organic compounds

N-(5-Methylsulfanyl-1,3,4-thiadiazol-2-yl)acetamide

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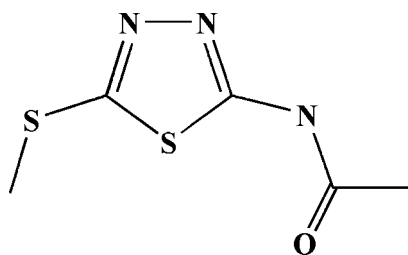Correspondence e-mail: hsxyzgy@mail.tjnu.edu.cn

In the title compound, $C_5H_7N_3OS_2$, inversion dimers linked by pairs of N—H···N hydrogen bonds occur, forming $R_2^2(8)$ ring motifs. These dimers are arranged into chains *via* intermolecular C—H···O hydrogen bonds between the methylsulfanyl groups and the O atoms of the carbonyl groups. The acetamido-1,3,4-thiadiazole unit is essentially planar [r.m.s. deviation 0.045 (8) Å].

Related literature

For the applications of 1,3,4-thiadiazole and its derivatives in antimicrobial drugs and in the construction of metal-organic frameworks, see: Gardinier *et al.* (2007); Mrozek *et al.* (2000); Xue *et al.* (2008). For the synthesis, see: Clerici & Pocar (2001).

Experimental

Crystal data

$C_5H_7N_3OS_2$	Triclinic, $P\bar{1}$
$M_r = 189.26$	$a = 5.0797\ (10)$ Å
$b = 7.9894\ (16)$ Å	$Z = 2$
$c = 10.081\ (2)$ Å	Mo $K\alpha$ radiation
$\alpha = 91.96\ (3)°$	$\mu = 0.62$ mm^{-1}
$\beta = 90.94\ (3)°$	$T = 293$ K
$\gamma = 105.27\ (3)°$	$0.30 \times 0.30 \times 0.10$ mm
$V = 394.32\ (14)$ Å^3	

Data collection

Rigaku R-AXIS RAPID-S diffractometer	3437 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)	1382 independent reflections
$T_{min} = 0.836$, $T_{max} = 0.941$	1259 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.016$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.078$	
$S = 1.07$	
1382 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
106 parameters	$\Delta\rho_{min} = -0.24$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···N2^{i}	0.77 (2)	2.12 (2)	2.881 (2)	173 (2)
C1—H1B···O1^{ii}	0.96	2.58	3.289 (3)	131 (2)

Symmetry codes: (i) $-x + 2, -y + 2, -z + 2$; (ii) $-x + 1, -y + 1, -z + 1$.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

References

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Gardinier, J. R., Silva, R. M., Gwengo, C. & Lindeman, S. V. (2007). *Chem. Commun.* pp. 1524–1526.
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supporting information

N-(5-Methylsulfanyl-1,3,4-thiadiazol-2-yl)acetamide

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S1. Comment

1,3,4-Thiodiazole is important for biological systems, and its derivatives have attracted widespread interest due to their further expanded application in antimicrobial drugs and in the construction of some interesting metal-organic frameworks (Gardinier *et al.*, 2007; Mrozek *et al.*, 2000; Xue *et al.*, 2008). Recently, we synthesized a new thiodiazole-ligand, namely 2-acetamido-5-methylmercapto-1,3,4-thiodiazole, (I). Herein we report the crystal structure of this ligand.

The molecular structure of (I) is shown in Fig. 1. The acetamido-1,3,4-thiadiazole moiety is essentially planar (r.m.s. deviation 0.045 (8) Å), forming a dihedral angle with the C1, S1 and C2 plane of atoms of 14.6 (9)°. In the crystal, inversion dimers linked by pairs of N—H⋯N hydrogen bonds occur, forming $R_2^2(8)$ ring motifs. These dimers are arranged into chains *via* intermolecular C—H⋯O hydrogen bonds between the methyl groups and the O atoms of the carbonyl groups (Fig. 2).

S2. Experimental

The title compound was prepared according to the literature (Clerici *et al.*, 2001). 5-Methylsulfanyl-1,3,4-thiadiazol-2-yl-amine (3.239 g, 0.022 mol) was suspended in acetic anhydride (2.28 ml, 0.024 mol), and acetic acid (9 ml) was added under stirring. The reaction mixture was further stirred at 313 K for 20 min. After cooling, water (10 ml) was added to the mixture, and then the precipitate was recrystallized in EtOH, which gave single crystals suitable for X-ray diffraction analysis (yield: 3.331 g, 80%).

