Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

1-(2,6-Difluorobenzoyl)-3-(1,3,4-thiadiazol-2-yl)urea

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.005 Å
R factor = 0.049
wR factor = 0.144
Data-to-parameter ratio = 13.6

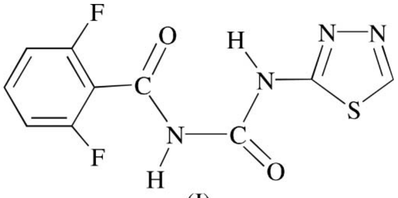
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 2 June 2005
Accepted 27 June 2005
Online 6 July 2005

In the title compound, $C_{10}H_6F_2N_4O_2S$, the urea group is essentially coplanar with the thiadiazole ring and nearly perpendicular to the benzene ring. The face-to-face distance of 3.2225 (15) Å and the centroid separation of 3.5862 (17) Å between parallel thiadiazole rings indicate the existence of π–π stacking between neighbouring molecules. There are two molecules in the asymmetric unit.

Comment

Aroyl ureas can be used as insecticides, herbicides and plant-growth regulators (Wang *et al.*, 1998, 2004). 1,3,4-Thiadiazole derivatives have shown significant biological activities (Nakagawa *et al.*, 1996; Wang *et al.*, 1999). As part of our ongoing investigation of aroyl ureas containing a 1,3,4-thiadiazole group as plant-growth regulators, the title compound, (I), has recently been prepared in our laboratory. We present its X-ray crystal structure here, to provide a basis for consideration of stereochemical structure–activity relationships.



(I)

The molecular structure of (I) is shown in Fig. 1. The asymmetric unit of the crystal contains two independent

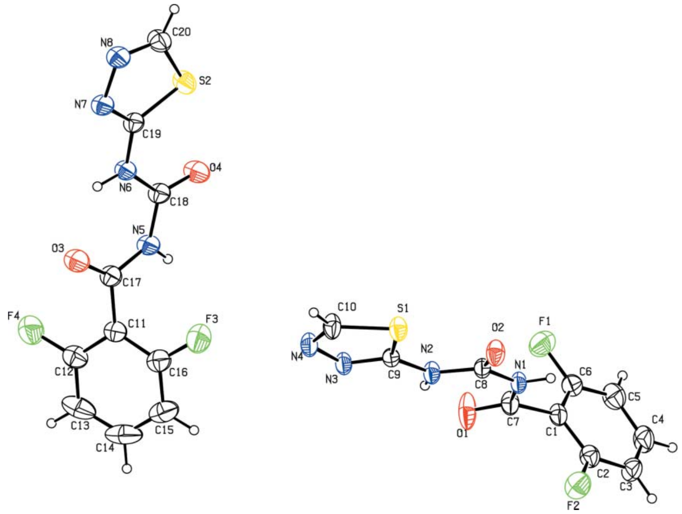


Figure 1
The asymmetric unit of (I), with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

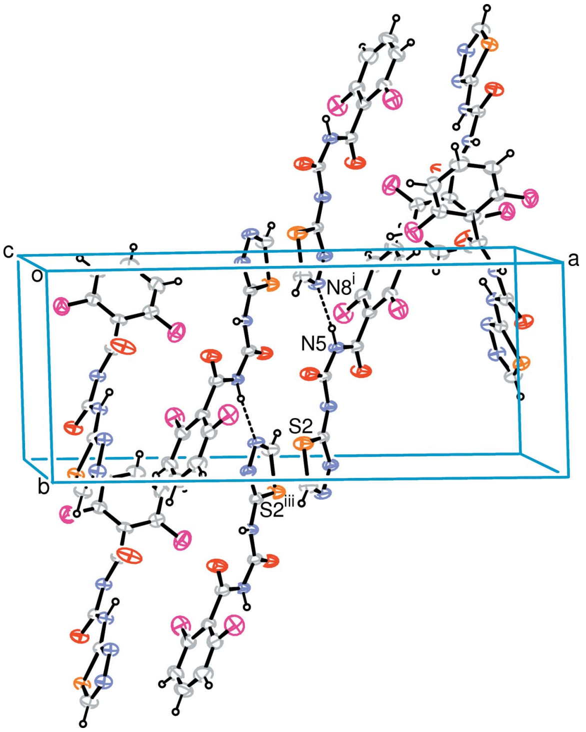


Figure 2
A packing diagram, showing the intermolecular hydrogen bonds (dashed lines) and π–π stacking [symmetry codes: (i) $x, y - 1, z$; (iii) $1 - x, 2 - y, 1 - z$].

