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

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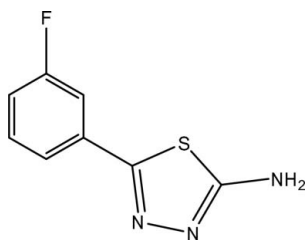
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å;
 R factor = 0.047; wR factor = 0.124; data-to-parameter ratio = 13.4.

The title compound, $\text{C}_8\text{H}_6\text{FN}_3\text{S}$, was synthesized by the reaction of 3-fluorobenzoic acid and thiosemicarbazide. The dihedral angle between the planes of the thiadiazole and benzene rings is $37.3(2)^\circ$. In the structure, two crystallographically independent molecules form a centrosymmetric dimer, in which two intermolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds generate an $R_2^2(8)$ motif.

Related literature

For the biological activity of 1,3,4-thiadiazole derivatives, see: Nakagawa *et al.* (1996); Wang *et al.* (1999). For a similar structure, see: Wan *et al.* (2006). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_8\text{H}_6\text{FN}_3\text{S}$
 $M_r = 195.23$
Monoclinic, $P2_1/c$
 $a = 11.345(2)$ Å
 $b = 7.3130(15)$ Å

$c = 11.269(2)$ Å
 $\beta = 111.64(3)^\circ$
 $V = 869.0(3)$ Å³
 $Z = 4$
Mo $K\alpha$ radiation

$\mu = 0.34$ 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)
 $T_{\text{min}} = 0.935$, $T_{\text{max}} = 0.967$
1584 measured reflections

1584 independent reflections
1177 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.124$
 $S = 1.01$
1584 reflections
118 parameters

13 restraints
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.37$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N3}-\text{H3A}\cdots\text{N2}^i$	0.86	2.14	2.981 (5)	165

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2787).

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supplementary materials

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

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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). We are focusing our synthetic and structural studies on thiadiazole derivatives and have published the structure of 5-(4-fluoro-phenyl)-[1,3,4]thiadiazol-2-ylamine (Wan *et al.*, 2006). Here we report the crystal structure, (I).

In the title molecule (I), (Fig. 1), the dihedral angle between the thiadiazole and benzene ring is $37.3 (2)^\circ$, which is bigger than the angle in the structure of 5-(4-fluoro-phenyl)-[1,3,4]thiadiazol-2-ylamine (Wan *et al.*, 2006), which is $30.1 (2)^\circ$. In the structure, two crystallographically independent molecules form a dimer structure, in which two intermolecular N—H \cdots N hydrogen bonds generate a motif $R_2^2(8)$ (Fig. 2).

Experimental

3-Fluoro-benzoic 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 filtered. 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.

Refinement

All 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 $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$ of the carrier atom.

Figures

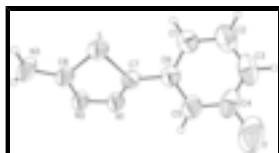


Fig. 1. A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

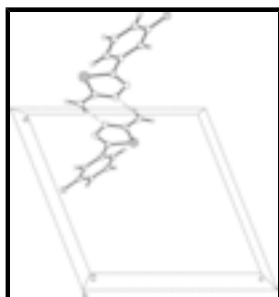


Fig. 2. A view of a dimer structure, in which two intermolecular N—H \cdots N hydrogen bonds generate a motif $R_2^2(8)$.

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

Crystal data

$C_8H_6FN_3S$	$F_{000} = 400$
$M_r = 195.23$	$D_x = 1.492 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Melting point: 515 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation
$a = 11.345 (2) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 7.3130 (15) \text{ \AA}$	Cell parameters from 25 reflections
$c = 11.269 (2) \text{ \AA}$	$\theta = 10\text{--}13^\circ$
$\beta = 111.64 (3)^\circ$	$\mu = 0.34 \text{ mm}^{-1}$
$V = 869.0 (3) \text{ \AA}^3$	$T = 293 \text{ K}$
$Z = 4$	Block, colourless
	$0.20 \times 0.10 \times 0.10 \text{ mm}$

