Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.048
wR factor = 0.133
Data-to-parameter ratio = 14.4

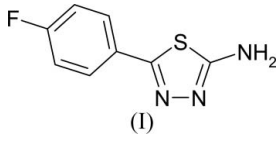
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 19 September 2006
Accepted 27 September 2006

The title compound, $C_8H_6FN_3S$, was synthesized by the reaction of 4-fluorobenzoic acid and thiosemicarbazide. The two H atoms of the amine are involved in N—H···N hydrogen bonding, resulting in the formation of layers parallel to the (100) plane.

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 and fungicidal activities (Wang *et al.*, 1999) and are widely used in the field of medicine (Sato *et al.*, 1991), for example as anti-cancer drugs (Omar *et al.*, 1986). We are focusing our synthetic and structural studies on thiadiazole derivatives and have recently published the structure of 3-[5-(4-fluorophenyl)-1,3,4-thiadiazol-2-yl]-2-phenylthiazolidin-4-one, (II) (Wan *et al.*, 2006).



(I)

The structure of the title compound, (I), is shown in Fig. 1. The thiadiazole and the fluorophenyl rings make a dihedral angle of 30.1 (2)°, much larger than that observed in the structure of (II) [12.7 (2)°].

N—H···N hydrogen bonds form a pseudo-dimer using one H atom of the NH_2 group whereas the other H atom links the dimers, forming an infinite layer parallel to the (100) plane (Table 1 and Fig. 2).

Experimental

4-Fluorobenzoic acid (5 mmol) and thiosemicarbazide (5 mmol) were added to toluene (50 ml), which was heated under reflux for 4 h. The

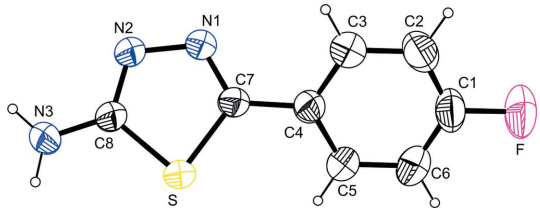


Figure 1
A view of the molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level. H atoms are represented as spheres of arbitrary radii.

reaction mixture was left to cool to room temperature, poured into ice–water and filtered. The filter cake was crystallized from acetone to give pure compound (I) (m.p. 513–514 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an acetone solution.

Crystal data

$C_8H_6FN_3S$
M_r = 195.22
Monoclinic, $P2_1/c$
a = 8.8020 (18) Å
b = 8.9610 (18) Å
c = 11.035 (2) Å
β = 97.19 (3)°
V = 863.5 (3) Å^3
Z = 4
D_x = 1.502 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 293 (2) K
Plate, yellow
0.40 × 0.30 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.875, T_{max} = 0.967
1810 measured reflections
1699 independent reflections
1378 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 26.0°
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.133
S = 1.03
1699 reflections
118 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0831P)^2 + 0.1422P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···N2^{i}	0.86	2.14	2.987 (3)	170
N3—H3B···N1^{ii}	0.86	2.27	3.065 (3)	155

Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

All H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H = 0.93 Å, N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(C,N)

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, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

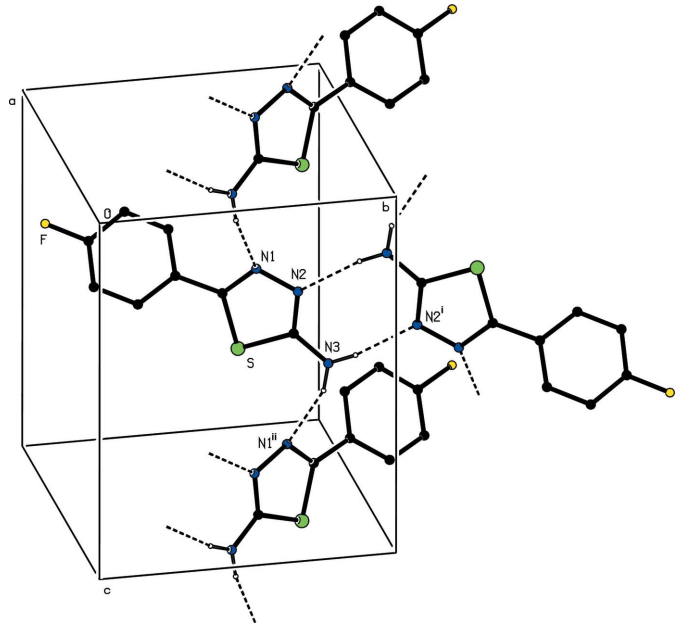


Figure 2
Partial packing view showing the hydrogen-bonded (dashed lines) network. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes: (i) $1-x, 2-y, 1-z$; (ii) $x, \frac{3}{2}-y, \frac{1}{2}+z$.]

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