organic compounds

2-{[3-(4-Chlorophenyl)-1,2,4-oxadiazol-5-yl]methylsulfanyl}-5-methyl-1,3,4-thiadiazole

Pin-liang Wang, Hai-ling Li, Si-shun Kang and Hai-bo Wang*

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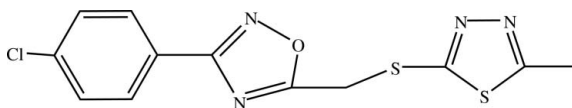Correspondence e-mail: wanghaibo@njut.edu.cn

The title compound, $C_{12}H_9ClN_4OS_2$, was synthesized *via* condensation of 5-chloromethyl-3-(4-chlorophenyl)-1,2,4-oxadiazole with 5-mercapto-2-methyl-1,3,4-thiadiazole. The benzene and oxadiazole rings are coplanar due to the extended aromatic system. The angle between this plane and the thiadiazole ring is 82.2 (3)°.

Related literature

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

Experimental

Crystal data

$C_{12}H_9ClN_4OS_2$	$V = 1397.0$ (5) Å^3
$M_r = 324.80$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 5.857$ (1) Å	$\mu = 0.57$ mm^{-1}
$b = 27.104$ (5) Å	$T = 293$ (2) K
$c = 9.026$ (2) Å	$0.40 \times 0.20 \times 0.10$ mm
$\beta = 102.85$ (3)°	

Data collection

Enraf–Nonius CAD-4 diffractometer	2733 independent reflections
Absorption correction: ψ scan (North *et al.*, 1968)	1783 reflections with $I > 2\sigma(I)$
$T_{min} = 0.804$, $T_{max} = 0.945$	$R_{int} = 0.043$
2999 measured reflections	3 standard reflections every 200 reflections intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.057$	182 parameters
$wR(F^2) = 0.159$	H-atom parameters constrained
$S = 1.05$	$\Delta\rho_{max} = 0.31$ e Å^{-3}
2733 reflections	$\Delta\rho_{min} = -0.36$ 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*.

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supplementary materials

2-{[3-(4-Chlorophenyl)-1,2,4-oxadiazol-5-yl]methylsulfanyl}-5-methyl-1,3,4-thiadiazole

P. Wang, 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 sulfurether compounds exhibited considerably strong inhibiting activity to Staphylococcus aureus (Talar & Dejai, 1996). We report here the structure of its close analogue with a thiadiazole sulfanylether group present in the molecule, (I). This compound crystallizes in the monoclinic system, space group $P2_1/c$. 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 measures to 82.2 (3)°. 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 (20 mmol) was dissolved in ethanol (70 ml) and water (70 mmol). Sodium acetate (20 mmol) was added to this mixture. Then 3-[4-(chloro)phenyl]-5-chloromethyl-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 obtained by recrystallization from a mixture of ethyl acetate (8 ml) and light petrolum (bp. 333–363 K) (4 ml). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanolic solution. ^{1}H NMR (CDCl$_3$, δ, p.p.m.): 7.45–7.47(m, 2H), 7.35–7.36 (m, 2H), 4.17–4.18 (s, 2H), 2.35–2.36 (s,3*H*).

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.
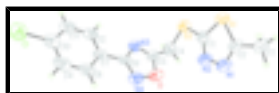
Figures

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

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Crystal data

C$_{12}$H$_9$ClN$_4$OS$_2$	$F_{000} = 664$
$M_r = 324.80$	$D_x = 1.544$ Mg m^{-3}

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 5.857 (1)$ Å

$b = 27.104 (5)$ Å

$c = 9.026 (2)$ Å

$\beta = 102.85 (3)°$

$V = 1397.0 (5)$ Å^3

$Z = 4$

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

Cell parameters from 25 reflections

$\theta = 9$–13º

$\mu = 0.57$ mm^{-1}

$T = 293 (2)$ K

Block, colourless

$0.40 \times 0.20 \times 0.10$ 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)

$T_{min} = 0.804$, $T_{max} = 0.945$

2999 measured reflections

2733 independent reflections

1783 reflections with $I > 2\sigma(I)$

$R_{int} = 0.043$

$\theta_{max} = 26.0°$

$\theta_{min} = 1.5°$

$h = -7 \rightarrow 7$

$k = 0 \rightarrow 33$

$l = 0 \rightarrow 11$

3 standard reflections

every 200 reflections

intensity decay: none

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.057$

$wR(F^2) = 0.159$

$S = 1.05$

2733 reflections

182 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

$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 1.5P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.31$ e Å^{-3}