Data collection

Enraf-Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.021$
Radiation source: fine-focus sealed tube	$\theta_{\text{max}} = 25.3^\circ$
Monochromator: graphite	$\theta_{\text{min}} = 1.9^\circ$
$T = 293 \text{ K}$	$h = -13 \rightarrow 0$
$\omega/2\theta$ scans	$k = 0 \rightarrow 8$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$l = -12 \rightarrow 13$
$T_{\text{min}} = 0.935$, $T_{\text{max}} = 0.967$	3 standard reflections
1667 measured reflections	every 200 reflections
1584 independent reflections	intensity decay: 1%
1177 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
$R[F^2 > 2\sigma(F^2)] = 0.047$	H-atom parameters constrained
$wR(F^2) = 0.124$	$w = 1/[\sigma^2(F_o^2) + (0.06P)^2]$
$S = 1.01$	where $P = (F_o^2 + 2F_c^2)/3$
1584 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
118 parameters	$\Delta\rho_{\text{max}} = 0.37 \text{ e \AA}^{-3}$
13 restraints	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S	0.82144 (7)	0.08639 (11)	0.55908 (6)	0.0548 (3)
F	0.5162 (3)	-0.2428 (3)	0.0198 (2)	0.1125 (9)
N1	0.8658 (2)	0.1443 (3)	0.3566 (2)	0.0523 (6)
C1	0.7322 (3)	-0.2918 (5)	0.3984 (3)	0.0713 (9)
H1B	0.7804	-0.3036	0.4850	0.086*
N2	0.9262 (2)	0.2841 (3)	0.4381 (2)	0.0551 (6)
C2	0.6592 (4)	-0.4354 (5)	0.3322 (4)	0.0817 (11)
H2B	0.6585	-0.5443	0.3746	0.098*
N3	0.9606 (3)	0.3931 (4)	0.6429 (2)	0.0733 (9)
H3A	1.0043	0.4846	0.6340	0.088*
H3B	0.9481	0.3787	0.7131	0.088*
C3	0.5871 (4)	-0.4202 (5)	0.2040 (4)	0.0822 (12)
H3C	0.5378	-0.5175	0.1592	0.099*
C4	0.5898 (3)	-0.2597 (6)	0.1447 (3)	0.0741 (10)
C5	0.6637 (3)	-0.1132 (4)	0.2071 (3)	0.0639 (9)
H5A	0.6658	-0.0066	0.1628	0.077*
C6	0.7338 (3)	-0.1272 (4)	0.3350 (3)	0.0516 (7)
C7	0.8082 (3)	0.0307 (4)	0.4039 (2)	0.0475 (7)
C8	0.9121 (3)	0.2733 (4)	0.5476 (2)	0.0499 (7)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0686 (5)	0.0626 (5)	0.0402 (4)	-0.0130 (4)	0.0282 (3)	0.0002 (3)
F	0.1200 (19)	0.122 (2)	0.0888 (16)	-0.0253 (16)	0.0307 (14)	-0.0193 (15)
N1	0.0646 (15)	0.0583 (14)	0.0421 (12)	-0.0113 (12)	0.0293 (11)	-0.0079 (11)
C1	0.070 (2)	0.068 (2)	0.073 (2)	-0.0121 (18)	0.0235 (17)	0.0023 (18)
N2	0.0753 (16)	0.0580 (15)	0.0433 (12)	-0.0172 (13)	0.0353 (12)	-0.0097 (11)
C2	0.074 (2)	0.060 (2)	0.112 (3)	-0.0081 (19)	0.035 (2)	0.001 (2)
N3	0.110 (2)	0.0800 (19)	0.0452 (14)	-0.0379 (17)	0.0462 (15)	-0.0191 (13)
C3	0.074 (2)	0.079 (3)	0.104 (3)	-0.020 (2)	0.045 (2)	-0.040 (2)
C4	0.075 (2)	0.091 (3)	0.062 (2)	-0.024 (2)	0.0319 (18)	-0.0344 (19)

supplementary materials

C5	0.075 (2)	0.068 (2)	0.0514 (17)	-0.0144 (17)	0.0271 (16)	-0.0140 (16)
C6	0.0557 (17)	0.0533 (17)	0.0519 (16)	0.0000 (13)	0.0270 (14)	-0.0051 (13)
C7	0.0541 (16)	0.0513 (16)	0.0421 (14)	0.0000 (13)	0.0236 (13)	-0.0013 (12)
C8	0.0634 (18)	0.0556 (17)	0.0374 (13)	-0.0068 (14)	0.0265 (13)	-0.0025 (13)

Geometric parameters (\AA , $^\circ$)

