

1-(4-Bromobenzoyl)-3-(5-trifluoromethyl-1,3,4-thiadiazol-2-yl)urea

Yin-Xiang Gong^{a*} and
Xiao-Hong Yang^b^aCollege of Chemistry and Environmental Engineering, Yangtze University, Jingzhou, Hubei 434023, People's Republic of China, and^bSchool of Chemical and Environmental Engineering, Hubei Institute for Nationalities, Enshi, Hubei 445000, People's Republic of China

Correspondence e-mail: xjsong@126.com

Key indicators

Single-crystal X-ray study

T = 292 K

Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$

Disorder in main residue

R factor = 0.043

wR factor = 0.115

Data-to-parameter ratio = 14.7

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

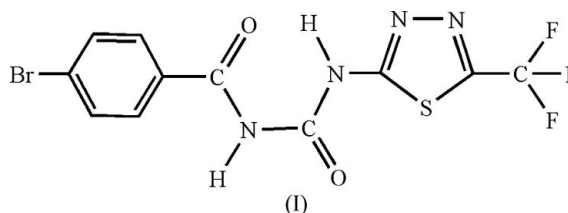
There are two molecules in the asymmetric unit of the title compound, $\text{C}_{11}\text{H}_6\text{ClF}_3\text{N}_4\text{O}_2\text{S}$. The urea linkage in each molecule is essentially planar due to the presence of intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. The crystal structure is stabilized by several intermolecular hydrogen bonds, of both the classical NH -donor type and the non-classical CH -donor type.

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Comment

The 1,3,4-thiadiazole ring system is known to possess several biological activities (Foroumadi *et al.*, 2002; Wang *et al.*, 2004). Aroyl ureas are reported to exhibit diverse biological effects, such as insecticidal, fungicidal, herbicidal and plant-growth regulating activities (Chen *et al.*, 2005; Wang *et al.*, 1998). Based on its combination of these potential properties, the title compound, (I), is of interest to us. It was prepared and its crystal structure is here presented.



The crystal structure (Fig. 1) reveals that the urea linkage unit, $\text{O2}-\text{C7}-\text{N4}-\text{C8}-\text{N3}-\text{H3A}$, adopts a planar conformation due to the formation of an intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond, giving a six-membered ring, as has been reported in other substituted ureas (Song *et al.*, 2006; Tan *et al.*, 2005).

In the crystal structure, the molecules are linked by intermolecular hydrogen bonds formed with CH and NH donors (Fig. 2 and Table 1). The asymmetric unit contains two molecules, which form an essentially planar hydrogen-bonded dimer; these units are all perpendicular to the b axis, so that the overall packing is a set of parallel-stacked essentially planar units.

Experimental

The title compound, (I), was prepared by the reaction of 2-amino-5-trifluoromethyl-1,3,4-thiadiazole with 4-bromobenzoyl isocyanate according to the procedure of Song *et al.* (2005). Suitable crystals were obtained by vapor diffusion of methanol into an acetone solution at room temperature (m.p. 511–512 K). Elemental analysis calculated for $\text{C}_{11}\text{H}_6\text{BrF}_3\text{N}_4\text{O}_2\text{S}$: C 33.43, H 1.53, N 14.18%; found: C 33.59, H 1.65, N 14.06%.

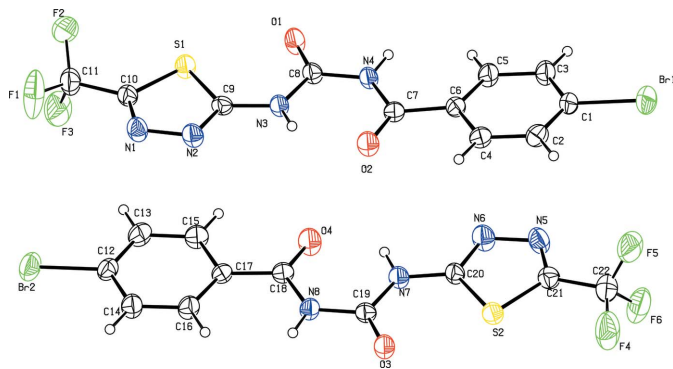


Figure 1
View of the two molecules of (I) in the asymmetric unit, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

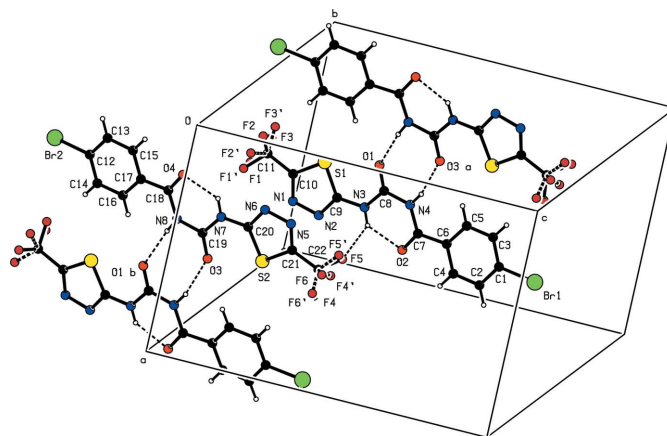


Figure 2
Partial packing diagram of (I), showing hydrogen bonds as dashed lines. [Symmetry codes: (a) $x + 1, y, z$; (b) $x - 1, y, z$; (c) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$]

Crystal data

$C_{11}H_6BrF_3N_4O_2S$
 $M_r = 395.17$
 Monoclinic, $P2_1/n$
 $a = 11.8573$ (12) Å
 $b = 13.7774$ (13) Å
 $c = 17.3795$ (17) Å
 $\beta = 99.396$ (2)°
 $V = 2801.1$ (5) Å³

$Z = 8$
 $D_x = 1.874$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 3.13$ mm⁻¹
 $T = 292$ (2) K
 Block, colorless
 $0.20 \times 0.10 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.573, T_{max} = 0.735$

23517 measured reflections
 6391 independent reflections
 3739 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.041$
 $\theta_{max} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.115$
 $S = 0.95$
 6391 reflections
 435 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0612P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.48$ e Å⁻³
 $\Delta\rho_{min} = -0.26$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
N8—H8...O1 ⁱ	0.86	2.15	2.977 (3)	162
C16—H16...O1 ⁱ	0.93	2.42	3.334 (3)	167
N7—H7...O4	0.86	1.90	2.591 (3)	136
C5—H5...O3 ⁱⁱ	0.93	2.41	3.301 (3)	161
N4—H4A...O3 ⁱⁱ	0.86	2.18	2.995 (3)	159
N3—H3A...O2	0.86	1.90	2.588 (3)	136
N3—H3A...F5 ⁱⁱⁱ	0.86	2.66	2.990 (5)	104

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

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 Å, N—

H = 0.86 Å and $U_{iso}(H) = 1.2U_{eq}(\text{carrier})$. The F atoms in the trifluoromethyl groups in each of the two molecules in the asymmetric unit are disordered and have elongated displacement ellipsoids. The occupancies of all the disordered F/F' positions were refined to 0.647 (14)/0.353 (14) for molecule 1 and to 0.740 (10)/0.260 (10) for molecule 2.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT-Plus (Bruker, 2001); data reduction: SAINT-Plus; 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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