)
C3—S1—C2—N1	−1.9 (4)	N4—C6—C7—C12	−5.3 (7)
C3—S1—C2—C1	178.9 (4)	N3—C6—C7—C12	176.9 (4)
N1—N2—C3—S1	−1.5 (5)	C12—C7—C8—C9	−1.1 (7)
N1—N2—C3—S2	178.4 (3)	C6—C7—C8—C9	179.9 (4)
C2—S1—C3—N2	1.9 (4)	C7—C8—C9—C10	0.5 (7)
C2—S1—C3—S2	−178.0 (3)	C8—C9—C10—C11	1.3 (7)
C4—S2—C3—N2	−4.6 (5)	C8—C9—C10—N5	−178.8 (4)
C4—S2—C3—S1	175.3 (3)	O2—N5—C10—C11	161.8 (4)
C3—S2—C4—C5	68.1 (5)	O3—N5—C10—C11	−17.3 (6)
C6—N3—C5—O1	−0.8 (5)	O2—N5—C10—C9	−18.1 (7)
C6—N3—C5—C4	−175.6 (5)	O3—N5—C10—C9	162.8 (4)
N4—O1—C5—N3	0.7 (5)	C9—C10—C11—C12	−2.4 (7)
N4—O1—C5—C4	176.0 (4)	N5—C10—C11—C12	177.7 (4)
S2—C4—C5—N3	−160.2 (4)	C10—C11—C12—C7	1.7 (7)
S2—C4—C5—O1	25.4 (6)	C8—C7—C12—C11	0.0 (7)
O1—N4—C6—N3	−0.3 (5)	C6—C7—C12—C11	179.0 (4)
O1—N4—C6—C7	−178.2 (4)		

Fig. 1