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5-(2-Fluoro-4-nitrophenyl)-1,3,4-thiadiazole-2-amine

Yao Wang, Rong Wan,* Feng Han and Peng Wang

Department of Applied Chemistry, College of Science, Nanjing University of Technology, No.5 Xinmofan Road, Nanjing, Nanjing 210009, People's Republic of China
Correspondence e-mail: rwan@njut.edu.cn

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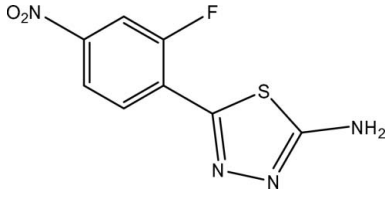
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.056; wR factor = 0.194; data-to-parameter ratio = 11.9.

The title compound, $C_8H_5FN_4O_2S$, was synthesized by the reaction of 2-fluoro-4-nitrobenzoic acid and thiosemicarbazide. The dihedral angle between the thiadiazole and benzene rings is 27.1 (2)°. In the crystal, intermolecular N—H···N and C—H···O hydrogen bonds link the molecules.

Related literature

For general background to the biological activity of 1,3,4-thiadiazole derivatives, see: Nakagawa *et al.* (1996); Wang *et al.* (1999). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_8H_5FN_4O_2S$
M_r = 240.22
Monoclinic, $P2_1/c$
a = 9.1780 (18) Å
b = 9.3720 (19) Å
c = 11.413 (2) Å
β = 102.55 (3)°
V = 958.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.34 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} = 0.935, T_{max} = 0.967
1832 measured reflections
1720 independent reflections
1301 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.194
S = 1.01
1720 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···N3^{i}	0.86	2.16	3.000 (5)	167
C3—H3B···O1^{ii}	0.93	2.54	3.182 (7)	126

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

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, 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: AT2775).

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supporting information

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S1. Comment

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

The structure of the title compound, (I), is shown in Fig. 1, in which the bond lengths and angles are generally within normal ranges (Allen *et al.*, 1987). The dihedral angle between the thiadiazole and benzene ring is 27.1 (2)°. There are intermolecular N—H···N and C—H···O hydrogen bonds, linking the molecules, forming chains along the *b* axis (Fig. 2),.

S2. Experimental

2-Fluoro-4-nitrobenzoic 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 (I) precipitated and was filted. Pure compound (I) was obtained by crystallization from ethanol (20 ml). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an acetone solution.

S3. Refinement

H atoms were placed geometrically with C—H = 0.93 Å and N—H = 0.86 Å, and included in the refinement in riding motion approximation with U_{iso}(H) = 1.2 or 1.5U_{eq} of the carrier atom.

