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5-(4-Pyridyl)-1,3,4-thiadiazol-2-amine

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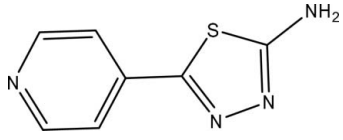
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.067; wR factor = 0.142; data-to-parameter ratio = 13.9.

The title compound, $C_7H_6N_4S$, was synthesized by reacting pyridine-4-carboxylic acid and thiosemicarbazide. The asymmetric unit contains two independent molecules, which present different conformations, the dihedral angles between the thiadiazole and pyridine rings being 18.2 (2) and 30.3 (2)°. In the crystal, intermolecular N—H···N hydrogen bonds involving the amine groups as donors link molecules into a two-dimensional framework.

Related literature

For the biological activity of 1,3,4-thiadiazoles, see: Nakagawa *et al.* (1996); Wang *et al.* (1999). For the structure of 2-amino-5-phenyl-1,3,4-thiadiazole, see: Öztürk *et al.* (2004).



Experimental

Crystal data

$C_7H_6N_4S$
M_r = 178.22
Monoclinic, $P2_1/c$
a = 14.794 (3) Å
b = 10.686 (2) Å
c = 10.477 (2) Å
β = 106.52 (3)°
V = 1587.9 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 293 K
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.933, T_{max} = 0.966
3203 measured reflections
3023 independent reflections
1516 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.142
S = 1.00
3023 reflections
217 parameters
43 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4A—H4A···N1B^{i}	0.86	2.08	2.940 (5)	177
N4A—H4B···N2A^{ii}	0.86	2.21	3.053 (5)	166
N4B—H8A···N1A^{iii}	0.86	2.10	2.945 (5)	168
N4B—H8B···N2B^{iv}	0.86	2.13	2.988 (5)	178

Symmetry codes: (i) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+1, y+\frac{1}{2}, -z-\frac{1}{2}$; (iv) $-x+1, y-\frac{1}{2}, -z-\frac{1}{2}$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1989); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo,1995); 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*.

The authors thank Professor Hua-qin Wang of the Analysis Centre, Nanjing University, for providing the Enraf–Nonius CAD-4 diffractometer for this research project.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2224).

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supplementary materials

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5-(4-Pyridyl)-1,3,4-thiadiazol-2-amine

Y. Wang, R. Wan, F. Han, P. Wang and B. Wang

Comment

1,3,4-Thiadiazole derivatives represent an interesting class of compounds possessing a broad spectrum of biological activity (Nakagawa *et al.*, 1996). These compounds are known to exhibit diverse biological effects, such as insecticidal and fungicidal activities (Wang *et al.*, 1999).

The asymmetric unit of the title compound contains two independent molecules (A and B, see Fig. 1), with bond lengths and angles in expected ranges. (Öztürk *et al.*, 2004). Dihedral angles between thiadiazole and pyridine rings are different in each molecule: 18.2 (2)° for molecule A and 30.3 (2)° in molecule B. In the crystal, molecules are linked through N—H···N hydrogen bonds, forming a two-dimensional supramolecular structure.

Experimental

4-Pyridinecarboxylic acid (2 mmol) and thiosemicarbazide (5 mmol) were mixed in a 25 ml flask, and kept in the oil bath at 363 K for 6 h. After cooling, the crude product precipitated and was filtered. Pure compound was obtained by crystallization from ethanol. Crystals suitable for X-ray diffraction were obtained by slow evaporation of an acetone solution.

Refinement

All H atoms were placed geometrically with C—H and N—H bond lengths fixed to 0.93 and 0.86 Å, respectively, and included in the refinement in the riding motion approximation, with U_{iso}(H) = 1.2 U_{eq}(carrier atom). In the B molecule, displacement parameters for atoms C3B/C4B/C5B/C6B/N4B/C7B were restrained to approximate an isotropic behaviour, and a rigid bond restraint was applied to fragments C3B/C4B/C5B/C6B and N4B C7B.

Figures

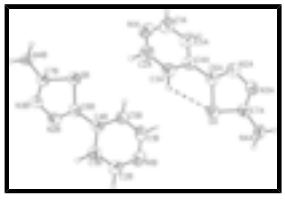

Fig. 1. A view of the molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. The dashed line indicates an intramolecular C—H···S hydrogen bond.

