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

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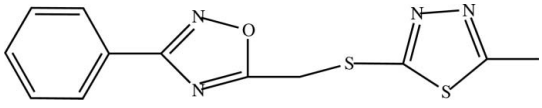
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.009 Å; R factor = 0.076; wR factor = 0.204; data-to-parameter ratio = 15.2.

The title compound, $C_{12}H_{10}N_4OS_2$, was synthesized *via* condensation of 3-phenyl-5-chloromethyl-1,2,4-oxadiazole with 5-mercapto-2-methyl-1,3,4-thiadiazole. The benzene and oxadiazole rings are roughly coplanar, making a twist angle of only 4.6 (3)°, whereas the thiadiazole ring makes 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

$C_{12}H_{10}N_4OS_2$
M_r = 290.36
Monoclinic, $P2_1/c$
a = 9.926 (2) Å
b = 5.9340 (12) Å
c = 23.049 (5) Å
β = 100.39 (3)°
V = 1335.3 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.40 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} = 0.858, T_{max} = 0.962
2764 measured reflections
2608 independent reflections
1492 reflections with $I > 2\sigma(I)$
R_{int} = 0.065
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.204
S = 1.00
2608 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 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: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2233).

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supplementary materials

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

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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 molecule is built up from a phenyl substituted oxadiazole linked to a methyl-thiadiazole through a methylenethio linker. The benzene and oxadiazole ring are roughly coplanar making a twist angle of only 4.6 (3)° whereas the thiadiazole ring make a dihedral angle of 87.9 (3)° with the oxadiazole ring (Fig. 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-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.): 8.05–8.09(m, 2H), 7.48–7.51 (m, 3H), 4.81–4.82 (s, 2H), 2.76–2.77 (s,2*H*).

Refinement

All H atoms were placed geometrically and treated as riding on their parent atoms with C—H= 0.93 Å(aromatic), 0.97Å (methylene) and 0.96Å (methyl) with U_{iso}(H) = 1.2 or 1.5(methyl)U_{eq}(C).

Figures

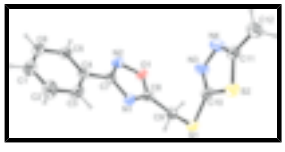

Fig. 1. A view of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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