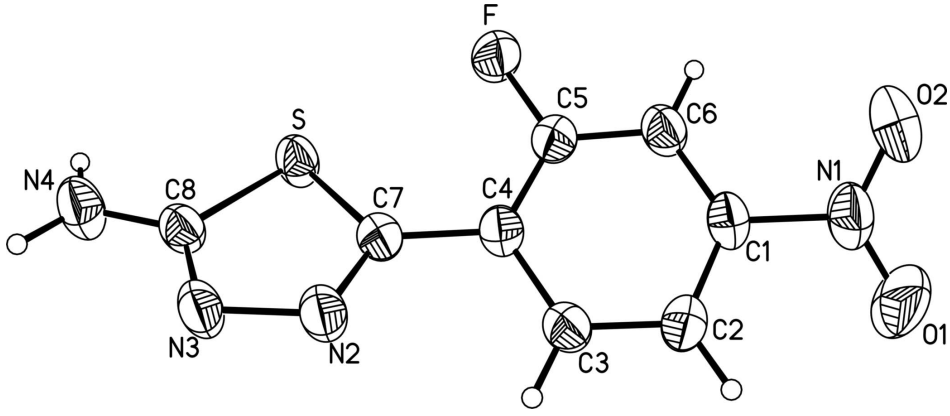

Figure 1

A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

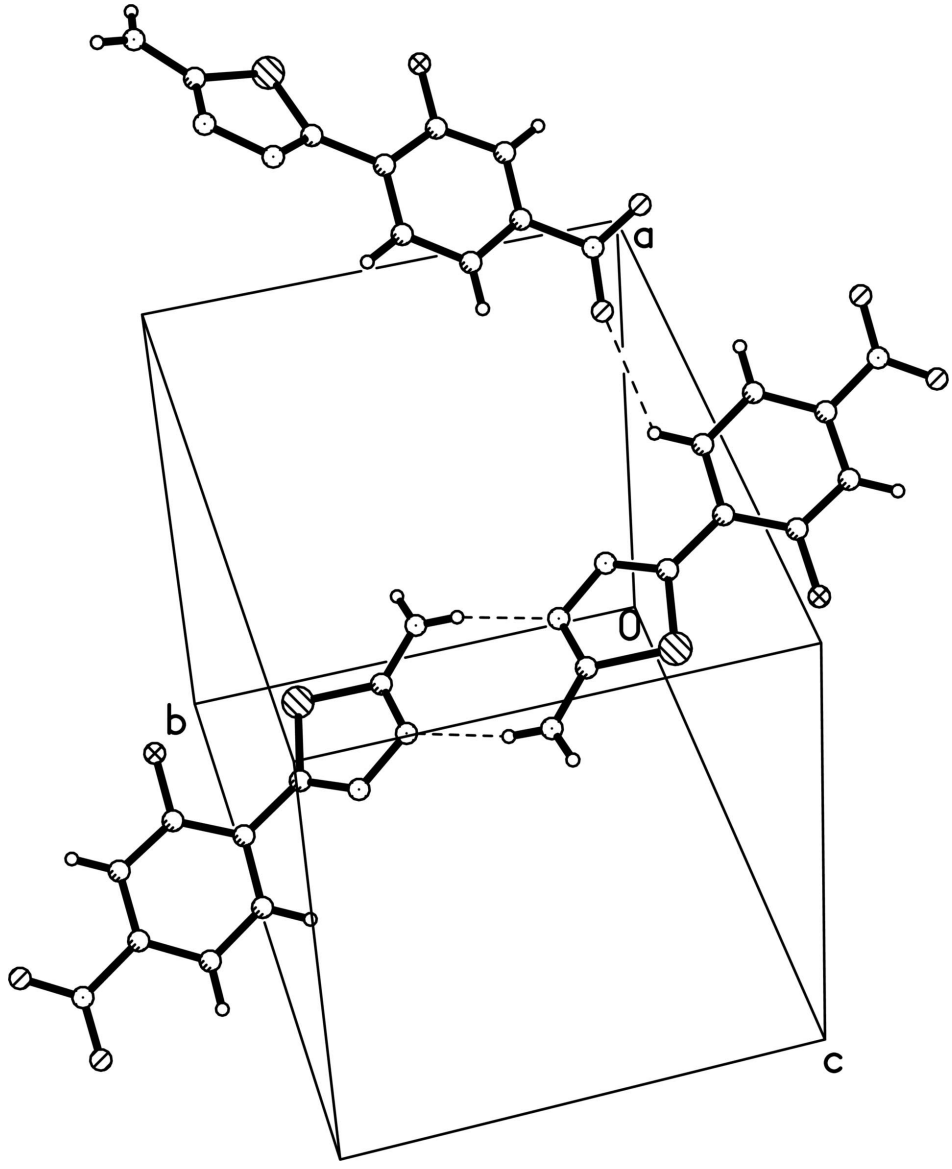

Figure 2

Partial packing view showing the hydrogen-bonded network. Dashed lines indicate intermolecular N—H···N and C—H···O hydrogen bonds.