molecules with similar structures (Table 1). The urea group of (I) is essentially coplanar with the thiadiazole ring and nearly perpendicular to the benzene ring [dihedral angles 5.6 (2) and 69.8 (1)° for the S1-containing molecule, and 9.0 (1) and 55.3 (1)° for the S2-containing molecule].

Neighbouring molecules are linked by intermolecular N—H$\cdots$N hydrogen bonding between the thiadiazole and imino groups (Fig. 2 and Table 2). There is π–π stacking between parallel thiadiazole rings of neighbouring molecules (Fig. 2). The face-to-face distance is 3.2225 (15) Å and the centroid separation is 3.5862 (17) Å.

Experimental

Compound (I) was prepared according to the procedure of Li *et al.* (2003). Suitable single crystals were obtained by slow evaporation of an ethyl acetate–acetone solution (1:2) at room temperature (m.p. 472 K). Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 3186, 3099, 1720, 1696; ^{1}H NMR (DMSO-d_6, δ, p.p.m.): 11.79 (*d*, 2H), 9.21 (*s*, 1H), 7.72–7.24 (*m*, 3H). Elemental analysis, calculated for $C_{10}H_6F_2N_4O_2S$: C 42.11, H 2.12, N 19.91%; found: C 42.23, H 2.19, N 19.98%.

Crystal data

$C_{10}H_6F_2N_4O_2S$
$M_r = 284.26$
Monoclinic, $P2_1/c$
$a = 20.674$ (2) Å
$b = 8.2442$ (9) Å
$c = 14.9541$ (16) Å
$\beta = 111.106$ (2)°
$V = 2377.8$ (4) Å^3
$Z = 8$
$D_x = 1.588$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 3500 reflections
$\theta = 2.7$–24.1°
$\mu = 0.30$ mm^{-1}
$T = 292$ (2) K
Block, colourless
0.40 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.889$, $T_{\max} = 0.970$
12506 measured reflections
4661 independent reflections
3583 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\max} = 26.0°$
$h = -25 \rightarrow 18$
$k = -9 \rightarrow 10$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.144$
$S = 1.09$
4661 reflections
343 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0741P)^2 + 0.3849P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

Table 1
Selected bond lengths (Å).

C7—O1	1.213 (3)	C17—O3	1.214 (3)
C7—N1	1.360 (3)	C17—N5	1.367 (3)
C8—O2	1.209 (3)	C18—O4	1.206 (3)
C8—N2	1.360 (3)	C18—N6	1.355 (3)
C8—N1	1.387 (3)	C18—N5	1.397 (3)
C9—N2	1.383 (3)	C19—N6	1.368 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N6—H6$\cdots$O3	0.86	1.99	2.653 (3)	133
N2—H2$\cdots$O1	0.86	1.97	2.648 (3)	134
N5—H5*A*$\cdots$N8$^{\rm i}$	0.86	2.00	2.860 (3)	175
N1—H1$\cdots$N4$^{\rm ii}$	0.86	2.02	2.875 (3)	178

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

All H atoms were placed in geometrically idealized positions with C—H = 0.93 Å and N—H = 0.86 Å, and constrained to ride on their parent atoms with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(carrier).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2001); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge financial support from the Hubei Provincial Department of Education Scientific Research Fund for Distinguished Young Scholars (grant No. Q200529003).

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Acta Cryst. (2005). E**61**, o2360–o2362 Song *et al.* • $C_{10}H_6F_2N_4O_2S$ **o2361**

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