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3-(3-Methoxyphenyl)-5-[(5-methyl-1,3,4-thiadiazol-2-yl)sulfanylmethyl]-1,2,4-oxadiazole

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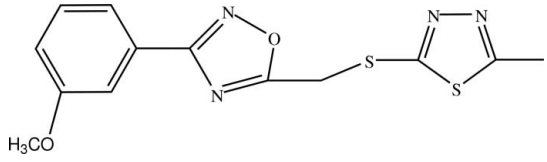
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.046; wR factor = 0.128; data-to-parameter ratio = 14.7.

The title compound, $C_{13}H_{12}N_4O_2S_2$, was synthesized *via* condensation of 5-chloromethyl-3-(3-methoxyphenyl)-1,2,4-oxadiazole with (5-methyl-1,3,4-thiadiazol-2-yl)methanethiol. The benzene and oxadiazole rings are coplanar due to the extended aromatic system. The angle between this plane and the thiadiazole ring is 77.8 (3)°.

Related literature

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



Experimental

Crystal data

$C_{13}H_{12}N_4O_2S_2$
M_r = 320.39
Triclinic, $P\overline{1}$
a = 6.139 (1) Å
b = 9.890 (2) Å
c = 12.658 (3) Å
α = 72.45 (3)°
β = 85.91 (3)°
γ = 77.12 (3)°
V = 714.3 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 293 (2) K
0.30 × 0.10 × 0.10 mm

Data collection

Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.894, T_{max} = 0.963
3078 measured reflections
2802 independent reflections
2063 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.128
S = 1.06
2802 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 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: IM2042).

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supplementary materials

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3-(3-Methoxyphenyl)-5-[(5-methyl-1,3,4-thiadiazol-2-yl)sulfanylmethyl]-1,2,4-oxadiazole

P. Wang, H. Zeng, H. Li, S. Kang and H. 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 thioether 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 triclinic system, space group $P\bar{1}$. There are three rings in the molecule. The benzene and oxadiazole ring are of course coplanar due to the extended aromatic system. The angle between the before mentioned plane and the thiadiazole moiety is 102.2 (3)°. There is no classic hydrogen bond in the crystal structure. The molecular structure of (I) is shown in Fig. 1. Bond lengths and angles are given in Table 1.

Experimental

(5-Methyl-[1,3,4]thiadiazol-2-yl)methanethiol (20 mmol) was dissolved in ethanol (70 ml) and water (70 mmol). Sodium acetate (20 mmol) was added to this mixture. Then 5-chlormethyl-3-(3-(methoxy)phenyl)-[1,2,4]oxadiazole (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 obstained by crystallization from a mixture of ethyl acetate (8 ml) and light petrolum (bp. 333–363 K) (6 ml). Crystals of (I) suitable for X-ray diffraction were obstained by slow evaporation of an ethanolic solution. ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 7.21–7.22 (m, 1H), 7.03–7.04 (m, 1H), 6.95–6.97 (m, 1H), 6.75–6.76 (m, 1H), 4.18–4.19 (s, 2H), 3.72–3.73 (s, 3H), 2.32–2.33 (s, 3H).

Refinement

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

Figures

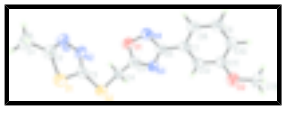

Fig. 1. Molecular structure of (I), showing displacement ellipsoids at the 30% probability level.

3-(3-Methoxyphenyl)-5-[(5-methyl-1,3,4-thiadiazol-2-yl)sulfanylmethyl]- 1,2,4-oxadiazole

Crystal data

$C_{13}H_{12}N_4O_2S_2$	$Z = 2$
$M_r = 320.39$	$F_{000} = 332$

Triclinic, $P\bar{1}$	D_x = 1.490 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 6.139 (1) Å	Cell parameters from 25 reflections
b = 9.890 (2) Å	θ = 9–13º
c = 12.658 (3) Å	μ = 0.38 mm^{-1}
α = 72.45 (3)º	T = 293 (2) K
β = 85.91 (3)º	Block, colourless
γ = 77.12 (3)º	0.30 × 0.10 × 0.10 mm
V = 714.3 (3) Å^3	

