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5-(2,3,4,5,6-Pentafluorophenyl)-1,3,4-thiadiazol-2-amine

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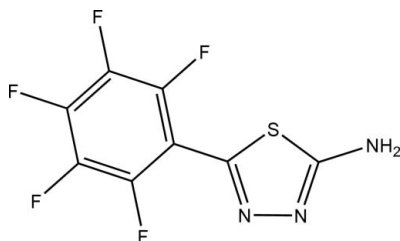
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.043; wR factor = 0.128; data-to-parameter ratio = 11.1.

The title compound, $\text{C}_8\text{H}_2\text{F}_5\text{N}_3\text{S}$, was synthesized by the reaction of perfluorobenzoic acid and thiosemicarbazide. The dihedral angle between the thiadiazole and perfluorophenyl ring is $35.41(6)^\circ$. In the crystal, intermolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds link the molecules, forming a three-dimensional network.

Related literature

For the fungicidal and herbicidal activity of thiadiazole derivatives, see: Chen *et al.* (2000); Kidwai *et al.* (2000); Vicentini *et al.* (1998) and for their insecticidal activity, see: Arun *et al.* (1999); Wasfy *et al.* (1996). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_8\text{H}_2\text{F}_5\text{N}_3\text{S}$
 $M_r = 267.19$
 Monoclinic, $P2_1/c$
 $a = 11.897(2)$ Å

$b = 7.0680(14)$ Å
 $c = 11.553(2)$ Å
 $\beta = 104.66(3)^\circ$
 $V = 939.8(3)$ Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.40$ mm⁻¹

$T = 293$ K
 $0.30 \times 0.20 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.889$, $T_{\max} = 0.961$
 3428 measured reflections

1709 independent reflections
 1283 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.064$
 3 standard reflections every 200 reflections
 intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.128$
 $S = 1.00$
 1709 reflections

154 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.27$ e Å⁻³
 $\Delta\rho_{\min} = -0.29$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N1}-\text{H1A}\cdots\text{N2}^i$ | 0.86 | 2.18 | 3.001 (4) | 160 |
| $\text{N1}-\text{H1B}\cdots\text{N3}^{ii}$ | 0.86 | 2.19 | 3.013 (3) | 161 |

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); 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 would like to thank Professor Hua-qin Wang of the Analysis Centre, Nanjing University, for carrying out the X-ray crystallographic analysis.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2081).

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supplementary materials

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5-(2,3,4,5,6-Pentafluorophenyl)-1,3,4-thiadiazol-2-amine

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Comment

Thiadiazole derivatives containing the thiazolidinone unit are of great interest because of their chemical and pharmaceutical properties. Some derivatives have fungicidal and herbicidal activities (Chen *et al.*, 2000; Kidwai *et al.*, 2000; Vicentini *et al.*, 1998); some show insecticidal activities (Arun *et al.*, 1999; Wasfy *et al.*, 1996).

We report here the crystal structure of the title compound, (I). The molecular structure of (I) is shown in Fig. 1, in which the bond lengths (Allen *et al.*, 1987) and angles are generally within normal ranges. Ring(C1/S/C2/N3/N2) is planar, and the mean deviation from plane is 0.0012 Å. The dihedral angle between the thiadiazole and perfluorophenyl ring is 35.41 (6)°. In the crystal structure, intermolecular N—H···N hydrogen bonds (Table 1) link the molecules to form a three-dimensional network (Fig. 2), in which they may be effective in the stabilization of the structure.

Experimental

Perfluorobenzoic acid (5 mmol) and thiosemicarbazide (5 mmol) were added in toluene (50 ml), which is heated under reflux for 4 h. The reaction mixture was left to cool to room temperature, poured into ice water, filtered, and the filter cake was crystallized from acetone to give pure compound (I) (m.p. 523–525 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an acetone solution.

Refinement

All H atoms bonded to the C atoms were placed geometrically at the distances of 0.93–0.97 Å and included in the refinement in riding motion approximation with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5U_{\text{eq}}$ of the carrier atom.

Figures

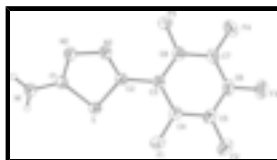


Fig. 1. A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

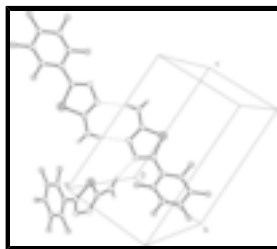


Fig. 2. Partial packing view showing the hydrogen-bonded network. Dashed lines indicate intermolecular N—H···N hydrogen bonds.

