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5-(4-Pyridyl)-1,3,4-thiadiazole-2(3*H*)-thione

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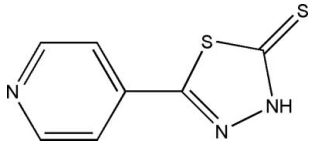
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.076; data-to-parameter ratio = 17.1.

The title compound $C_7H_5N_3S_2$, occurs as the thione tautomer in the solid state; the dihedral angle between the pyridine and thiadiazole ring planes is 2.08 (6)°. In the crystal, molecules are linked by N—H···N hydrogen bonds, generating $C(8)$ chains propagating in [010].

Related literature

For details of the synthesis, see: Song *et al.* (2005). For the biological activity of related compounds, see: Liu *et al.* (2007, 2009*a*,*b*,*c*).



Experimental

Crystal data

$C_7H_5N_3S_2$
M_r = 195.26
Monoclinic, $P2_1/c$
a = 7.837 (3) Å
b = 15.971 (5) Å
c = 6.694 (2) Å
β = 103.680 (4)°
V = 814.1 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.59 mm^{-1}
T = 113 K
0.20 × 0.20 × 0.08 mm

Data collection

Rigaku Saturn CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.891, T_{max} = 0.954
8141 measured reflections
1928 independent reflections
1642 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.076
S = 1.06
1928 reflections
113 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N3^{i}	0.90 (1)	1.85 (1)	2.7395 (19)	169 (2)

Symmetry code: (i) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5770).

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supporting information

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5-(4-Pyridyl)-1,3,4-thiadiazole-2(3*H*)-thione

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S1. Comment

Thidiazoles had excellent biological activities, such as fungicide, KARI (Liu *et al.*, 2007, 2009*a*,*b*,*c*). Meanwhile, some nicotine structure are also exhibited good biological activity. In continuated our work, a thidiazoles derivatives had been synthesized. The strucuture was confirmed by X-ray crstallography.

Single-crystal X-ray diffraction analysis reveals that the title compound crystallizes in the monoclinic space group P2(1)/c. As shown in Fig. 1, the pyridine ring and the thiadiazole ring are nearly in the same plane [dihedral angle = 2.1 °]. As shown in Fig. 2, the crystal structure is stabilized by weak N—H···N intermolecular interactions.

S2. Experimental

Potassium hydroxide (0.11 mol) was dissolved in minimum amount of ethanol, and 4-nicotinehydrazide (0.1 mol) was added to it. The reaction mixture was cooled to 0–5 °C followed by dropwise addition of carbon disulfide (0.11 mol). After addition, the reaction mixture was stirred for 30 min to afford solid potassium dithiocarbazate salt. It was filtered, washed with EtOH, dried, and used as such for further reaction. Potassium dithiocarbazate salt (0.1 mol) was added slowly in small lots to conc sulfuric acid (2.5 times of salt) at 5 °C with constant stirring. The reaction mixture was stirred for 30 min, and the resulting viscous liquid was poured over crushed ice slowly. The solid obtained was filtered and washed and dried to get the title compound. The compound was recrystallized in DMF to yield colorless prisms.