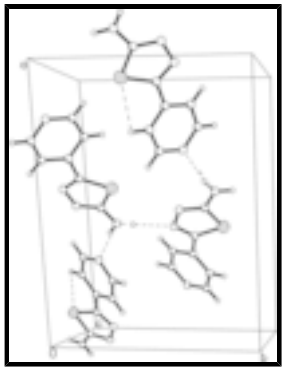

Fig. 2. Partial packing view showing the hydrogen bonds network. Dashed lines indicate intermolecular N—H···N hydrogen bonds.

5-(4-Pyridyl)-1,3,4-thiadiazol-2-amine

Crystal data

$C_7H_6N_4S$	F_{000} = 736
M_r = 178.22	D_x = 1.491 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 543 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 14.794 (3) Å	Cell parameters from 25 reflections
b = 10.686 (2) Å	θ = 9–12º
c = 10.477 (2) Å	μ = 0.35 mm^{-1}
β = 106.52 (3)º	T = 293 K
V = 1587.9 (5) Å^3	Block, colorless
Z = 8	0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.042
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 1.4º
T = 293 K	h = −18→17
ω/2θ scans	k = −13→0
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→12
T_{min} = 0.933, T_{max} = 0.966	3 standard reflections
3203 measured reflections	every 200 reflections
3023 independent reflections	intensity decay: 1%
1516 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

$R[F^2 > 2\sigma(F^2)] = 0.067$

$wR(F^2) = 0.142$

$S = 1.00$

3023 reflections

217 parameters

43 restraints

Primary atom site location: structure-invariant direct methods

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0435P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.24$ e Å^{-3}

$\Delta\rho_{min} = -0.31$ e Å^{-3}

Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
SA	0.08251 (8)	0.54450 (10)	0.19829 (12)	0.0483 (4)
N1A	0.3034 (3)	0.3300 (4)	−0.0749 (4)	0.0551 (11)
N2A	0.0328 (3)	0.3203 (3)	0.1338 (4)	0.0513 (10)
N3A	−0.0258 (2)	0.3605 (3)	0.2081 (4)	0.0509 (10)
N4A	−0.0553 (2)	0.5382 (3)	0.3223 (3)	0.0548 (11)
H4A	−0.0999	0.5013	0.3454	0.066*
H4B	−0.0408	0.6143	0.3462	0.066*
C1A	0.2262 (3)	0.2621 (5)	−0.0932 (4)	0.0604 (14)
H1B	0.2192	0.1944	−0.1510	0.073*
C2A	0.3105 (3)	0.4259 (5)	0.0056 (5)	0.0601 (14)
H2B	0.3635	0.4769	0.0204	0.072*
C3A	0.2430 (3)	0.4551 (4)	0.0697 (4)	0.0485 (12)
H3B	0.2515	0.5243	0.1256	0.058*
C4A	0.1643 (3)	0.3827 (4)	0.0511 (4)	0.0393 (11)
C5A	0.1561 (3)	0.2820 (4)	−0.0357 (4)	0.0549 (13)
H5A	0.1038	0.2296	−0.0538	0.066*
C6A	0.0928 (3)	0.4040 (4)	0.1217 (4)	0.0413 (11)
C7A	−0.0070 (3)	0.4754 (4)	0.2470 (4)	0.0396 (11)
SB	0.43176 (9)	0.68957 (10)	−0.17070 (12)	0.0505 (4)
N1B	0.2053 (3)	0.9156 (4)	0.0901 (4)	0.0573 (11)
N2B	0.4342 (3)	0.9258 (3)	−0.1864 (4)	0.0518 (11)
N3B	0.4926 (2)	0.8856 (3)	−0.2569 (4)	0.0489 (10)
N4B	0.5574 (2)	0.7052 (3)	−0.3157 (3)	0.0456 (10)
H8A	0.5907	0.7475	−0.3556	0.055*
H8B	0.5607	0.6249	−0.3136	0.055*
C1B	0.1980 (3)	0.8094 (5)	0.0207 (5)	0.0562 (13)
H8C	0.1499	0.7540	0.0226	0.067*
C2B	0.2762 (3)	0.9914 (4)	0.0858 (4)	0.0577 (13)
H9A	0.2837	1.0650	0.1352	0.069*
C3B	0.3375 (3)	0.9687 (4)	0.0150 (4)	0.0507 (12)
H10A	0.3841	1.0269	0.0141	0.061*
C4B	0.3308 (3)	0.8588 (4)	−0.0563 (4)	0.0362 (10)
C5B	0.2573 (3)	0.7782 (4)	−0.0528 (4)	0.0506 (12)
H12A	0.2489	0.7035	−0.1003	0.061*
C6B	0.3977 (3)	0.8349 (4)	−0.1355 (4)	0.0389 (10)