S3. Refinement

All H atoms bound to C atoms were geometrically positioned and refined using a riding model, with C—H = 0.96 Å and $U_{iso}(H) = U_{eq}(C)$. H atom on amino N was located from difference Fourier map and its position was refined freely, with $U_{iso}(H) = U_{eq}(N)$. The refined N—H distance is 0.77 (2) Å.
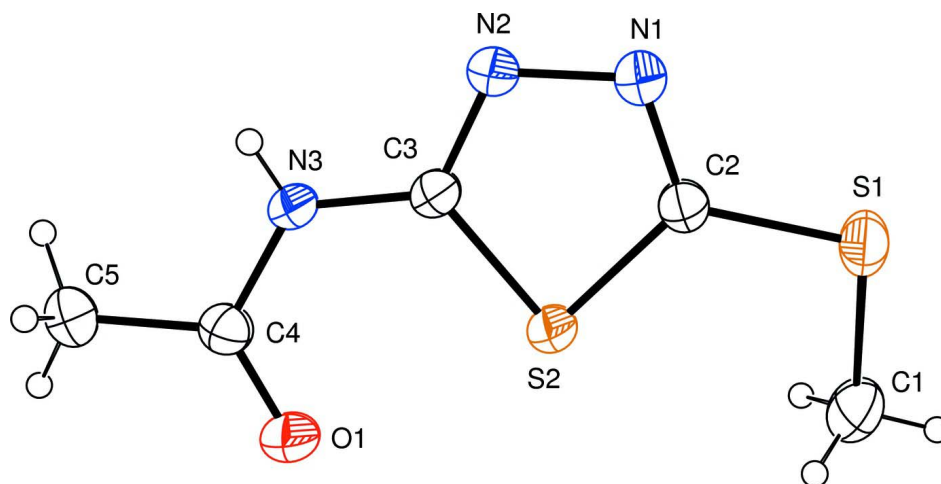
Figure 1

The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.
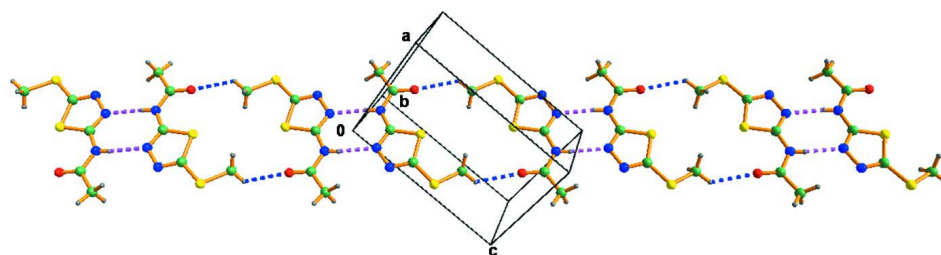
Figure 2

The chain structure linked by N—H···N (pink) and C—H···O (blue) hydrogen bonds in (I).

N-(5-Methylsulfanyl-1,3,4-thiadiazol-2-yl)acetamide

Crystal data

$C_5H_7N_3OS_2$	$Z = 2$
$M_r = 189.26$	$F(000) = 196$
Triclinic, $P\overline{1}$	$D_x = 1.594$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 5.0797$ (10) Å	Cell parameters from 3437 reflections
$b = 7.9894$ (16) Å	$\theta = 3.3$–$27.6°$
$c = 10.081$ (2) Å	$\mu = 0.62$ mm^{-1}
$\alpha = 91.96$ (3)°	$T = 293$ K
$\beta = 90.94$ (3)°	Block, yellow
$\gamma = 105.27$ (3)°	$0.30 \times 0.30 \times 0.10$ mm
$V = 394.32$ (14) Å^3	

Data collection

Rigaku R-AXIS RAPID-S diffractometer	Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
Radiation source: fine-focus sealed tube	$T_{min} = 0.836$, $T_{max} = 0.941$
Graphite monochromator	3437 measured reflections
ω scans	1382 independent reflections
	1259 reflections with $I > 2\sigma(I)$

$R_{int} = 0.016$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.3°$
$h = -6→6$