S3. Refinement

All the H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

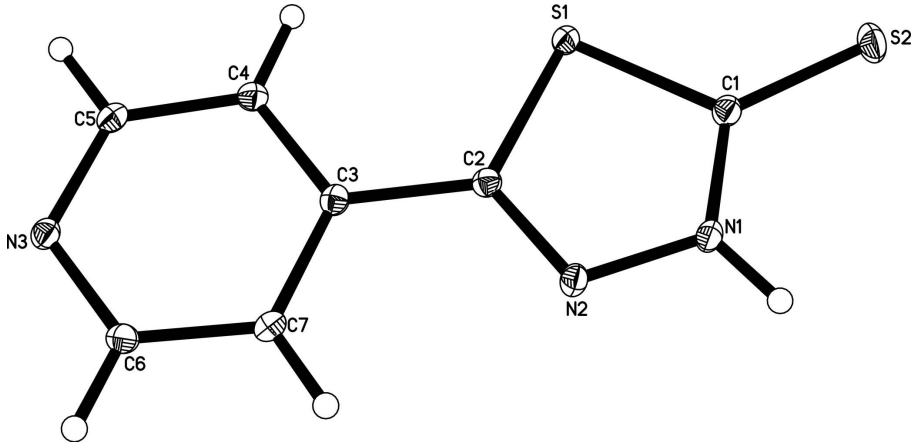

Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

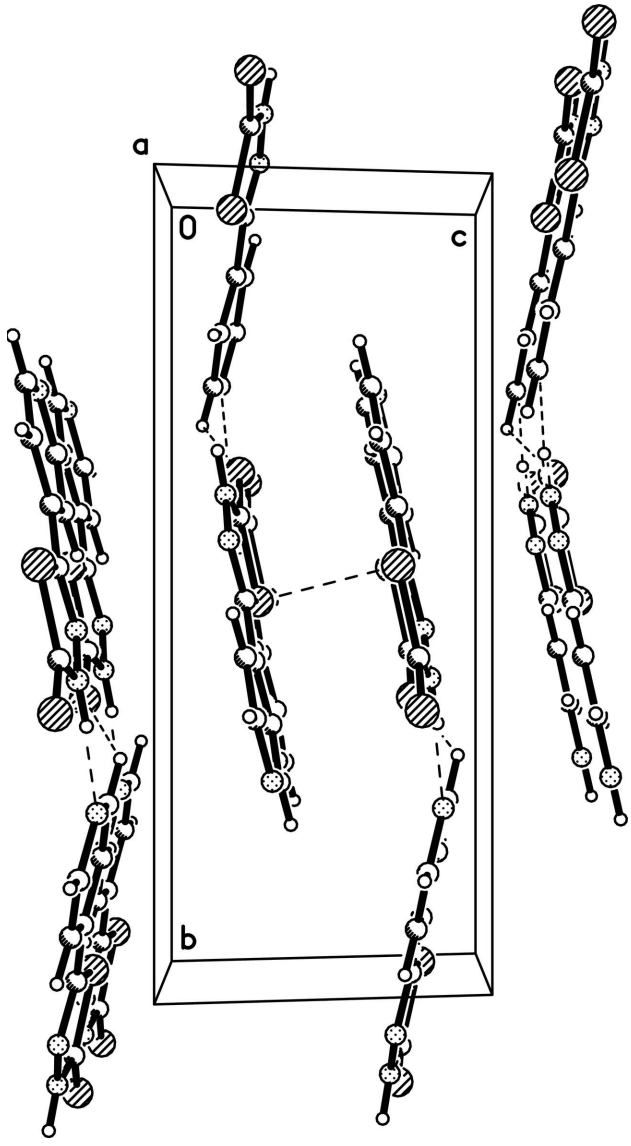

Figure 2

The crystal packing for (I).

5-(4-Pyridyl)-1,3,4-thiadiazole-2(3*H*)-thione

Crystal data

$C_7H_5N_3S_2$
$M_r = 195.26$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.837\ (3)$ Å
$b = 15.971\ (5)$ Å
$c = 6.694\ (2)$ Å
$\beta = 103.680\ (4)°$
$V = 814.1\ (5)$ Å^3
$Z = 4$
$F(000) = 400$
$D_x = 1.593$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2887 reflections
$\theta = 2.6–27.9°$
$\mu = 0.59$ mm^{-1}
$T = 113$ K
Prism, colorless
0.20 × 0.20 × 0.08 mm

Data collection

Rigaku Saturn CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 14.63 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.891, T_{max} = 0.954

8141 measured reflections
1928 independent reflections
1642 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 27.8°, θ_{min} = 2.6°
h = −10→10
k = −20→21
l = −8→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.076
S = 1.06
1928 reflections
113 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0461P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
S1	0.40446 (4)	0.02171 (2)	0.20702 (6)	0.01704 (11)
S2	0.60139 (5)	−0.14291 (2)	0.26909 (6)	0.02389 (13)
N1	0.27180 (16)	−0.11033 (7)	0.30185 (19)	0.0159 (3)
N2	0.13949 (15)	−0.05360 (7)	0.28751 (18)	0.0151 (2)
N3	−0.14570 (15)	0.23252 (7)	0.15350 (18)	0.0154 (3)
C1	0.42543 (17)	−0.08492 (8)	0.2646 (2)	0.0159 (3)
C2	0.19024 (17)	0.01952 (8)	0.2396 (2)	0.0138 (3)
C3	0.07477 (17)	0.09310 (8)	0.2109 (2)	0.0138 (3)
C4	0.13479 (18)	0.17060 (8)	0.1615 (2)	0.0160 (3)
H4	0.2515	0.1772	0.1463	0.019*
C5	0.02020 (18)	0.23800 (8)	0.1349 (2)	0.0159 (3)
H5	0.0617	0.2909	0.1018	0.019*
C6	−0.20252 (18)	0.15758 (8)	0.2012 (2)	0.0156 (3)
H6	−0.3202	0.1529	0.2145	0.019*
C7	−0.09795 (17)	0.08668 (9)	0.2320 (2)	0.0156 (3)