$\Delta\rho_{min} = -0.36$ 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$^2 > 2$sigma(F^2) is used only for calculat-

ing *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*	$U_{iso}*/U_{eq}$
Cl	1.1111 (2)	−0.13088 (5)	1.04622 (16)	0.0869 (5)
O	0.5610 (6)	0.09306 (13)	0.4953 (4)	0.0756 (10)
S1	0.6416 (2)	0.25547 (5)	0.78126 (12)	0.0620 (4)
N1	0.7360 (6)	0.23958 (14)	0.5242 (4)	0.0604 (10)
C1	0.9769 (8)	0.30697 (17)	0.6594 (5)	0.0669 (12)
H1B	1.0282	0.3101	0.5660	0.100*
H1C	1.1078	0.2985	0.7400	0.100*
H1D	0.9114	0.3377	0.6826	0.100*
S2	0.2753 (2)	0.17581 (4)	0.71433 (13)	0.0600 (3)
N2	0.5640 (6)	0.20583 (15)	0.5361 (4)	0.0634 (10)
C2	0.7962 (7)	0.26766 (16)	0.6434 (4)	0.0546 (10)
C3	0.4946 (7)	0.21095 (16)	0.6626 (4)	0.0529 (10)
N3	0.4483 (6)	0.05758 (13)	0.6854 (4)	0.0558 (9)
N4	0.7201 (7)	0.05395 (15)	0.5459 (5)	0.0727 (11)
C4	0.2231 (8)	0.13062 (17)	0.5637 (5)	0.0622 (11)
H4B	0.0729	0.1149	0.5593	0.075*
H4C	0.2159	0.1471	0.4673	0.075*
C5	0.4090 (8)	0.09241 (16)	0.5865 (5)	0.0568 (11)
C6	0.6443 (7)	0.03463 (16)	0.6587 (4)	0.0552 (11)
C7	0.7599 (7)	−0.00683 (16)	0.7487 (4)	0.0528 (10)
C8	0.9555 (8)	−0.02945 (18)	0.7194 (5)	0.0658 (12)
H8A	1.0164	−0.0187	0.6383	0.079*
C9	1.0614 (8)	−0.06754 (19)	0.8081 (6)	0.0719 (13)
H9A	1.1929	−0.0827	0.7865	0.086*
C10	0.9735 (8)	−0.08369 (17)	0.9301 (5)	0.0625 (11)
C11	0.7767 (8)	−0.06139 (18)	0.9601 (5)	0.0683 (13)
H11A	0.7159	−0.0720	1.0414	0.082*
C12	0.6719 (8)	−0.02366 (18)	0.8695 (5)	0.0638 (12)
H12A	0.5385	−0.0090	0.8895	0.077*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0857 (9)	0.0915 (10)	0.0884 (10)	0.0198 (7)	0.0295 (7)	0.0077 (8)
O	0.091 (2)	0.081 (2)	0.067 (2)	−0.0018 (19)	0.0442 (18)	0.0078 (18)
S1	0.0772 (7)	0.0716 (8)	0.0434 (6)	−0.0138 (6)	0.0266 (5)	−0.0068 (5)
N1	0.066 (2)	0.077 (3)	0.0416 (19)	−0.0086 (19)	0.0185 (17)	0.0008 (18)
C1	0.081 (3)	0.070 (3)	0.054 (3)	−0.012 (2)	0.025 (2)	0.007 (2)
S2	0.0631 (7)	0.0634 (7)	0.0582 (7)	−0.0014 (5)	0.0238 (5)	0.0031 (5)
N2	0.075 (2)	0.077 (3)	0.0411 (19)	−0.010 (2)	0.0197 (17)	−0.0036 (18)
C2	0.060 (2)	0.066 (3)	0.039 (2)	−0.004 (2)	0.0143 (18)	0.008 (2)
C3	0.062 (2)	0.057 (3)	0.041 (2)	0.0049 (19)	0.0156 (18)	0.0045 (19)

N3	0.061 (2)	0.062 (2)	0.050 (2)	−0.0076 (17)	0.0250 (16)	−0.0032 (18)
N4	0.087 (3)	0.072 (3)	0.074 (3)	−0.007 (2)	0.050 (2)	−0.004 (2)
C4	0.057 (2)	0.067 (3)	0.062 (3)	−0.010 (2)	0.011 (2)	0.007 (2)
C5	0.066 (3)	0.062 (3)	0.047 (2)	−0.015 (2)	0.024 (2)	−0.006 (2)
C6	0.069 (3)	0.060 (3)	0.047 (2)	−0.011 (2)	0.036 (2)	−0.015 (2)
C7	0.060 (2)	0.057 (3)	0.049 (2)	−0.0140 (19)	0.0288 (19)	−0.0159 (19)
C8	0.070 (3)	0.073 (3)	0.069 (3)	−0.009 (2)	0.046 (2)	−0.011 (3)
C9	0.064 (3)	0.081 (3)	0.082 (3)	−0.001 (3)	0.043 (3)	−0.015 (3)
C10	0.062 (3)	0.070 (3)	0.060 (3)	−0.002 (2)	0.023 (2)	−0.008 (2)
C11	0.076 (3)	0.078 (3)	0.062 (3)	0.003 (3)	0.040 (2)	0.000 (3)
C12	0.073 (3)	0.073 (3)	0.059 (3)	0.008 (2)	0.044 (2)	−0.002 (2)