C7B	0.4998 (3)	0.7650 (4)	−0.2563 (4)	0.0380 (10)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
SA	0.0477 (7)	0.0469 (7)	0.0499 (8)	−0.0047 (6)	0.0134 (6)	−0.0041 (6)
N1A	0.054 (3)	0.071 (3)	0.041 (2)	0.013 (2)	0.015 (2)	−0.002 (2)
N2A	0.045 (2)	0.057 (2)	0.047 (2)	−0.001 (2)	0.005 (2)	−0.003 (2)
N3A	0.046 (2)	0.049 (2)	0.056 (3)	−0.0055 (19)	0.012 (2)	0.003 (2)
N4A	0.058 (3)	0.065 (3)	0.050 (3)	−0.012 (2)	0.028 (2)	0.000 (2)
C1A	0.054 (3)	0.080 (4)	0.043 (3)	−0.004 (3)	0.006 (3)	−0.024 (3)
C2A	0.043 (3)	0.067 (4)	0.071 (4)	0.004 (3)	0.018 (3)	0.011 (3)
C3A	0.046 (3)	0.052 (3)	0.046 (3)	−0.003 (2)	0.011 (2)	−0.004 (2)
C4A	0.029 (2)	0.050 (3)	0.031 (3)	0.003 (2)	−0.006 (2)	0.006 (2)
C5A	0.041 (3)	0.076 (4)	0.041 (3)	−0.001 (3)	0.001 (2)	−0.014 (3)
C6A	0.036 (3)	0.048 (3)	0.037 (3)	−0.004 (2)	0.004 (2)	0.007 (2)
C7A	0.035 (2)	0.046 (3)	0.032 (2)	0.009 (2)	0.002 (2)	0.012 (2)
SB	0.0587 (8)	0.0404 (6)	0.0564 (8)	0.0000 (6)	0.0229 (7)	0.0013 (6)
N1B	0.048 (3)	0.069 (3)	0.055 (3)	0.010 (2)	0.015 (2)	0.007 (2)
N2B	0.056 (3)	0.038 (2)	0.065 (3)	0.0007 (19)	0.022 (2)	0.003 (2)
N3B	0.049 (2)	0.045 (2)	0.055 (3)	0.0003 (19)	0.019 (2)	−0.004 (2)
N4B	0.063 (2)	0.036 (2)	0.047 (2)	0.0107 (18)	0.031 (2)	0.0063 (18)
C1B	0.039 (3)	0.069 (3)	0.060 (3)	−0.009 (3)	0.013 (3)	0.007 (3)
C2B	0.063 (3)	0.058 (3)	0.057 (3)	−0.006 (3)	0.025 (3)	−0.004 (3)
C3B	0.046 (3)	0.055 (3)	0.053 (3)	−0.008 (2)	0.017 (2)	−0.007 (2)
C4B	0.031 (2)	0.039 (2)	0.034 (2)	0.0008 (19)	0.002 (2)	−0.001 (2)
C5B	0.047 (3)	0.046 (3)	0.059 (3)	−0.002 (2)	0.016 (2)	0.001 (2)
C6B	0.035 (2)	0.042 (2)	0.034 (2)	−0.001 (2)	0.001 (2)	0.001 (2)
C7B	0.033 (2)	0.037 (2)	0.036 (2)	0.002 (2)	−0.003 (2)	0.005 (2)

Geometric parameters (Å, °)

