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4-(2-Methylprop-2-enyl)-1-[3-(trifluoromethyl)phenyl]thiosemicarbazide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
Disorder in main residue
R factor = 0.076
wR factor = 0.249
Data-to-parameter ratio = 14.9

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

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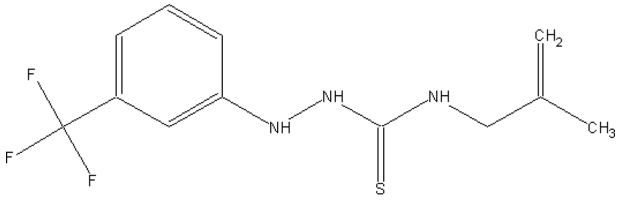
The title compound, $C_{12}H_{14}F_3N_3S$, is a biologically active anti-implantation agent. Its crystal structure is stabilized by intermolecular N—H···S hydrogen bonds, which form dimers in a head-to-tail arrangement and link them into a polymeric chain.

Comment

Efforts have been made in the past few years by many research groups to develop anti-infertility drugs because such compounds would lead to inhibition of implantation. The title compound, (I), has been found to exhibit anti-implantation activity (Nagarajan *et al.*, 1984) and these drugs have been examined in rats. The molecular structure of (I) and a packing diagram are illustrated in Figs. 1 and 2, respectively. The torsion angles C1—N1—N2—C8, N1—N2—C8—N3 and N2—C8—N3—C9 are −133.5 (4), −12.9 (5) and 168.5 (4)°, respectively. The bond lengths C1—N1, C9—N3, C8—N3 and C8—N2 are 1.411 (5), 1.454 (5), 1.328 (5) and 1.346 Å, respectively, indicating that the electronic environment around each N atom is different. Intermolecular N—H···S hydrogen bonds (Table 1) form dimers, which are further connected by other N—H···S hydrogen bonds, to give a polymeric chain (Fig. 3).



(I)

Experimental

Compound (I) was synthesized according to a procedure reported in the literature (Nagarajan *et al.*, 1984). Crystals were obtained from solutions in a mixture of methanol and carbon tetrachloride, by slow evaporation at 278 K.

Crystal data

$C_{12}H_{14}F_3N_3S$
$M_r = 289.31$
Monoclinic, $P2_1/n$
$a = 14.964$ (2) Å
$b = 6.0900$ (8) Å
$c = 15.754$ (2) Å
$\beta = 108.387$ (2)°
$V = 1362.3$ (3) Å^3
$Z = 4$

$D_x = 1.41$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 975 reflections
$\theta = 1.4$–25.2°
$\mu = 0.26$ mm^{-1}
$T = 293$ (2) K
Block, orange
0.3 × 0.3 × 0.2 mm

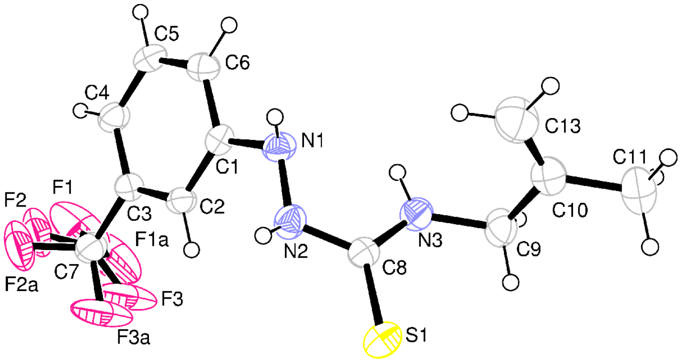


Figure 1
View of (I), showing 40% probability displacement ellipsoids and both components of the disordered trifluoromethyl group.