5-(2,3,4,5,6-Pentafluorophenyl)-1,3,4-thiadiazol-2-amine

Crystal data

| | |
|-------------------------------|---|
| $C_8H_2F_5N_3S$ | $F(000) = 528$ |
| $M_r = 267.19$ | $D_x = 1.888 \text{ Mg m}^{-3}$ |
| Monoclinic, $P2_1/c$ | Melting point = 523–525 K |
| Hall symbol: -P 2ybc | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| $a = 11.897 (2) \text{ \AA}$ | Cell parameters from 25 reflections |
| $b = 7.0680 (14) \text{ \AA}$ | $\theta = 9\text{--}13^\circ$ |
| $c = 11.553 (2) \text{ \AA}$ | $\mu = 0.40 \text{ mm}^{-1}$ |
| $\beta = 104.66 (3)^\circ$ | $T = 293 \text{ K}$ |
| $V = 939.8 (3) \text{ \AA}^3$ | Block, colorless |
| $Z = 4$ | $0.30 \times 0.20 \times 0.10 \text{ mm}$ |

Data collection

| | |
|---|--|
| Enraf–Nonius CAD-4 diffractometer | 1283 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{\text{int}} = 0.064$ |
| graphite | $\theta_{\text{max}} = 25.3^\circ$, $\theta_{\text{min}} = 1.8^\circ$ |
| $\omega/2\theta$ scans | $h = -14 \rightarrow 0$ |
| Absorption correction: ψ scan (North <i>et al.</i> , 1968) | $k = -8 \rightarrow 8$ |
| $T_{\text{min}} = 0.889$, $T_{\text{max}} = 0.961$ | $l = -13 \rightarrow 13$ |
| 3428 measured reflections | 3 standard reflections every 200 reflections |
| 1709 independent reflections | intensity decay: 1% |

Refinement

| | |
|---------------------------------|--|
| Refinement on F^2 | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.043$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.128$ | H-atom parameters constrained |
| $S = 1.00$ | $w = 1/[\sigma^2(F_o^2) + (0.080P)^2]$ |
| 1709 reflections | where $P = (F_o^2 + 2F_c^2)/3$ |
| 154 parameters | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 0 restraints | $\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$ |
| | $\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$ |

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|---------------|----------------------------------|
| S | 0.82940 (7) | 0.06662 (10) | 0.05893 (6) | 0.0461 (3) |
| F1 | 0.78775 (15) | -0.3360 (2) | -0.00245 (14) | 0.0569 (5) |
| C1 | 0.9155 (2) | 0.2602 (4) | 0.0479 (2) | 0.0404 (6) |
| N1 | 0.9659 (2) | 0.3683 (4) | 0.1416 (2) | 0.0524 (6) |
| H1A | 1.0105 | 0.4601 | 0.1330 | 0.063* |
| H1B | 0.9537 | 0.3459 | 0.2106 | 0.063* |
| C2 | 0.8165 (2) | 0.0294 (4) | -0.0925 (2) | 0.0389 (6) |
| F2 | 0.65099 (17) | -0.6042 (2) | -0.12921 (18) | 0.0685 (6) |
| N2 | 0.9298 (2) | 0.2855 (4) | -0.06011 (19) | 0.0472 (6) |
| F3 | 0.53266 (17) | -0.5342 (3) | -0.35867 (18) | 0.0747 (6) |
| N3 | 0.8725 (2) | 0.1519 (3) | -0.13946 (19) | 0.0451 (6) |
| C3 | 0.7437 (2) | -0.1198 (4) | -0.1630 (2) | 0.0387 (6) |
| F4 | 0.55308 (16) | -0.1942 (3) | -0.45890 (14) | 0.0689 (6) |
| C4 | 0.7312 (2) | -0.2972 (4) | -0.1154 (2) | 0.0429 (6) |
| F5 | 0.68393 (17) | 0.0780 (2) | -0.33282 (15) | 0.0609 (5) |
| C5 | 0.6609 (3) | -0.4358 (4) | -0.1800 (3) | 0.0489 (7) |
| C6 | 0.6009 (2) | -0.4016 (4) | -0.2954 (3) | 0.0502 (7) |
| C7 | 0.6116 (2) | -0.2282 (4) | -0.3459 (2) | 0.0482 (7) |
| C8 | 0.6812 (2) | -0.0900 (4) | -0.2800 (2) | 0.0453 (7) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|-------------|-------------|-------------|--------------|-------------|--------------|
| S | 0.0633 (5) | 0.0429 (4) | 0.0347 (4) | -0.0129 (3) | 0.0171 (3) | -0.0005 (3) |
| F1 | 0.0665 (10) | 0.0469 (10) | 0.0521 (10) | -0.0049 (8) | 0.0052 (8) | 0.0130 (8) |
| C1 | 0.0472 (14) | 0.0406 (15) | 0.0352 (13) | -0.0044 (12) | 0.0139 (12) | -0.0027 (11) |
| N1 | 0.0725 (16) | 0.0530 (15) | 0.0339 (12) | -0.0221 (13) | 0.0176 (11) | -0.0081 (10) |
| C2 | 0.0477 (14) | 0.0358 (14) | 0.0354 (13) | -0.0023 (11) | 0.0147 (12) | -0.0012 (11) |
| F2 | 0.0801 (13) | 0.0418 (10) | 0.0824 (14) | -0.0148 (9) | 0.0184 (11) | 0.0058 (9) |
| N2 | 0.0600 (14) | 0.0494 (14) | 0.0368 (12) | -0.0185 (11) | 0.0206 (11) | -0.0092 (10) |
| F3 | 0.0815 (13) | 0.0659 (13) | 0.0740 (13) | -0.0330 (10) | 0.0145 (11) | -0.0233 (10) |
| N3 | 0.0586 (14) | 0.0442 (13) | 0.0362 (12) | -0.0136 (11) | 0.0191 (11) | -0.0087 (10) |
| C3 | 0.0450 (14) | 0.0351 (14) | 0.0378 (13) | -0.0027 (11) | 0.0139 (11) | -0.0040 (11) |
| F4 | 0.0762 (12) | 0.0821 (14) | 0.0411 (9) | -0.0162 (11) | 0.0012 (8) | -0.0056 (9) |
| C4 | 0.0458 (15) | 0.0405 (15) | 0.0426 (15) | -0.0010 (12) | 0.0114 (12) | 0.0025 (12) |
| F5 | 0.0840 (12) | 0.0443 (10) | 0.0477 (10) | -0.0082 (9) | 0.0041 (9) | 0.0086 (7) |
| C5 | 0.0553 (16) | 0.0349 (15) | 0.0603 (18) | -0.0060 (13) | 0.0217 (14) | -0.0005 (13) |

