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1-(4-Methylbenzoyl)-3-{5-[4-(trifluoromethyl)-phenyl]-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) = 0.004 Å
R factor = 0.054
wR factor = 0.177
Data-to-parameter ratio = 13.5

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

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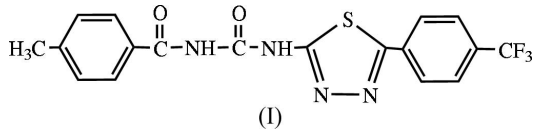
The title compound, $C_{18}H_{13}F_3N_4O_2S$, forms stacks *via* a three-dimensional hydrogen-bonding network, which involves intramolecular N—H···O, intermolecular C—H···O and paired N—H···O hydrogen bonds.

Comment

1,3,4-Thiadiazole derivatives have been found to possess many important bioactivities (Wang *et al.*, 1999; Nakagawa *et al.*, 1996). Aroyl ureas can be used as insecticides, herbicides and plant-growth regulators (Wang *et al.*, 1998, 2004). As part of our continuing interest in aroyl ureas containing the 1,3,4-thiadiazole group, we have synthesized the title compound, (I).



(I)

The crystal structure of (I) (Fig. 1) reveals that the two benzene rings in the molecule are approximately coplanar, with a dihedral angle of 3.2 (1)°, but neither of them is coplanar with the thiadiazole plane. Complementary hydrogen bonding (N—H···O) between centrosymmetrically related molecules is observed. The acyl urea scaffold adopts the most stable configuration, as shown in Fig. 1, mediated by the intramolecular N—H···O hydrogen bond. The carbonyl O atom also participates in intermolecular interactions (C—H···O) with another neighbouring molecule. The molecules thus form stacks *via* a three-dimensional hydrogen-bonding network (Fig. 2).

Experimental

The title compound, (I), was prepared according to the procedure of Wang *et al.* (2003). Suitable crystals were obtained by vapour diffusion of methanol in dimethylformamide at room temperature (m.p. > 573 K). IR (KBr, ν cm^{-1}): 3262, 3138, 1701, 1673, 1542; ^{1}H NMR (DMSO-d_6): δ 12.32 (*s*, 1H), 11.63 (*s*, 1H), 8.23–7.38 (*m*, 8H), 2.41 (*s*,

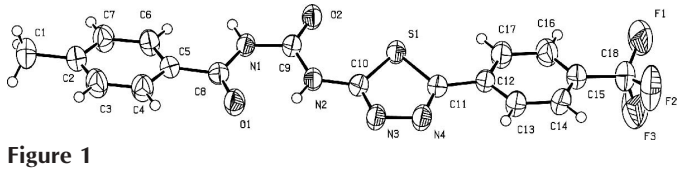


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

3H); analysis calculated for $C_{18}H_{13}F_3N_4O_2S$: C 53.20, H 3.22, N 13.79%; found: C 53.27, H 3.12, N 13.91%.

Crystal data

$C_{18}H_{13}F_3N_4O_2S$
M_r = 406.38
Monoclinic, $P2_1/c$
a = 16.4728 (16) Å
b = 13.8150 (14) Å
c = 7.6914 (8) Å
β = 93.829 (2)°
V = 1746.4 (3) Å^3
Z = 4
D_x = 1.546 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1950 reflections
θ = 2.5–22.7°
μ = 0.24 mm^{-1}
T = 292 (2) K
Plate, colourless
0.60 × 0.25 × 0.06 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.870, T_{max} = 0.986
9386 measured reflections
3428 independent reflections
2474 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 26.0°
$h = -20 \rightarrow 20$
$k = -17 \rightarrow 12$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.177
S = 1.06
3428 reflections
254 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1051P)^2 + 0.1106P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.506 (4)	C9—N2	1.349 (3)
C8—O1	1.223 (3)	C9—N1	1.380 (3)
C8—N1	1.383 (3)	C11—C12	1.468 (4)
C9—O2	1.220 (3)	C15—C18	1.492 (4)
N1—C8—C5	117.1 (2)	C9—N1—C8	127.7 (2)
N2—C9—N1	116.5 (2)	C9—N2—C10	124.2 (2)
C6—C5—C8—N1	16.0 (4)	C5—C8—N1—C9	−177.2 (2)
S1—C11—C12—C17	−13.7 (4)	N1—C9—N2—C10	179.5 (2)
N2—C9—N1—C8	3.1 (4)	N2—C10—S1—C11	179.1 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O1^{i}	0.93	2.55	3.388 (4)	150
N1—H1···O2^{ii}	0.86	2.13	2.939 (3)	156
N2—H2···O1	0.86	1.92	2.602 (3)	135

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

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C), but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93 Å, N—H distances of 0.86 Å and U_{iso}(H) = 1.2U_{eq}(C,N).

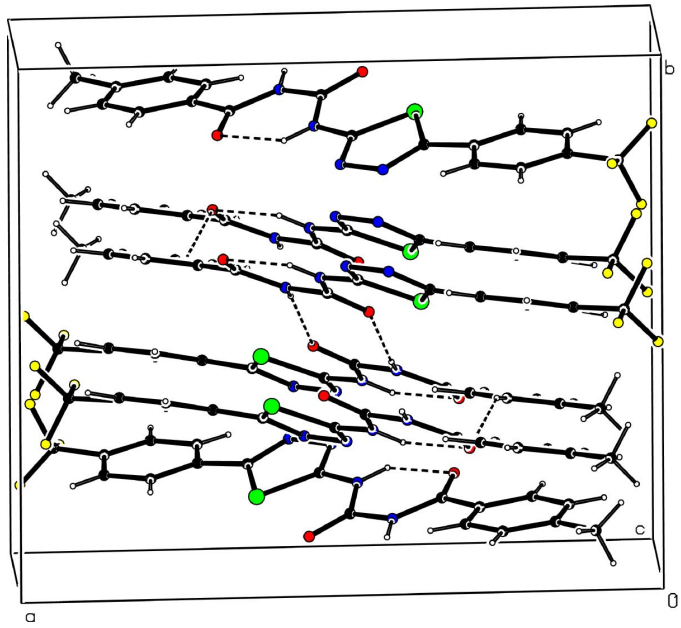


Figure 2
The molecular packing of (I), viewed approximately along the c axis. The hydrogen-bonding interactions are indicated by dashed lines.

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*.

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