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Crystal data

$C_8H_5FN_4O_2S$
$M_r = 240.22$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.1780\ (18)$ Å
$b = 9.3720\ (19)$ Å
$c = 11.413\ (2)$ Å
$\beta = 102.55\ (3)°$
$V = 958.2\ (3)$ Å^3
$Z = 4$
$F(000) = 488$
$D_x = 1.665$ Mg m^{-3}
Melting point: 476 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–12°$
$\mu = 0.34$ mm^{-1}
$T = 293$ K
Block, colourless
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.935, T_{max} = 0.967
1832 measured reflections
1720 independent reflections
1301 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 25.2°, θ_{min} = 2.3°
h = 0→11
k = 0→11
l = −13→13
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.194
S = 1.01
1720 reflections
145 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 2.P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 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}
S	0.65883 (14)	0.15613 (12)	0.66472 (10)	0.0442 (4)
F	0.6863 (3)	−0.1515 (3)	0.6466 (2)	0.0539 (8)
N1	0.9259 (5)	−0.4021 (5)	0.3718 (4)	0.0537 (11)
C1	0.8706 (5)	−0.2697 (4)	0.4162 (4)	0.0404 (10)
O1	1.0171 (6)	−0.3935 (5)	0.3093 (4)	0.0892 (15)
O2	0.8765 (4)	−0.5153 (4)	0.4009 (4)	0.0656 (11)
N2	0.6452 (5)	0.2128 (4)	0.4420 (3)	0.0449 (9)
C2	0.8888 (5)	−0.1427 (5)	0.3565 (4)	0.0448 (11)
H2B	0.9387	−0.1415	0.2938	0.054*
N3	0.5854 (5)	0.3311 (4)	0.4867 (3)	0.0475 (10)
C3	0.8298 (5)	−0.0180 (5)	0.3938 (4)	0.0412 (10)
H3B	0.8386	0.0674	0.3544	0.049*
N4	0.5320 (5)	0.4162 (4)	0.6663 (3)	0.0535 (11)
H4A	0.4951	0.4943	0.6329	0.064*
H4B	0.5347	0.4016	0.7412	0.064*

C4	0.7568 (5)	−0.0197 (4)	0.4903 (4)	0.0358 (10)
C5	0.7505 (5)	−0.1484 (5)	0.5495 (4)	0.0388 (10)
C6	0.8026 (5)	−0.2754 (5)	0.5131 (4)	0.0421 (10)
H6A	0.7925	−0.3609	0.5519	0.050*
C7	0.6885 (5)	0.1134 (4)	0.5224 (4)	0.0367 (10)
C8	0.5851 (5)	0.3175 (4)	0.6019 (4)	0.0378 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0663 (8)	0.0343 (6)	0.0343 (6)	0.0124 (5)	0.0166 (5)	0.0053 (4)
F	0.0753 (19)	0.0440 (16)	0.0517 (16)	0.0078 (13)	0.0340 (14)	0.0074 (12)
N1	0.069 (3)	0.038 (2)	0.054 (2)	0.013 (2)	0.012 (2)	−0.0076 (19)
C1	0.047 (2)	0.031 (2)	0.042 (2)	0.0064 (19)	0.0066 (19)	−0.0057 (19)
O1	0.125 (4)	0.067 (3)	0.096 (3)	0.032 (3)	0.068 (3)	−0.003 (2)
O2	0.075 (3)	0.041 (2)	0.075 (3)	0.0038 (18)	0.003 (2)	−0.0161 (19)
N2	0.068 (3)	0.0328 (19)	0.0379 (19)	0.0120 (18)	0.0200 (18)	0.0030 (16)
C2	0.053 (3)	0.040 (3)	0.045 (2)	0.006 (2)	0.019 (2)	−0.005 (2)
N3	0.073 (3)	0.0292 (19)	0.045 (2)	0.0150 (18)	0.0237 (19)	0.0044 (16)
C3	0.055 (3)	0.032 (2)	0.038 (2)	0.0018 (19)	0.014 (2)	0.0020 (18)
N4	0.084 (3)	0.040 (2)	0.039 (2)	0.020 (2)	0.018 (2)	0.0030 (17)
C4	0.040 (2)	0.032 (2)	0.034 (2)	0.0034 (18)	0.0068 (18)	−0.0020 (17)
C5	0.043 (2)	0.037 (2)	0.038 (2)	0.0015 (19)	0.0131 (18)	−0.0014 (18)
C6	0.056 (3)	0.030 (2)	0.041 (2)	0.003 (2)	0.012 (2)	0.0016 (18)
C7	0.042 (2)	0.031 (2)	0.038 (2)	0.0026 (18)	0.0120 (19)	0.0016 (18)
C8	0.048 (2)	0.032 (2)	0.035 (2)	0.0058 (18)	0.0101 (19)	0.0053 (17)