S—C8	1.744 (3)	C2—H2B	0.9300
S—C7	1.747 (3)	N3—C8	1.338 (3)
F—C4	1.351 (4)	N3—H3A	0.8600
N1—C7	1.288 (3)	N3—H3B	0.8600
N1—N2	1.377 (3)	C3—C4	1.357 (5)
C1—C2	1.375 (5)	C3—H3C	0.9300
C1—C6	1.403 (4)	C4—C5	1.382 (4)
C1—H1B	0.9300	C5—C6	1.369 (4)
N2—C8	1.303 (3)	C5—H5A	0.9300
C2—C3	1.378 (5)	C6—C7	1.472 (4)
C8—S—C7	86.76 (13)	F—C4—C3	118.3 (3)
C7—N1—N2	114.0 (2)	F—C4—C5	119.0 (4)
C2—C1—C6	119.8 (3)	C3—C4—C5	122.7 (3)
C2—C1—H1B	120.1	C6—C5—C4	119.0 (3)
C6—C1—H1B	120.1	C6—C5—H5A	120.5
C8—N2—N1	112.5 (2)	C4—C5—H5A	120.5
C1—C2—C3	120.9 (4)	C5—C6—C1	119.3 (3)
C1—C2—H2B	119.6	C5—C6—C7	119.6 (3)
C3—C2—H2B	119.6	C1—C6—C7	121.1 (3)
C8—N3—H3A	120.0	N1—C7—C6	124.6 (2)
C8—N3—H3B	120.0	N1—C7—S	113.2 (2)
H3A—N3—H3B	120.0	C6—C7—S	122.1 (2)
C4—C3—C2	118.3 (3)	N2—C8—N3	124.3 (3)
C4—C3—H3C	120.9	N2—C8—S	113.5 (2)
C2—C3—H3C	120.9	N3—C8—S	122.19 (19)
C7—N1—N2—C8	-0.2 (4)	N2—N1—C7—S	0.5 (3)
C6—C1—C2—C3	-0.1 (5)	C5—C6—C7—N1	-36.0 (4)
C1—C2—C3—C4	-0.1 (6)	C1—C6—C7—N1	144.6 (3)
C2—C3—C4—F	-178.1 (3)	C5—C6—C7—S	141.4 (3)
C2—C3—C4—C5	1.3 (6)	C1—C6—C7—S	-37.9 (4)
F—C4—C5—C6	177.0 (3)	C8—S—C7—N1	-0.4 (2)
C3—C4—C5—C6	-2.4 (5)	C8—S—C7—C6	-178.2 (2)
C4—C5—C6—C1	2.2 (5)	N1—N2—C8—N3	-179.5 (3)
C4—C5—C6—C7	-177.2 (3)	N1—N2—C8—S	-0.1 (3)
C2—C1—C6—C5	-1.0 (5)	C7—S—C8—N2	0.3 (2)
C2—C1—C6—C7	178.4 (3)	C7—S—C8—N3	179.7 (3)
N2—N1—C7—C6	178.1 (2)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H3A \cdots N2 ⁱ	0.86	2.14	2.981 (5)	165

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

Fig. 1

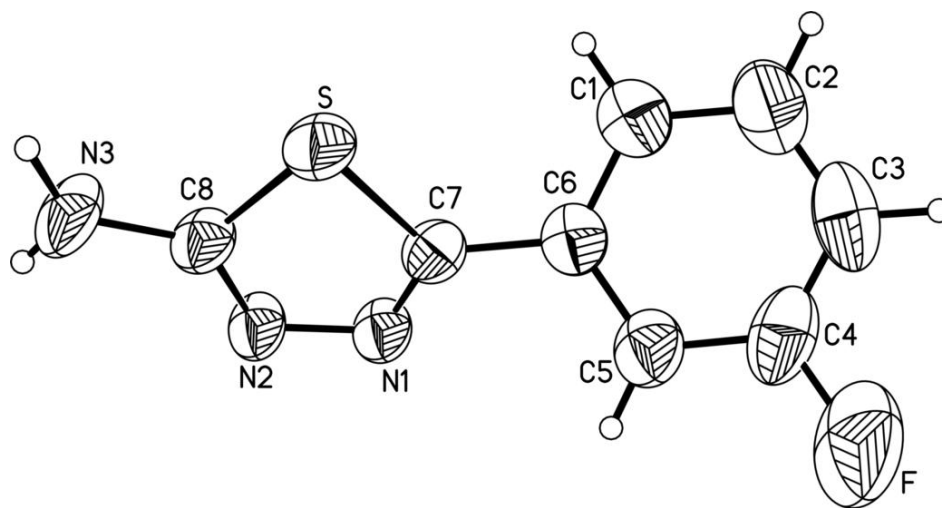


Fig. 2