supplementary materials

| | | | | | | |
|----|-------------|-------------|-------------|--------------|-------------|--------------|
| C6 | 0.0508 (16) | 0.0486 (17) | 0.0531 (17) | -0.0159 (14) | 0.0165 (14) | -0.0172 (13) |
| C7 | 0.0492 (16) | 0.0547 (18) | 0.0406 (15) | -0.0051 (13) | 0.0108 (13) | -0.0087 (13) |
| C8 | 0.0542 (16) | 0.0404 (15) | 0.0429 (15) | -0.0015 (12) | 0.0155 (13) | -0.0008 (11) |

Geometric parameters (Å, °)

| | | | |
|-------------|-------------|-------------|------------|
| S—C1 | 1.733 (3) | N2—N3 | 1.372 (3) |
| S—C2 | 1.737 (3) | F3—C6 | 1.331 (3) |
| F1—C4 | 1.336 (3) | C3—C8 | 1.383 (4) |
| C1—N2 | 1.313 (3) | C3—C4 | 1.392 (4) |
| C1—N1 | 1.337 (3) | F4—C7 | 1.336 (3) |
| N1—H1A | 0.8600 | C4—C5 | 1.379 (4) |
| N1—H1B | 0.8600 | F5—C8 | 1.339 (3) |
| C2—N3 | 1.292 (3) | C5—C6 | 1.365 (5) |
| C2—C3 | 1.473 (4) | C6—C7 | 1.377 (4) |
| F2—C5 | 1.345 (3) | C7—C8 | 1.379 (4) |
| C1—S—C2 | 87.05 (12) | F1—C4—C5 | 118.0 (3) |
| N2—C1—N1 | 123.5 (2) | F1—C4—C3 | 119.5 (2) |
| N2—C1—S | 113.5 (2) | C5—C4—C3 | 122.4 (3) |
| N1—C1—S | 122.92 (19) | F2—C5—C6 | 120.1 (3) |
| C1—N1—H1A | 120.0 | F2—C5—C4 | 120.0 (3) |
| C1—N1—H1B | 120.0 | C6—C5—C4 | 119.9 (3) |
| H1A—N1—H1B | 120.0 | F3—C6—C5 | 120.5 (3) |
| N3—C2—C3 | 122.8 (2) | F3—C6—C7 | 120.2 (3) |
| N3—C2—S | 113.4 (2) | C5—C6—C7 | 119.3 (2) |
| C3—C2—S | 123.72 (19) | F4—C7—C6 | 119.5 (2) |
| C1—N2—N3 | 112.3 (2) | F4—C7—C8 | 120.3 (3) |