Geometric parameters (Å, º)

S—C8	1.746 (4)	C2—H2B	0.9300
S—C7	1.750 (4)	N3—C8	1.321 (6)
F—C5	1.363 (5)	C3—C4	1.409 (6)
N1—O1	1.214 (6)	C3—H3B	0.9300
N1—O2	1.227 (5)	N4—C8	1.338 (6)
N1—C1	1.472 (6)	N4—H4A	0.8600
C1—C6	1.384 (6)	N4—H4B	0.8600
C1—C2	1.400 (6)	C4—C5	1.389 (6)
N2—C7	1.308 (5)	C4—C7	1.478 (6)
N2—N3	1.382 (5)	C5—C6	1.381 (6)
C2—C3	1.392 (6)	C6—H6A	0.9300
C8—S—C7	86.65 (19)	H4A—N4—H4B	120.0
O1—N1—O2	124.0 (4)	C5—C4—C3	117.9 (4)
O1—N1—C1	118.6 (4)	C5—C4—C7	123.2 (4)
O2—N1—C1	117.4 (4)	C3—C4—C7	118.9 (4)
C6—C1—C2	123.0 (4)	F—C5—C6	117.7 (4)
C6—C1—N1	119.4 (4)	F—C5—C4	119.0 (4)
C2—C1—N1	117.6 (4)	C6—C5—C4	123.3 (4)

C7—N2—N3	113.4 (3)	C5—C6—C1	116.9 (4)
C3—C2—C1	118.0 (4)	C5—C6—H6A	121.5
C3—C2—H2B	121.0	C1—C6—H6A	121.5
C1—C2—H2B	121.0	N2—C7—C4	120.6 (4)
C8—N3—N2	112.1 (3)	N2—C7—S	113.8 (3)
C2—C3—C4	120.8 (4)	C4—C7—S	125.7 (3)
C2—C3—H3B	119.6	N3—C8—N4	124.0 (4)
C4—C3—H3B	119.6	N3—C8—S	114.1 (3)
C8—N4—H4A	120.0	N4—C8—S	121.9 (3)
C8—N4—H4B	120.0		

O1—N1—C1—C6	−161.4 (5)	C4—C5—C6—C1	3.3 (7)
O2—N1—C1—C6	18.5 (6)	C2—C1—C6—C5	0.5 (7)
O1—N1—C1—C2	19.2 (7)	N1—C1—C6—C5	−178.9 (4)
O2—N1—C1—C2	−161.0 (5)	N3—N2—C7—C4	−179.2 (4)
C6—C1—C2—C3	−2.8 (7)	N3—N2—C7—S	0.2 (5)
N1—C1—C2—C3	176.7 (4)	C5—C4—C7—N2	−153.1 (4)
C7—N2—N3—C8	0.0 (6)	C3—C4—C7—N2	25.6 (6)
C1—C2—C3—C4	1.3 (7)	C5—C4—C7—S	27.5 (6)
C2—C3—C4—C5	2.2 (7)	C3—C4—C7—S	−153.7 (4)
C2—C3—C4—C7	−176.6 (4)	C8—S—C7—N2	−0.2 (4)
C3—C4—C5—F	177.2 (4)	C8—S—C7—C4	179.2 (4)
C7—C4—C5—F	−4.0 (7)	N2—N3—C8—N4	−179.3 (4)
C3—C4—C5—C6	−4.7 (7)	N2—N3—C8—S	−0.2 (5)
C7—C4—C5—C6	174.1 (4)	C7—S—C8—N3	0.2 (4)
F—C5—C6—C1	−178.5 (4)	C7—S—C8—N4	179.4 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*A*···N3^{i}	0.86	2.16	3.000 (5)	167
C3—H3*B*···O1^{ii}	0.93	2.54	3.182 (7)	126

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