Data collection

Nonius CAD-4 diffractometer	R_{int} = 0.042
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 1.7º
T = 293(2) K	h = −7→7
$\omega/2\theta$ scans	k = −11→12
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→15
T_{min} = 0.894, T_{max} = 0.963	3 standard reflections
3078 measured reflections	every 200 reflections
2802 independent reflections	intensity decay: none
2063 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
$R[F^2 > 2\sigma(F^2)]$ = 0.046	H-atom parameters constrained
$wR(F^2)$ = 0.128	$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.4476P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max}$ = 0.002
2802 reflections	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
191 parameters	$\Delta\rho_{min}$ = −0.28 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	0.10119 (14)	0.96648 (9)	0.81705 (7)	0.0591 (3)
O1	−0.0772 (3)	0.5239 (2)	0.82199 (18)	0.0542 (5)
N1	−0.1954 (4)	0.8596 (3)	0.9446 (2)	0.0597 (7)
C1	−0.3048 (6)	1.1241 (4)	0.8789 (3)	0.0724 (10)
H1B	−0.4264	1.1101	0.9302	0.109*
H1C	−0.3623	1.1662	0.8049	0.109*
H1D	−0.2248	1.1878	0.8960	0.109*
S2	0.37028 (13)	0.66652 (9)	0.84080 (7)	0.0526 (2)
O2	0.3255 (4)	0.1410 (3)	0.4522 (2)	0.0658 (6)
N2	−0.0301 (4)	0.7422 (3)	0.9376 (2)	0.0572 (7)
C2	−0.1523 (5)	0.9826 (3)	0.8873 (2)	0.0512 (7)
N3	0.1928 (4)	0.3948 (2)	0.74697 (19)	0.0436 (6)
C3	0.1342 (5)	0.7819 (3)	0.8740 (2)	0.0455 (7)
N4	−0.1791 (4)	0.4826 (3)	0.7427 (2)	0.0542 (6)
C4	0.2916 (5)	0.4923 (3)	0.8934 (2)	0.0451 (7)
H4B	0.4258	0.4161	0.9054	0.054*
H4C	0.2177	0.4863	0.9646	0.054*
C5	0.1416 (4)	0.4666 (3)	0.8184 (2)	0.0431 (7)
C6	−0.0109 (4)	0.4076 (3)	0.7011 (2)	0.0395 (6)
C7	−0.0365 (4)	0.3450 (3)	0.6138 (2)	0.0413 (6)
C8	−0.2466 (5)	0.3615 (3)	0.5687 (3)	0.0530 (8)
H8A	−0.3742	0.4107	0.5963	0.064*
C9	−0.2626 (5)	0.3047 (4)	0.4837 (3)	0.0578 (8)
H9A	−0.4018	0.3172	0.4533	0.069*
C10	−0.0764 (5)	0.2296 (3)	0.4427 (3)	0.0508 (7)
H10A	−0.0903	0.1914	0.3854	0.061*
C11	0.1300 (5)	0.2115 (3)	0.4868 (2)	0.0463 (7)
C12	0.1486 (5)	0.2702 (3)	0.5724 (2)	0.0440 (7)
H12A	0.2884	0.2584	0.6019	0.053*
C13	0.3205 (6)	0.0859 (4)	0.3617 (3)	0.0632 (9)
H13A	0.4692	0.0399	0.3462	0.095*
H13B	0.2624	0.1640	0.2977	0.095*
H13C	0.2266	0.0163	0.3798	0.095*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0622 (5)	0.0439 (4)	0.0664 (5)	−0.0132 (4)	0.0141 (4)	−0.0110 (4)
O1	0.0392 (11)	0.0621 (13)	0.0624 (13)	0.0001 (10)	0.0046 (10)	−0.0288 (11)