Crystal data

$C_{12}H_{10}N_4OS_2$ F_{000} = 600

M_r = 290.36	D_x = 1.444 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 = 9.926 (2) Å	θ = 9–12º
b = 5.9340 (12) Å	μ = 0.40 mm^{-1}
c = 23.049 (5) Å	T = 293 (2) K
β = 100.39 (3)º	Block, colourless
V = 1335.3 (5) Å^3	0.40 × 0.10 × 0.10 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.065
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 1.8º
T = 293(2) K	h = −12→12
ω/2θ scans	k = 0→7
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→28
T_{min} = 0.858, T_{max} = 0.962	3 standard reflections
2764 measured reflections	every 200 reflections
2608 independent reflections	intensity decay: none
1492 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
$wR(F^2)$ = 0.204	$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 7P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max} < 0.001$
2608 reflections	$\Delta\rho_{max}$ = 0.47 e Å^{-3}
172 parameters	$\Delta\rho_{min}$ = −0.50 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	0.29941 (17)	0.5721 (2)	0.27537 (7)	0.0558 (5)
S2	0.05050 (19)	0.4212 (3)	0.18937 (8)	0.0710 (6)
O1	0.4942 (5)	0.0277 (7)	0.33950 (18)	0.0609 (12)
N1	0.3586 (5)	0.2231 (8)	0.3857 (2)	0.0477 (11)
N2	0.4626 (5)	−0.1135 (8)	0.3843 (2)	0.0562 (13)
N3	0.2582 (5)	0.1682 (9)	0.2216 (2)	0.0681 (16)
N4	0.1670 (5)	0.0436 (10)	0.1801 (3)	0.0761 (18)
C1	0.2304 (8)	−0.1991 (13)	0.5618 (3)	0.077 (2)
H1A	0.1983	−0.2411	0.5957	0.093*
C2	0.1964 (7)	0.0078 (15)	0.5358 (3)	0.079 (2)
H2B	0.1396	0.1044	0.5522	0.095*
C3	0.2448 (7)	0.0740 (11)	0.4862 (3)	0.0658 (18)
H3B	0.2206	0.2138	0.4693	0.079*
C4	0.3295 (6)	−0.0677 (10)	0.4615 (2)	0.0469 (13)
C5	0.3619 (6)	−0.2772 (10)	0.4860 (3)	0.0538 (15)
H5A	0.4164	−0.3752	0.4688	0.065*
C6	0.3123 (7)	−0.3418 (12)	0.5367 (3)	0.0649 (18)
H6A	0.3350	−0.4825	0.5534	0.078*
C7	0.3822 (5)	0.0092 (9)	0.4097 (2)	0.0388 (12)
C8	0.4290 (6)	0.2220 (10)	0.3438 (3)	0.0502 (14)
C9	0.4496 (6)	0.4124 (10)	0.3049 (2)	0.0510 (14)
H9A	0.4884	0.3538	0.2722	0.061*
H9B	0.5165	0.5140	0.3269	0.061*
C10	0.2114 (6)	0.3699 (9)	0.2295 (3)	0.0484 (14)
C11	0.0558 (6)	0.1539 (11)	0.1618 (3)	0.0591 (16)
C12	−0.0546 (7)	0.0550 (13)	0.1150 (3)	0.081 (2)
H12A	−0.0309	−0.0971	0.1069	0.122*
H12B	−0.0628	0.1433	0.0797	0.122*
H12C	−0.1402	0.0561	0.1288	0.122*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0789 (11)	0.0321 (8)	0.0584 (9)	−0.0002 (8)	0.0180 (8)	−0.0005 (7)
S2	0.0760 (12)	0.0531 (11)	0.0816 (12)	0.0154 (9)	0.0081 (9)	−0.0025 (9)
O1	0.094 (3)	0.038 (2)	0.056 (2)	0.008 (2)	0.028 (2)	0.001 (2)
N1	0.058 (3)	0.034 (3)	0.054 (3)	0.002 (2)	0.018 (2)	0.001 (2)
N2	0.079 (4)	0.036 (3)	0.055 (3)	0.011 (3)	0.016 (3)	−0.001 (2)
N3	0.070 (4)	0.049 (3)	0.078 (4)	0.013 (3)	−0.006 (3)	−0.017 (3)

N4	0.067 (4)	0.057 (4)	0.096 (4)	0.006 (3)	−0.009 (3)	−0.031 (3)
C1	0.102 (6)	0.073 (5)	0.064 (4)	−0.005 (5)	0.034 (4)	0.019 (4)
C2	0.078 (5)	0.095 (6)	0.075 (5)	0.010 (4)	0.042 (4)	0.012 (4)
C3	0.088 (5)	0.042 (4)	0.074 (4)	0.013 (3)	0.033 (4)	0.012 (3)
C4	0.056 (3)	0.035 (3)	0.049 (3)	−0.006 (3)	0.009 (3)	−0.006 (3)
C5	0.067 (4)	0.033 (3)	0.058 (4)	0.003 (3)	0.003 (3)	0.000 (3)
C6	0.084 (5)	0.052 (4)	0.059 (4)	−0.015 (4)	0.012 (3)	0.010 (3)
C7	0.039 (3)	0.034 (3)	0.040 (3)	0.002 (2)	−0.001 (2)	−0.005 (2)
C8	0.069 (4)	0.034 (3)	0.049 (3)	−0.002 (3)	0.015 (3)	−0.005 (3)
C9	0.057 (3)	0.050 (4)	0.046 (3)	0.000 (3)	0.007 (3)	−0.008 (3)
C10	0.048 (3)	0.037 (3)	0.060 (3)	0.003 (3)	0.008 (3)	0.007 (3)
C11	0.064 (4)	0.047 (4)	0.064 (4)	0.007 (3)	0.007 (3)	0.005 (3)
C12	0.080 (5)	0.074 (5)	0.084 (5)	−0.005 (4)	−0.001 (4)	−0.012 (4)

Geometric parameters (Å, °)