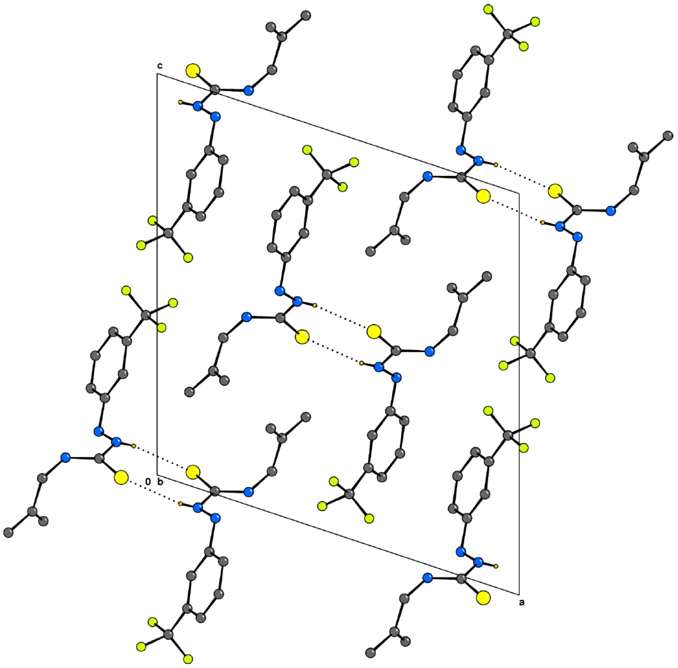


Figure 2
Packing diagram, viewed down the *b* axis. Dotted lines represent the dimer-forming N—H···S hydrogen bonds. Only the major component of the disordered trifluoromethyl group is shown. H atoms not involved in this hydrogen bond have been omitted.

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.913$, $T_{\max} = 0.941$
10483 measured reflections
2913 independent reflections
1741 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.052$
$\theta_{\max} = 27.7°$
$h = -18 \rightarrow 18$
$k = -7 \rightarrow 7$
$l = -19 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.076$
$wR(F^2) = 0.249$
$S = 0.91$
2913 reflections
195 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.2P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.84$ e Å^{-3}
$\Delta\rho_{\min} = -0.41$ e Å^{-3}

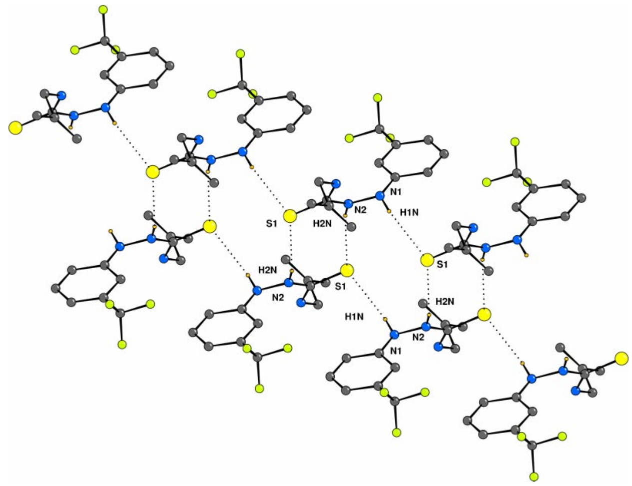


Figure 3
The polymeric chain consisting of alternate hydrogen-bonded tetramers and dimers with only the major components of the disordered trifluoromethyl groups shown.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2N···S1$^{\rm i}$	0.70 (5)	2.69 (5)	3.366 (5)	162 (5)
N1—H1N···S1$^{\rm ii}$	0.77 (5)	2.62 (5)	3.375 (4)	167 (4)

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

The trifluoromethyl group exhibits orientational disorder over two sites with occupancy factors of 0.772 (9) and 0.228 (9), respectively. The methyl H atoms were constrained to an ideal geometry [C—H = 0.96 Å and $U_{\rm iso} = 1.5U_{\rm eq}$(C)], but were allowed to rotate freely about the C—C bond. The methylene and benzene ring H atoms were placed in calculated positions. The amino H atoms were located in a difference Fourier map and refined isotropically. The N—H bond lengths are in the range 0.70 (4)–0.85 (5) Å.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2003).

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