SA—C7A	1.716 (4)	SB—C6B	1.705 (4)
SA—C6A	1.729 (4)	SB—C7B	1.726 (4)
N1A—C2A	1.312 (5)	N1B—C1B	1.335 (5)
N1A—C1A	1.320 (5)	N1B—C2B	1.336 (5)
N2A—C6A	1.292 (5)	N2B—C6B	1.298 (5)
N2A—N3A	1.387 (4)	N2B—N3B	1.356 (4)
N3A—C7A	1.298 (5)	N3B—C7B	1.293 (5)
N4A—C7A	1.380 (5)	N4B—C7B	1.349 (4)
N4A—H4A	0.8600	N4B—H8A	0.8600
N4A—H4B	0.8600	N4B—H8B	0.8600
C1A—C5A	1.356 (6)	C1B—C5B	1.363 (6)
C1A—H1B	0.9300	C1B—H8C	0.9300
C2A—C3A	1.389 (6)	C2B—C3B	1.347 (5)
C2A—H2B	0.9300	C2B—H9A	0.9300
C3A—C4A	1.365 (5)	C3B—C4B	1.380 (5)
C3A—H3B	0.9300	C3B—H10A	0.9300
C4A—C5A	1.392 (5)	C4B—C5B	1.396 (5)

C4A—C6A	1.470 (5)	C4B—C6B	1.483 (5)
C5A—H5A	0.9300	C5B—H12A	0.9300
C7A—SA—C6A	86.7 (2)	C6B—SB—C7B	86.5 (2)
C2A—N1A—C1A	115.6 (4)	C1B—N1B—C2B	116.1 (4)
C6A—N2A—N3A	113.4 (4)	C6B—N2B—N3B	113.0 (3)
C7A—N3A—N2A	110.9 (3)	C7B—N3B—N2B	112.2 (4)
C7A—N4A—H4A	120.0	C7B—N4B—H8A	120.0
C7A—N4A—H4B	120.0	C7B—N4B—H8B	120.0
H4A—N4A—H4B	120.0	H8A—N4B—H8B	120.0
N1A—C1A—C5A	126.0 (5)	N1B—C1B—C5B	123.4 (4)
N1A—C1A—H1B	117.0	N1B—C1B—H8C	118.3
C5A—C1A—H1B	117.0	C5B—C1B—H8C	118.3
N1A—C2A—C3A	123.2 (5)	N1B—C2B—C3B	124.5 (5)
N1A—C2A—H2B	118.4	N1B—C2B—H9A	117.7
C3A—C2A—H2B	118.4	C3B—C2B—H9A	117.7
C4A—C3A—C2A	120.4 (4)	C2B—C3B—C4B	119.7 (4)
C4A—C3A—H3B	119.8	C2B—C3B—H10A	120.2
C2A—C3A—H3B	119.8	C4B—C3B—H10A	120.2
C3A—C4A—C5A	116.3 (4)	C3B—C4B—C5B	116.6 (4)
C3A—C4A—C6A	123.2 (4)	C3B—C4B—C6B	119.5 (4)
C5A—C4A—C6A	120.5 (4)	C5B—C4B—C6B	123.9 (4)
C1A—C5A—C4A	118.5 (5)	C1B—C5B—C4B	119.7 (4)
C1A—C5A—H5A	120.8	C1B—C5B—H12A	120.2
C4A—C5A—H5A	120.8	C4B—C5B—H12A	120.2
N2A—C6A—C4A	123.7 (4)	N2B—C6B—C4B	121.5 (4)
N2A—C6A—SA	113.7 (3)	N2B—C6B—SB	114.2 (3)
C4A—C6A—SA	122.6 (3)	C4B—C6B—SB	124.3 (3)
N3A—C7A—N4A	122.7 (4)	N3B—C7B—N4B	122.1 (4)
N3A—C7A—SA	115.3 (3)	N3B—C7B—SB	114.1 (3)
N4A—C7A—SA	121.9 (3)	N4B—C7B—SB	123.8 (3)
C6A—N2A—N3A—C7A	0.9 (5)	C6B—N2B—N3B—C7B	−0.7 (6)
C2A—N1A—C1A—C5A	−1.0 (7)	C2B—N1B—C1B—C5B	−0.9 (7)
C1A—N1A—C2A—C3A	0.8 (7)	C1B—N1B—C2B—C3B	1.6 (7)
N1A—C2A—C3A—C4A	0.1 (7)	N1B—C2B—C3B—C4B	−2.1 (8)
C2A—C3A—C4A—C5A	−1.0 (6)	C2B—C3B—C4B—C5B	1.7 (6)
C2A—C3A—C4A—C6A	176.4 (4)	C2B—C3B—C4B—C6B	179.3 (4)
N1A—C1A—C5A—C4A	0.1 (8)	N1B—C1B—C5B—C4B	0.7 (7)
C3A—C4A—C5A—C1A	0.9 (6)	C3B—C4B—C5B—C1B	−1.1 (6)
C6A—C4A—C5A—C1A	−176.6 (4)	C6B—C4B—C5B—C1B	−178.6 (4)
N3A—N2A—C6A—C4A	178.1 (4)	N3B—N2B—C6B—C4B	−179.1 (4)
N3A—N2A—C6A—SA	−1.2 (5)	N3B—N2B—C6B—SB	−0.4 (5)
C3A—C4A—C6A—N2A	−160.7 (4)	C3B—C4B—C6B—N2B	−29.4 (6)
C5A—C4A—C6A—N2A	16.7 (6)	C5B—C4B—C6B—N2B	147.9 (4)
C3A—C4A—C6A—SA	18.5 (6)	C3B—C4B—C6B—SB	152.0 (3)
C5A—C4A—C6A—SA	−164.1 (3)	C5B—C4B—C6B—SB	−30.7 (6)
C7A—SA—C6A—N2A	0.9 (3)	C7B—SB—C6B—N2B	1.0 (3)
C7A—SA—C6A—C4A	−178.4 (4)	C7B—SB—C6B—C4B	179.7 (4)
N2A—N3A—C7A—N4A	179.9 (4)	N2B—N3B—C7B—N4B	−177.2 (4)