N1	0.0627 (17)	0.0475 (15)	0.0632 (17)	−0.0066 (13)	0.0197 (14)	−0.0159 (13)
C1	0.079 (2)	0.052 (2)	0.073 (2)	0.0034 (18)	0.0129 (19)	−0.0144 (18)
S2	0.0426 (4)	0.0518 (5)	0.0632 (5)	−0.0071 (3)	0.0029 (3)	−0.0195 (4)
O2	0.0509 (13)	0.0825 (16)	0.0708 (15)	0.0001 (12)	−0.0045 (11)	−0.0421 (13)
N2	0.0592 (16)	0.0441 (14)	0.0604 (16)	−0.0060 (12)	0.0170 (13)	−0.0114 (12)
C2	0.0551 (18)	0.0469 (17)	0.0480 (17)	−0.0052 (14)	0.0051 (14)	−0.0137 (14)
N3	0.0405 (13)	0.0424 (13)	0.0441 (13)	−0.0010 (10)	−0.0007 (10)	−0.0125 (11)
C3	0.0503 (17)	0.0431 (15)	0.0437 (16)	−0.0086 (13)	−0.0007 (13)	−0.0143 (13)
N4	0.0429 (14)	0.0585 (16)	0.0656 (17)	−0.0059 (12)	−0.0008 (12)	−0.0281 (13)
C4	0.0441 (16)	0.0431 (15)	0.0453 (16)	0.0005 (12)	−0.0021 (13)	−0.0153 (13)
C5	0.0396 (15)	0.0364 (14)	0.0450 (16)	−0.0030 (12)	0.0012 (12)	−0.0039 (12)
C6	0.0341 (14)	0.0359 (14)	0.0427 (15)	−0.0057 (11)	0.0032 (11)	−0.0049 (12)
C7	0.0392 (15)	0.0378 (14)	0.0404 (15)	−0.0071 (11)	0.0021 (12)	−0.0032 (12)
C8	0.0361 (15)	0.0587 (19)	0.062 (2)	−0.0088 (14)	0.0015 (14)	−0.0154 (16)
C9	0.0414 (17)	0.072 (2)	0.062 (2)	−0.0154 (15)	−0.0060 (15)	−0.0185 (17)
C10	0.0559 (19)	0.0518 (17)	0.0480 (17)	−0.0179 (15)	−0.0039 (14)	−0.0139 (14)
C11	0.0437 (16)	0.0451 (16)	0.0478 (16)	−0.0082 (13)	−0.0006 (13)	−0.0110 (13)
C12	0.0397 (15)	0.0446 (15)	0.0467 (16)	−0.0101 (12)	−0.0039 (12)	−0.0103 (13)
C13	0.072 (2)	0.067 (2)	0.0543 (19)	−0.0092 (18)	0.0069 (17)	−0.0286 (17)

Geometric parameters (Å, °)

S1—C3	1.719 (3)	C4—C5	1.480 (4)
S1—C2	1.737 (3)	C4—H4B	0.9700
O1—C5	1.340 (3)	C4—H4C	0.9700
O1—N4	1.420 (3)	C6—C7	1.453 (4)
N1—C2	1.288 (4)	C7—C12	1.373 (4)
N1—N2	1.382 (3)	C7—C8	1.403 (4)
C1—C2	1.479 (4)	C8—C9	1.374 (4)
C1—H1B	0.9600	C8—H8A	0.9300
C1—H1C	0.9600	C9—C10	1.377 (4)
C1—H1D	0.9600	C9—H9A	0.9300
S2—C3	1.745 (3)	C10—C11	1.374 (4)
S2—C4	1.811 (3)	C10—H10A	0.9300
O2—C11	1.362 (3)	C11—C12	1.397 (4)
O2—C13	1.413 (4)	C12—H12A	0.9300
N2—C3	1.299 (4)	C13—H13A	0.9600
N3—C5	1.291 (3)	C13—H13B	0.9600
N3—C6	1.381 (3)	C13—H13C	0.9600
N4—C6	1.308 (3)		
C3—S1—C2	87.03 (15)	O1—C5—C4	118.0 (3)
C5—O1—N4	106.0 (2)	N4—C6—N3	113.8 (2)
C2—N1—N2	113.3 (3)	N4—C6—C7	122.9 (2)
C2—C1—H1B	109.5	N3—C6—C7	123.3 (2)
C2—C1—H1C	109.5	C12—C7—C8	118.9 (3)
H1B—C1—H1C	109.5	C12—C7—C6	119.6 (3)
C2—C1—H1D	109.5	C8—C7—C6	121.4 (3)
H1B—C1—H1D	109.5	C9—C8—C7	119.6 (3)
H1C—C1—H1D	109.5	C9—C8—H8A	120.2