$k = -9→9$
$l = -11→11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.078$
$S = 1.07$
1382 reflections
106 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0418P)^2 + 0.1675P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	$U_{iso}*/U_{eq}$
C1	0.8654 (4)	0.6728 (3)	0.3920 (2)	0.0434 (5)
H1A	0.8055	0.5632	0.4333	0.065*
H1B	0.9004	0.6534	0.3002	0.065*
H1C	0.7261	0.7336	0.3984	0.065*
C2	1.0570 (4)	0.8285 (2)	0.63353 (18)	0.0267 (4)
C3	0.8380 (4)	0.8321 (2)	0.83774 (18)	0.0260 (4)
C4	0.4332 (4)	0.6973 (2)	0.95823 (19)	0.0282 (4)
C5	0.2992 (4)	0.7042 (3)	1.0889 (2)	0.0358 (5)
H5A	0.1062	0.6539	1.0780	0.054*
H5B	0.3327	0.8229	1.1205	0.054*
H5C	0.3730	0.6404	1.1521	0.054*
H3	0.732 (5)	0.878 (3)	1.008 (2)	0.039 (7)*
N1	1.2055 (3)	0.9415 (2)	0.71786 (16)	0.0324 (4)
N2	1.0764 (3)	0.9425 (2)	0.83800 (16)	0.0312 (4)
N3	0.6785 (3)	0.8170 (2)	0.94718 (17)	0.0300 (4)
O1	0.3383 (3)	0.59336 (19)	0.86769 (14)	0.0406 (4)
S1	1.17277 (10)	0.80015 (7)	0.47435 (5)	0.03824 (18)
S2	0.74395 (9)	0.71212 (6)	0.69103 (5)	0.02978 (17)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0426 (13)	0.0547 (14)	0.0290 (11)	0.0073 (11)	−0.0009 (9)	−0.0095 (10)
C2	0.0247 (9)	0.0298 (10)	0.0247 (9)	0.0057 (7)	0.0016 (7)	0.0004 (7)
C3	0.0256 (10)	0.0268 (9)	0.0237 (10)	0.0041 (8)	0.0004 (7)	−0.0039 (7)
C4	0.0253 (10)	0.0280 (10)	0.0298 (10)	0.0046 (8)	0.0011 (8)	−0.0005 (8)
C5	0.0328 (11)	0.0379 (11)	0.0336 (11)	0.0036 (9)	0.0085 (9)	0.0009 (9)
N1	0.0286 (9)	0.0358 (9)	0.0290 (9)	0.0021 (7)	0.0051 (7)	−0.0041 (7)
N2	0.0264 (9)	0.0336 (9)	0.0279 (9)	−0.0012 (7)	0.0038 (7)	−0.0062 (7)
N3	0.0266 (9)	0.0327 (9)	0.0246 (9)	−0.0017 (7)	0.0023 (7)	−0.0087 (7)
O1	0.0339 (8)	0.0409 (8)	0.0369 (8)	−0.0064 (6)	0.0029 (6)	−0.0117 (7)
S1	0.0323 (3)	0.0532 (4)	0.0271 (3)	0.0080 (2)	0.0066 (2)	−0.0043 (2)
S2	0.0256 (3)	0.0336 (3)	0.0247 (3)	−0.0007 (2)	0.00124 (19)	−0.00701 (19)

Geometric parameters (Å, º)

C1—S1	1.796 (2)	C3—S2	1.7244 (19)
C1—H1A	0.9600	C4—O1	1.216 (2)
C1—H1B	0.9600	C4—N3	1.365 (3)
C1—H1C	0.9600	C4—C5	1.499 (3)
C2—N1	1.294 (3)	C5—H5A	0.9600
C2—S2	1.737 (2)	C5—H5B	0.9600
C2—S1	1.7457 (19)	C5—H5C	0.9600
C3—N2	1.297 (2)	N1—N2	1.387 (2)
C3—N3	1.369 (3)	N3—H3	0.77 (2)
S1—C1—H1A	109.5	N3—C4—C5	114.83 (17)
S1—C1—H1B	109.5	C4—C5—H5A	109.5
H1A—C1—H1B	109.5	C4—C5—H5B	109.5
S1—C1—H1C	109.5	H5A—C5—H5B	109.5
H1A—C1—H1C	109.5	C4—C5—H5C	109.5
H1B—C1—H1C	109.5	H5A—C5—H5C	109.5
N1—C2—S2	115.25 (14)	H5B—C5—H5C	109.5
N1—C2—S1	120.67 (15)	C2—N1—N2	111.35 (16)
S2—C2—S1	124.08 (11)	C3—N2—N1	112.70 (15)
N2—C3—N3	120.95 (17)	C4—N3—C3	124.71 (17)
N2—C3—S2	114.78 (14)	C4—N3—H3	117.4 (18)
N3—C3—S2	124.27 (14)	C3—N3—H3	117.8 (18)
O1—C4—N3	121.00 (18)	C2—S1—C1	101.30 (10)
O1—C4—C5	124.16 (18)	C3—S2—C2	85.91 (9)
S2—C2—N1—N2	0.3 (2)	S2—C3—N3—C4	4.8 (3)
S1—C2—N1—N2	−179.74 (13)	N1—C2—S1—C1	−166.91 (17)
N3—C3—N2—N1	−178.87 (17)	S2—C2—S1—C1	13.04 (15)
S2—C3—N2—N1	0.6 (2)	N2—C3—S2—C2	−0.35 (15)
C2—N1—N2—C3	−0.6 (2)	N3—C3—S2—C2	179.09 (17)
O1—C4—N3—C3	−0.2 (3)	N1—C2—S2—C3	0.01 (16)

C5—C4—N3—C3	178.85 (18)	S1—C2—S2—C3		−179.95 (13)
N2—C3—N3—C4	−175.77 (18)			

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···N2ⁱ	0.77 (2)	2.12 (2)	2.881 (2)	173 (2)
C1—H1*B*···O1ⁱⁱ	0.96	2.58	3.289 (3)	131 (2)

Symmetry codes: (i) −*x*+2, −*y*+2, −*z*+2; (ii) −*x*+1, −*y*+1, −*z*+1.