N2A—N3A—C7A—SA	−0.2 (5)	N2B—N3B—C7B—SB	1.5 (5)
C6A—SA—C7A—N3A	−0.4 (3)	C6B—SB—C7B—N3B	−1.4 (3)
C6A—SA—C7A—N4A	179.6 (4)	C6B—SB—C7B—N4B	177.3 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3A—H3B···SA	0.93	2.82	3.191 (5)	105
N4A—H4A···N1B^{i}	0.86	2.08	2.940 (5)	177
N4A—H4B···N2A^{ii}	0.86	2.21	3.053 (5)	166
N4B—H8A···N1A^{iii}	0.86	2.10	2.945 (5)	168
N4B—H8B···N2B^{iv}	0.86	2.13	2.988 (5)	178

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

Fig. 1

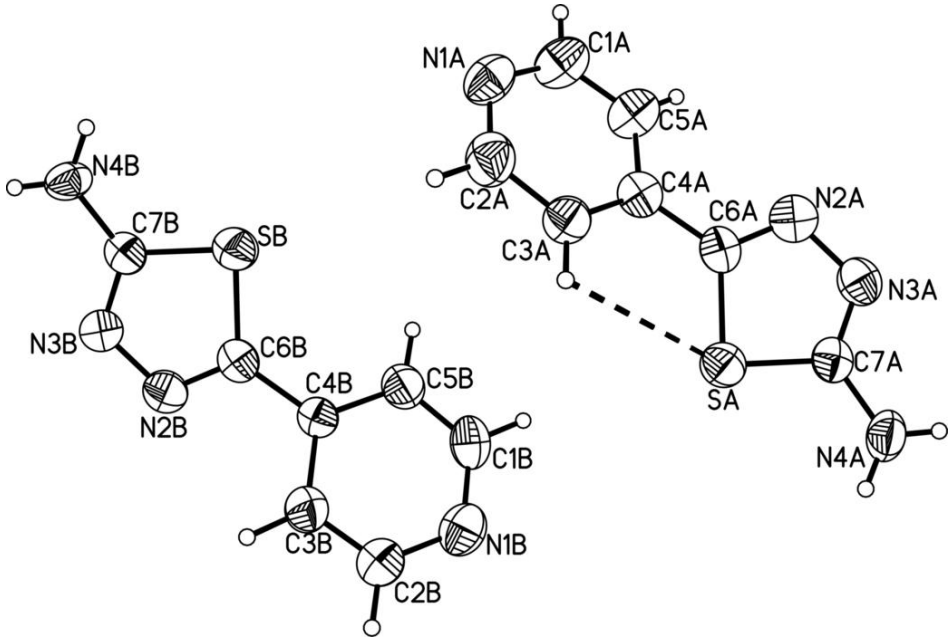
N1A
C1A
C5A
C2A
C4A
C6A
N2A
C3A
N3A
C7A
SA
N4A
N4B
C7B
SB
N3B
C6B
C4B
C5B
N2B
C1B
C3B
N1B
C2B

Fig. 2