C3—S2—C4	101.41 (14)	C7—C8—H8A	120.2
C11—O2—C13	118.5 (2)	C8—C9—C10	121.3 (3)
C3—N2—N1	112.0 (2)	C8—C9—H9A	119.4
N1—C2—C1	123.9 (3)	C10—C9—H9A	119.4
N1—C2—S1	113.4 (2)	C11—C10—C9	119.7 (3)
C1—C2—S1	122.7 (2)	C11—C10—H10A	120.1
C5—N3—C6	103.2 (2)	C9—C10—H10A	120.1
N2—C3—S1	114.2 (2)	O2—C11—C10	124.9 (3)
N2—C3—S2	126.1 (2)	O2—C11—C12	115.6 (3)
S1—C3—S2	119.65 (17)	C10—C11—C12	119.5 (3)
C6—N4—O1	103.5 (2)	C7—C12—C11	121.0 (3)
C5—C4—S2	113.3 (2)	C7—C12—H12A	119.5
C5—C4—H4B	108.9	C11—C12—H12A	119.5
S2—C4—H4B	108.9	O2—C13—H13A	109.5
C5—C4—H4C	108.9	O2—C13—H13B	109.5
S2—C4—H4C	108.9	H13A—C13—H13B	109.5
H4B—C4—H4C	107.7	O2—C13—H13C	109.5
N3—C5—O1	113.5 (3)	H13A—C13—H13C	109.5
N3—C5—C4	128.4 (2)	H13B—C13—H13C	109.5
C2—N1—N2—C3	−0.1 (4)	O1—N4—C6—C7	−178.8 (2)
N2—N1—C2—C1	178.5 (3)	C5—N3—C6—N4	−0.4 (3)
N2—N1—C2—S1	0.1 (4)	C5—N3—C6—C7	179.0 (2)
C3—S1—C2—N1	−0.1 (3)	N4—C6—C7—C12	178.6 (3)
C3—S1—C2—C1	−178.5 (3)	N3—C6—C7—C12	−0.8 (4)
N1—N2—C3—S1	0.0 (3)	N4—C6—C7—C8	−0.1 (4)
N1—N2—C3—S2	−178.1 (2)	N3—C6—C7—C8	−179.5 (3)
C2—S1—C3—N2	0.1 (2)	C12—C7—C8—C9	−0.9 (4)
C2—S1—C3—S2	178.2 (2)	C6—C7—C8—C9	177.8 (3)
C4—S2—C3—N2	10.8 (3)	C7—C8—C9—C10	1.0 (5)
C4—S2—C3—S1	−167.13 (17)	C8—C9—C10—C11	−0.4 (5)
C5—O1—N4—C6	−0.5 (3)	C13—O2—C11—C10	2.3 (4)
C3—S2—C4—C5	79.9 (2)	C13—O2—C11—C12	−176.6 (3)
C6—N3—C5—O1	0.0 (3)	C9—C10—C11—O2	−179.2 (3)
C6—N3—C5—C4	−179.7 (3)	C9—C10—C11—C12	−0.3 (4)
N4—O1—C5—N3	0.3 (3)	C8—C7—C12—C11	0.2 (4)
N4—O1—C5—C4	−179.9 (2)	C6—C7—C12—C11	−178.5 (2)
S2—C4—C5—N3	97.6 (3)	O2—C11—C12—C7	179.4 (2)
S2—C4—C5—O1	−82.1 (3)	C10—C11—C12—C7	0.4 (4)
O1—N4—C6—N3	0.6 (3)		

Fig. 1