S1—C10	1.729 (6)	C2—H2B	0.9300
S1—C9	1.794 (6)	C3—C4	1.382 (8)
S2—C11	1.714 (7)	C3—H3B	0.9300
S2—C10	1.722 (6)	C4—C5	1.378 (8)
O1—C8	1.335 (7)	C4—C7	1.461 (7)
O1—N2	1.408 (6)	C5—C6	1.401 (8)
N1—C8	1.291 (7)	C5—H5A	0.9300
N1—C7	1.388 (7)	C6—H6A	0.9300
N2—C7	1.296 (7)	C8—C9	1.480 (8)
N3—C10	1.308 (7)	C9—H9A	0.9700
N3—N4	1.403 (6)	C9—H9B	0.9700
N4—C11	1.287 (7)	C11—C12	1.510 (8)
C1—C6	1.372 (10)	C12—H12A	0.9600
C1—C2	1.381 (10)	C12—H12B	0.9600
C1—H1A	0.9300	C12—H12C	0.9600
C2—C3	1.375 (9)		
C10—S1—C9	99.1 (3)	N2—C7—N1	114.0 (5)
C11—S2—C10	87.0 (3)	N2—C7—C4	122.4 (5)
C8—O1—N2	106.5 (4)	N1—C7—C4	123.5 (5)
C8—N1—C7	102.6 (5)	N1—C8—O1	113.3 (5)
C7—N2—O1	103.6 (4)	N1—C8—C9	127.1 (5)
C10—N3—N4	112.1 (5)	O1—C8—C9	119.5 (5)
C11—N4—N3	111.4 (5)	C8—C9—S1	115.9 (4)
C6—C1—C2	118.9 (6)	C8—C9—H9A	108.3
C6—C1—H1A	120.6	S1—C9—H9A	108.3
C2—C1—H1A	120.6	C8—C9—H9B	108.3
C3—C2—C1	121.3 (7)	S1—C9—H9B	108.3
C3—C2—H2B	119.4	H9A—C9—H9B	107.4
C1—C2—H2B	119.4	N3—C10—S2	113.9 (4)
C2—C3—C4	119.9 (6)	N3—C10—S1	124.6 (4)
C2—C3—H3B	120.1	S2—C10—S1	121.5 (3)
C4—C3—H3B	120.1	N4—C11—C12	120.4 (6)
C5—C4—C3	119.7 (6)	N4—C11—S2	115.5 (5)

C5—C4—C7	121.6 (5)	C12—C11—S2	123.9 (5)
C3—C4—C7	118.7 (5)	C11—C12—H12A	109.5
C4—C5—C6	119.8 (6)	C11—C12—H12B	109.5
C4—C5—H5A	120.1	H12A—C12—H12B	109.5
C6—C5—H5A	120.1	C11—C12—H12C	109.5
C1—C6—C5	120.5 (6)	H12A—C12—H12C	109.5
C1—C6—H6A	119.8	H12B—C12—H12C	109.5
C5—C6—H6A	119.8		
C8—O1—N2—C7	0.5 (6)	C7—N1—C8—O1	−0.1 (6)
C10—N3—N4—C11	−3.0 (8)	C7—N1—C8—C9	−175.9 (6)
C6—C1—C2—C3	−1.1 (12)	N2—O1—C8—N1	−0.2 (7)
C1—C2—C3—C4	−0.2 (12)	N2—O1—C8—C9	175.9 (5)
C2—C3—C4—C5	1.8 (10)	N1—C8—C9—S1	−45.1 (8)
C2—C3—C4—C7	−178.5 (6)	O1—C8—C9—S1	139.4 (5)
C3—C4—C5—C6	−2.1 (9)	C10—S1—C9—C8	−67.6 (5)
C7—C4—C5—C6	178.2 (5)	N4—N3—C10—S2	2.2 (7)
C2—C1—C6—C5	0.8 (11)	N4—N3—C10—S1	−177.6 (5)
C4—C5—C6—C1	0.8 (10)	C11—S2—C10—N3	−0.7 (5)
O1—N2—C7—N1	−0.6 (6)	C11—S2—C10—S1	179.1 (4)
O1—N2—C7—C4	−176.7 (5)	C9—S1—C10—N3	−2.5 (6)
C8—N1—C7—N2	0.4 (6)	C9—S1—C10—S2	177.7 (4)
C8—N1—C7—C4	176.5 (5)	N3—N4—C11—C12	177.5 (6)
C5—C4—C7—N2	−0.5 (8)	N3—N4—C11—S2	2.5 (8)
C3—C4—C7—N2	179.7 (6)	C10—S2—C11—N4	−1.1 (6)
C5—C4—C7—N1	−176.2 (5)	C10—S2—C11—C12	−175.9 (6)
C3—C4—C7—N1	4.0 (8)		

Fig. 1