H7	−0.1426	0.0348	0.2667	0.019*
H1	0.244 (3)	−0.1638 (6)	0.325 (3)	0.047 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01417 (18)	0.01237 (18)	0.0254 (2)	−0.00003 (12)	0.00638 (15)	0.00162 (13)
S2	0.0190 (2)	0.0213 (2)	0.0318 (3)	0.00798 (14)	0.00682 (17)	0.00402 (15)
N1	0.0171 (6)	0.0109 (5)	0.0204 (7)	0.0014 (4)	0.0058 (5)	0.0016 (5)
N2	0.0171 (6)	0.0117 (5)	0.0170 (6)	0.0013 (5)	0.0048 (5)	0.0001 (4)
N3	0.0164 (6)	0.0132 (6)	0.0158 (6)	−0.0001 (5)	0.0021 (5)	−0.0016 (4)
C1	0.0169 (7)	0.0139 (6)	0.0162 (7)	0.0003 (5)	0.0025 (5)	−0.0005 (5)
C2	0.0142 (6)	0.0137 (6)	0.0137 (7)	−0.0005 (5)	0.0039 (5)	−0.0014 (5)
C3	0.0160 (7)	0.0136 (6)	0.0115 (7)	0.0002 (5)	0.0026 (5)	−0.0014 (5)
C4	0.0146 (7)	0.0158 (7)	0.0179 (7)	−0.0014 (5)	0.0046 (6)	−0.0002 (5)
C5	0.0183 (7)	0.0123 (6)	0.0167 (7)	−0.0022 (5)	0.0037 (6)	0.0000 (5)
C6	0.0142 (6)	0.0170 (7)	0.0158 (7)	−0.0015 (5)	0.0040 (6)	−0.0017 (5)
C7	0.0177 (7)	0.0126 (7)	0.0164 (7)	−0.0025 (5)	0.0042 (6)	−0.0006 (5)

Geometric parameters (Å, º)

S1—C2	1.7437 (15)	C2—C3	1.4679 (18)
S1—C1	1.7452 (15)	C3—C4	1.3916 (19)
S2—C1	1.6555 (14)	C3—C7	1.3974 (18)
N1—C1	1.3485 (18)	C4—C5	1.3862 (19)
N1—N2	1.3631 (16)	C4—H4	0.9500
N1—H1	0.902 (9)	C5—H5	0.9500
N2—C2	1.2980 (17)	C6—C7	1.3844 (19)
N3—C5	1.3378 (18)	C6—H6	0.9500
N3—C6	1.3417 (17)	C7—H7	0.9500
C2—S1—C1	89.77 (6)	C7—C3—C2	120.65 (12)
C1—N1—N2	118.98 (11)	C5—C4—C3	118.43 (13)
C1—N1—H1	125.1 (12)	C5—C4—H4	120.8
N2—N1—H1	115.7 (13)	C3—C4—H4	120.8
C2—N2—N1	110.06 (11)	N3—C5—C4	123.48 (12)
C5—N3—C6	117.71 (11)	N3—C5—H5	118.3
N1—C1—S2	127.20 (11)	C4—C5—H5	118.3
N1—C1—S1	107.03 (10)	N3—C6—C7	123.17 (13)
S2—C1—S1	125.77 (8)	N3—C6—H6	118.4
N2—C2—C3	122.50 (12)	C7—C6—H6	118.4
N2—C2—S1	114.16 (10)	C6—C7—C3	118.57 (12)
C3—C2—S1	123.33 (10)	C6—C7—H7	120.7
C4—C3—C7	118.64 (12)	C3—C7—H7	120.7
C4—C3—C2	120.71 (12)		
C1—N1—N2—C2	−0.77 (17)	N2—C2—C3—C7	−0.8 (2)
N2—N1—C1—S2	−178.87 (10)	S1—C2—C3—C7	177.65 (11)

N2—N1—C1—S1	0.50 (15)	C7—C3—C4—C5	−0.1 (2)
C2—S1—C1—N1	−0.08 (10)	C2—C3—C4—C5	179.66 (12)
C2—S1—C1—S2	179.29 (11)	C6—N3—C5—C4	0.3 (2)
N1—N2—C2—C3	179.24 (12)	C3—C4—C5—N3	−0.3 (2)
N1—N2—C2—S1	0.65 (15)	C5—N3—C6—C7	0.1 (2)
C1—S1—C2—N2	−0.34 (11)	N3—C6—C7—C3	−0.5 (2)
C1—S1—C2—C3	−178.92 (12)	C4—C3—C7—C6	0.5 (2)
N2—C2—C3—C4	179.40 (13)	C2—C3—C7—C6	−179.28 (12)
S1—C2—C3—C4	−2.14 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N3^{i}	0.90 (1)	1.85 (1)	2.7395 (19)	169 (2)

Symmetry code: (i) −*x*, *y*−1/2, −*z*+1/2.