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(*Z*)-3-[(2,4-Dinitrophenyl)hydrazono]-4,4,4-trifluoro-1-(2-thienyl)butan-1-one

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The title compound, $C_{14}H_9F_3N_4O_5S$, was prepared from 4,4,4-trifluoro-1-(2-thienyl)butane-1,3-dione and 2,4-dinitrophenylhydrazine. The crystal structure shows that strong intramolecular hydrogen bonding determines the conformation of the individual molecules, while intermolecular hydrogen bonding and aromatic π-stacking interactions stabilize the structure in the solid state.

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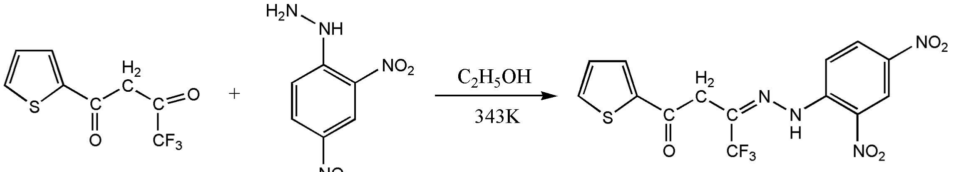
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.049
wR factor = 0.121
Data-to-parameter ratio = 13.4

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

Comment

Schiff bases have received considerable attention in the literature because of their potential pharmacological activity (Parashar *et al.*, 1988) and photochromic properties (Hadjoudis *et al.*, 1987). We report here the synthesis and molecular structure of the title compound, (I), which was prepared by an addition reaction between 4,4,4-trifluoro-1-(2-thienyl)butane-1,3-dione and 2,4-dinitrophenylhydrazine (see scheme). A perspective view of (I) is shown in Fig. 1.



(I)

The bond lengths of C3—N1 and N1—N2 are 1.276 (3) and 1.366 (3) Å, respectively, which are consistent with normal bond lengths (Jing *et al.*, 2005). The thiophene ring (C5–C8/S1) and the dinitrophenylhydrazone moiety (N2/N3/N4/C9–C14) are both planar, with r.m.s. deviations of the fitted atoms of 0.006 and 0.027 Å, respectively. The dihedral angle between the benzene and thienyl ring planes is 46.60 (8)°. Intramol-

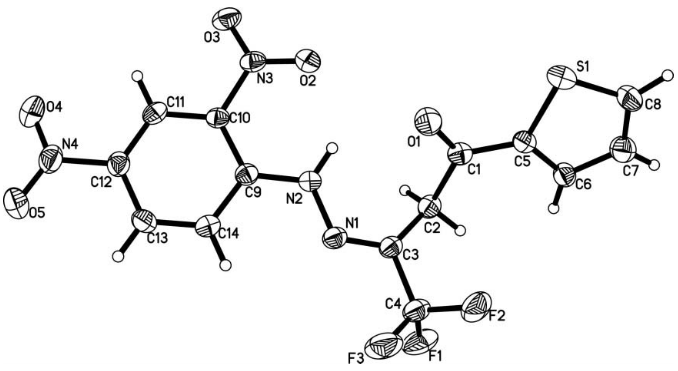


Figure 1
A view of the molecule of the title compound, shown with 30% probability displacement ellipsoids.

ecular N—H···O hydrogen bonds (Table 1) stabilize the conformation of the molecule. In addition, the molecules associate *via* intermolecular hydrogen bonds (Table 1) along the *a* and *b* axes and extend in a herring-bone pattern, as shown in Fig. 2. Further stabilization is provided by π-stacking interactions involving both the benzene and thienyl rings.

Experimental

4,4,4-Trifluoro-1-(2-thienyl)butane-1,3-dione (2.22 g, 10 mmol) was added to 2,4-dinitrophenylhydrazine (1.98 g, 10 mmol) in anhydrous ethanol. The mixture was stirred at 343 K for 6 h under nitrogen to yield a yellow precipitate of (I). The product was isolated in 86% yield and dried *in vacuo*. Bright-yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{14}H_9F_3N_4O_5S$
$M_r = 402.31$
Monoclinic, $P2_1/c$
$a = 4.8629$ (14) Å
$b = 18.290$ (5) Å
$c = 18.403$ (6) Å
$\beta = 94.642$ (6)°
$V = 1631.5$ (8) Å^3
$Z = 4$

$D_x = 1.638$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 932 reflections
$\theta = 2.5$–23.1°
$\mu = 0.27$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.24 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{min} = 0.734$, $T_{max} = 0.950$
9336 measured reflections

3324 independent reflections
1853 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 26.4°$
$h = -6 \rightarrow 4$
$k = -21 \rightarrow 22$
$l = -22 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.121$
$S = 1.01$
3324 reflections
248 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2	0.862 (10)	1.97 (2)	2.619 (3)	131 (2)
N2—H2···O1	0.862 (10)	2.205 (19)	2.883 (3)	135 (2)
C2—H2*B*···O4^{i}	0.97	2.49	3.392 (4)	154
C13—H13···O5^{ii}	0.93	2.58	3.345 (4)	140

Symmetry codes: (i) $3 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $4 - x, 1 - y, 1 - z$.

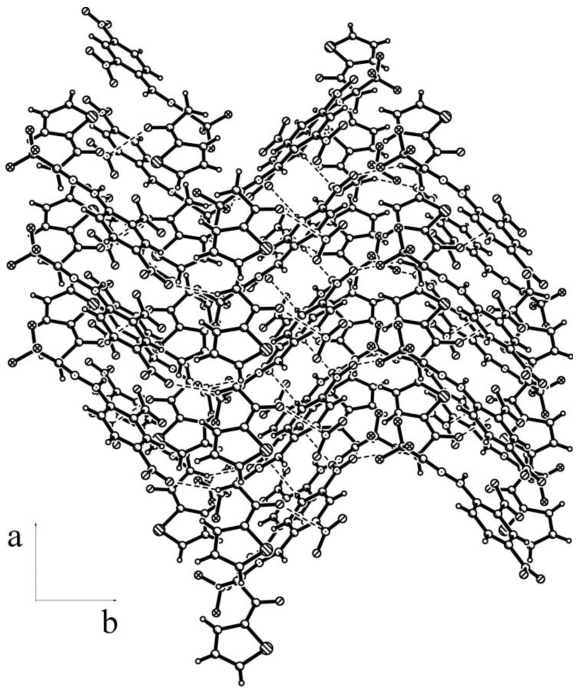


Figure 2
Intermolecular hydrogen-bonding interactions (dashed lines) in (I).

H atoms were located in a difference Fourier map and refined using a riding model [C—H = 0.93–0.97 Å; $U_{iso}(H) = 1.2U_{eq}(C)$], except for atom H2 (attached to N2), which was refined freely.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999; data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *WinGX* (Farrugia, 1999) and *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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