Geometric parameters (Å, °)

Cl—C10	1.735 (5)	N4—C6	1.307 (5)
O—C5	1.341 (5)	C4—C5	1.483 (6)
O—N4	1.418 (5)	C4—H4B	0.9700
S1—C3	1.713 (4)	C4—H4C	0.9700
S1—C2	1.726 (4)	C6—C7	1.461 (6)
N1—C2	1.301 (5)	C7—C8	1.376 (6)
N1—N2	1.382 (5)	C7—C12	1.383 (5)
C1—C2	1.486 (6)	C8—C9	1.368 (7)
C1—H1B	0.9600	C8—H8A	0.9300
C1—H1C	0.9600	C9—C10	1.386 (6)
C1—H1D	0.9600	C9—H9A	0.9300
S2—C3	1.744 (4)	C10—C11	1.380 (6)
S2—C4	1.805 (5)	C11—C12	1.367 (6)
N2—C3	1.302 (5)	C11—H11A	0.9300
N3—C5	1.284 (5)	C12—H12A	0.9300
N3—C6	1.373 (5)		
C5—O—N4	105.7 (3)	N3—C5—O	113.3 (4)
C3—S1—C2	87.5 (2)	N3—C5—C4	128.5 (4)
C2—N1—N2	113.0 (3)	O—C5—C4	118.2 (4)
C2—C1—H1B	109.5	N4—C6—N3	113.4 (4)
C2—C1—H1C	109.5	N4—C6—C7	123.0 (4)
H1B—C1—H1C	109.5	N3—C6—C7	123.6 (3)
C2—C1—H1D	109.5	C8—C7—C12	118.5 (4)
H1B—C1—H1D	109.5	C8—C7—C6	122.6 (4)
H1C—C1—H1D	109.5	C12—C7—C6	118.9 (4)
C3—S2—C4	100.4 (2)	C9—C8—C7	120.8 (4)
C3—N2—N1	111.9 (4)	C9—C8—H8A	119.6
N1—C2—C1	123.8 (4)	C7—C8—H8A	119.6
N1—C2—S1	113.3 (3)	C8—C9—C10	120.2 (4)
C1—C2—S1	122.9 (3)	C8—C9—H9A	119.9
N2—C3—S1	114.3 (3)	C10—C9—H9A	119.9
N2—C3—S2	124.7 (3)	C11—C10—C9	119.4 (5)
S1—C3—S2	121.0 (2)	C11—C10—Cl	120.0 (4)
C5—N3—C6	103.8 (3)	C9—C10—Cl	120.5 (4)
C6—N4—O	103.8 (3)	C12—C11—C10	119.6 (4)

C5—C4—S2	111.9 (3)	C12—C11—H11A	120.2
C5—C4—H4B	109.2	C10—C11—H11A	120.2
S2—C4—H4B	109.2	C11—C12—C7	121.5 (4)
C5—C4—H4C	109.2	C11—C12—H12A	119.3
S2—C4—H4C	109.2	C7—C12—H12A	119.3
H4B—C4—H4C	107.9		
C2—N1—N2—C3	2.3 (5)	O—N4—C6—N3	0.4 (5)
N2—N1—C2—C1	−179.9 (4)	O—N4—C6—C7	−178.7 (4)
N2—N1—C2—S1	−0.9 (5)	C5—N3—C6—N4	−1.2 (5)
C3—S1—C2—N1	−0.5 (3)	C5—N3—C6—C7	177.9 (4)
C3—S1—C2—C1	178.6 (4)	N4—C6—C7—C8	−1.4 (7)
N1—N2—C3—S1	−2.7 (5)	N3—C6—C7—C8	179.6 (4)
N1—N2—C3—S2	178.4 (3)	N4—C6—C7—C12	177.7 (4)
C2—S1—C3—N2	1.8 (4)	N3—C6—C7—C12	−1.4 (6)
C2—S1—C3—S2	−179.2 (3)	C12—C7—C8—C9	−0.3 (7)
C4—S2—C3—N2	7.5 (4)	C6—C7—C8—C9	178.7 (4)
C4—S2—C3—S1	−171.4 (3)	C7—C8—C9—C10	−0.5 (7)
C5—O—N4—C6	0.5 (4)	C8—C9—C10—C11	0.8 (7)
C3—S2—C4—C5	76.7 (3)	C8—C9—C10—Cl	−178.3 (4)
C6—N3—C5—O	1.6 (5)	C9—C10—C11—C12	−0.2 (7)
C6—N3—C5—C4	−178.0 (4)	Cl—C10—C11—C12	178.9 (4)
N4—O—C5—N3	−1.3 (5)	C10—C11—C12—C7	−0.6 (7)
N4—O—C5—C4	178.2 (4)	C8—C7—C12—C11	0.9 (7)
S2—C4—C5—N3	71.5 (5)	C6—C7—C12—C11	−178.1 (4)
S2—C4—C5—O	−108.0 (4)		

Fig. 1