| C2—N3—N2 | 113.8 (2) | C6—C7—C8 | 120.2 (3) |
| C8—C3—C4 | 116.0 (2) | F5—C8—C7 | 117.2 (2) |
| C8—C3—C2 | 121.8 (2) | F5—C8—C3 | 120.8 (2) |
| C4—C3—C2 | 122.2 (2) | C7—C8—C3 | 122.1 (3) |
| C2—S—C1—N2 | -0.2 (2) | F1—C4—C5—C6 | 179.5 (3) |
| C2—S—C1—N1 | -177.7 (3) | C3—C4—C5—C6 | 0.3 (4) |
| C1—S—C2—N3 | 0.2 (2) | F2—C5—C6—F3 | -0.4 (4) |
| C1—S—C2—C3 | -176.3 (2) | C4—C5—C6—F3 | -179.9 (3) |
| N1—C1—N2—N3 | 177.7 (3) | F2—C5—C6—C7 | 179.7 (2) |
| S—C1—N2—N3 | 0.2 (3) | C4—C5—C6—C7 | 0.2 (4) |
| C3—C2—N3—N2 | 176.4 (2) | F3—C6—C7—F4 | 0.0 (4) |
| S—C2—N3—N2 | -0.2 (3) | C5—C6—C7—F4 | 179.9 (3) |
| C1—N2—N3—C2 | 0.0 (4) | F3—C6—C7—C8 | 179.3 (3) |
| N3—C2—C3—C8 | -34.3 (4) | C5—C6—C7—C8 | -0.8 (4) |
| S—C2—C3—C8 | 141.9 (2) | F4—C7—C8—F5 | 2.1 (4) |
| N3—C2—C3—C4 | 147.3 (3) | C6—C7—C8—F5 | -177.2 (3) |
| S—C2—C3—C4 | -36.4 (4) | F4—C7—C8—C3 | -179.6 (2) |
| C8—C3—C4—F1 | -179.2 (2) | C6—C7—C8—C3 | 1.1 (4) |
| C2—C3—C4—F1 | -0.8 (4) | C4—C3—C8—F5 | 177.6 (2) |
| C8—C3—C4—C5 | 0.0 (4) | C2—C3—C8—F5 | -0.8 (4) |
| C2—C3—C4—C5 | 178.4 (3) | C4—C3—C8—C7 | -0.6 (4) |
| F1—C4—C5—F2 | -0.1 (4) | C2—C3—C8—C7 | -179.1 (3) |

C3—C4—C5—F2 -179.3 (2)

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N1—H1A \cdots N2 ⁱ | 0.86 | 2.18 | 3.001 (4) | 160 |
| N1—H1B \cdots N3 ⁱⁱ | 0.86 | 2.19 | 3.013 (3) | 161 |

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

Fig. 1

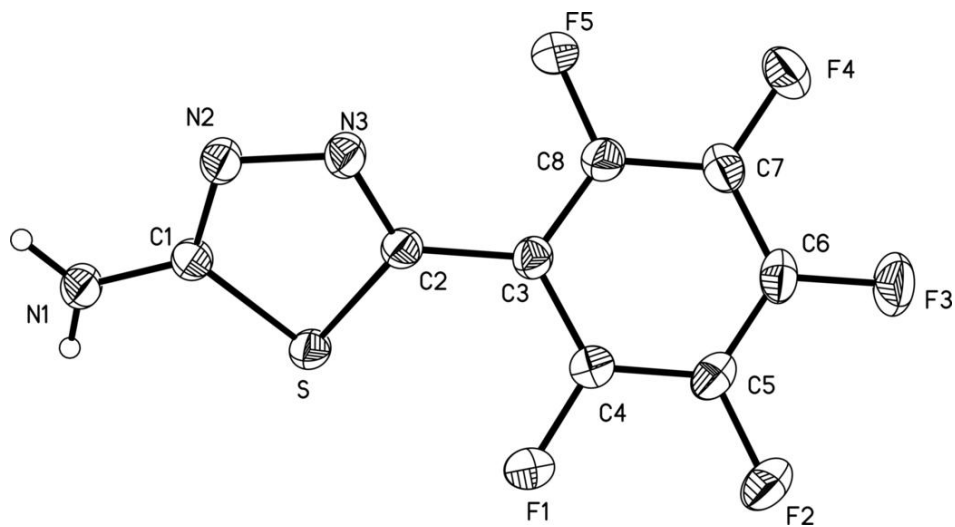


Fig. 2

