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2-Phenyl-3-{5-[3-(trifluoromethyl)phenyl]-1,3,4-thiadiazol-2-yl}thiazolidin-4-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.040
wR factor = 0.131
Data-to-parameter ratio = 12.5

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

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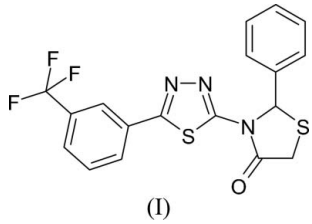
The title compound, $C_{18}H_{12}F_3N_3OS_2$, was synthesized by the reaction of [(*Z*)-1-phenylmethylidene]{5-[3-(trifluoromethyl)phenyl]-1,3,4-thiadiazol-2-yl}amine and mercaptoacetic acid. In the structure there are intramolecular C—H···S and C—H···N and intermolecular C—H···N hydrogen bonds.

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).



(I)

We are focusing our synthetic and structural studies on thiadiazole derivatives and we have published recently the structure of 3-[5-(4-fluorophenyl)-1,3,4-thiadiazol-2-yl]-2-phenylthiazolidin-4-one (Wan *et al.*, 2006). Here we report the crystal structure of a close analog, (I), in which the 4-fluorophenyl substituent is replaced by 3-trifluoromethyl. The dihedral angle between the thiadiazole and 3-(trifluoromethyl)phenyl rings is 6.4 (2)° and is larger than the angle between the thiadiazole and *p*-fluorobenzene rings [2.8 (2)°].

The molecular structure of (I) is shown in Fig. 1. The bond lengths and angles are normal (Allen *et al.*, 1987). There are intramolecular C—H···S and C—H···N and intermolecular C—H···N hydrogen bonds (Fig. 1 and Table 1). The thiazolidinone adopts a twist conformation; the dihedral angle between the C7/C8/S1 and C7/N1/C8 planes is 20.8 (15)°. The thiadiazole ring is a planar aromatic heterocycle. The phenyl substituent is approximately perpendicular to the mean plane of the thiazolidinone ring because of the lack of conjugation through the saturated sp^3 atom C7. Intermolecular C—H···N hydrogen bonds result in a three-dimensional network (Fig. 2 and Table 1).

Experimental

Benzylidene[5-[3-(trifluoromethyl)phenyl]-1,3,4-thiadiazol-2-yl]-amine (5 mmol) and mercaptoacetic acid (5 mmol) were dissolved in toluene (50 ml). The resulting water was removed by distillation over a period of 5 h. The reaction mixture was left to cool to room

temperature, filtered, and the solid was recrystallized from acetone to give pure compound (I) (m.p. 452–453 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an acetone solution.

Crystal data

$C_{18}H_{12}F_3N_3OS_2$
M_r = 407.43
Triclinic, $P\overline{1}$
a = 7.5370 (15) Å
b = 10.538 (2) Å
c = 11.380 (2) Å
α = 79.86 (3)°
β = 78.03 (3)°
γ = 84.70 (3)°
V = 868.9 (3) Å^3
Z = 2
D_x = 1.557 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 293 (2) K
Block, colorless
0.40 × 0.40 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.872, T_{max} = 0.902
3679 measured reflections
3405 independent reflections
2823 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 26.0°
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.131
S = 1.01
3405 reflections
272 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0016 (5)

Table 1
Selected geometric parameters (Å, °).

S1—C8	1.805 (2)	F3′—C18	1.326 (17)
S1—C7	1.828 (2)	O—C9	1.208 (3)
S2—C10	1.7295 (19)	N1—C9	1.378 (2)
S2—C11	1.7385 (19)	N1—C10	1.385 (3)
F1—C18	1.371 (14)	N1—C7	1.480 (2)
F2—C18	1.334 (16)	N2—C10	1.305 (2)
F3—C18	1.327 (18)	N2—N3	1.376 (2)
F2′—C18	1.323 (15)	N3—C11	1.303 (2)
F1′—C18	1.275 (12)		
C8—S1—C7	93.43 (10)	C9—C8—S1	108.04 (15)
C10—S2—C11	85.90 (9)	O—C9—N1	122.7 (2)
C9—N1—C10	122.03 (17)	O—C9—C8	126.02 (19)
C9—N1—C7	119.02 (17)	N1—C9—C8	111.29 (18)
C10—N1—C7	118.90 (15)	N2—C10—N1	119.94 (17)
C10—N2—N3	111.35 (16)	N2—C10—S2	115.44 (15)
C11—N3—N2	113.12 (16)	N1—C10—S2	124.57 (14)
N1—C7—C5	112.51 (16)	N3—C11—C12	122.10 (17)
N1—C7—S1	104.07 (13)	N3—C11—S2	114.17 (15)
C5—C7—S1	113.96 (13)	C12—C11—S2	123.67 (14)
N1—C7—H7*A*	108.7		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*B*···N2^{i}	0.97	2.62	3.573 (3)	168
C13—H13*A*···N3	0.93	2.52	2.831 (3)	100
C17—H17*A*···S2	0.93	2.81	3.192 (2)	106

Symmetry code: (i) $x+1, y, z$.

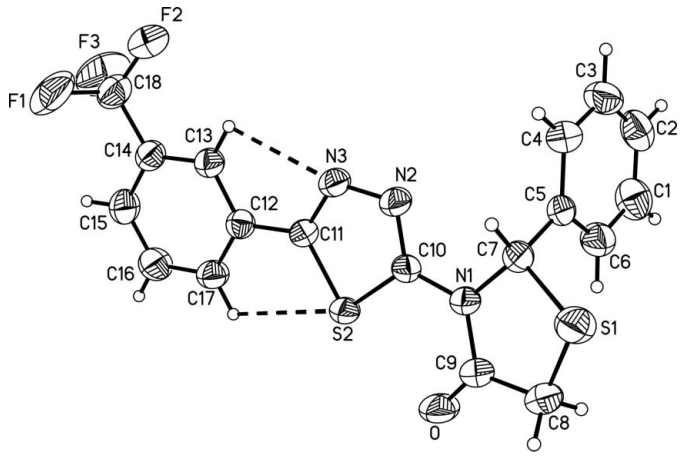


Figure 1
A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate hydrogen bonds.

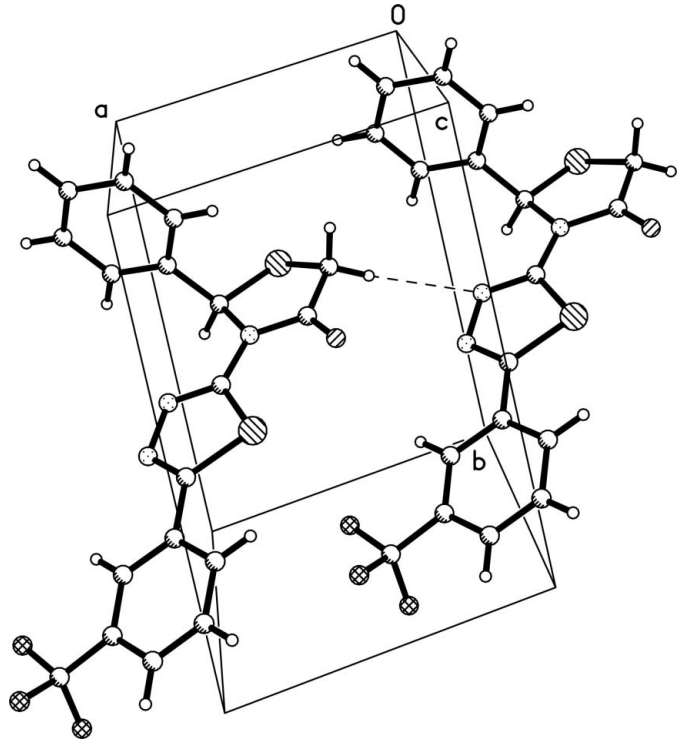


Figure 2
Part of the crystal structure of (I). The dashed line indicates an intermolecular hydrogen bond.

All H atoms were positioned geometrically, with C—H = 0.93–0.98 Å, and included in the refinement in a riding model approximation, with U_{iso}(H) = 1.2U_{eq}(C).

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: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXL97*.

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