

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

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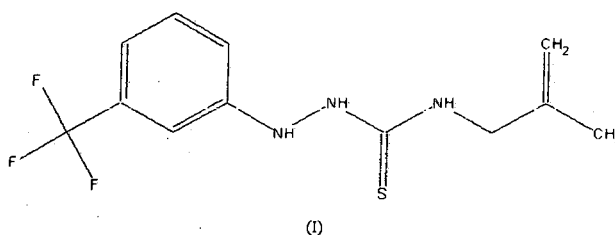
Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.006 \text{ \AA}$
Disorder in main residue
R factor = 0.076
wR factor = 0.249
Data-to-parameter ratio = 14.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>

The title compound, $\text{C}_{12}\text{H}_{14}\text{F}_3\text{N}_3\text{S}$, is a biologically active anti-implantation agent. Its crystal structure is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{S}$ 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-fertility 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 $\text{C}1-\text{N}1-\text{N}2-\text{C}8$, $\text{N}1-\text{N}2-\text{C}8-\text{N}3$ and $\text{N}2-\text{C}8-\text{N}3-\text{C}9$ are $-133.5(4)$, $-12.9(5)$ and $168.5(4)^\circ$, respectively. The bond lengths $\text{C}1-\text{N}1$, $\text{C}9-\text{N}3$, $\text{C}8-\text{N}3$ and $\text{C}8-\text{N}2$ are $1.411(5)$, $1.454(5)$, $1.328(5)$ and 1.346 \AA , respectively, indicating that the electronic environment around each N atom is different. Intermolecular $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds (Table 1) form dimers, which are further connected by other $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds, to give a polymeric chain (Fig. 3).



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

$\text{C}_{12}\text{H}_{14}\text{F}_3\text{N}_3\text{S}$
 $M_r = 289.31$
 Monoclinic, $P2_1/n$
 $a = 14.964(2) \text{ \AA}$
 $b = 6.0900(8) \text{ \AA}$
 $c = 15.754(2) \text{ \AA}$
 $\beta = 108.387(2)^\circ$
 $V = 1362.3(3) \text{ \AA}^3$
 $Z = 4$

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

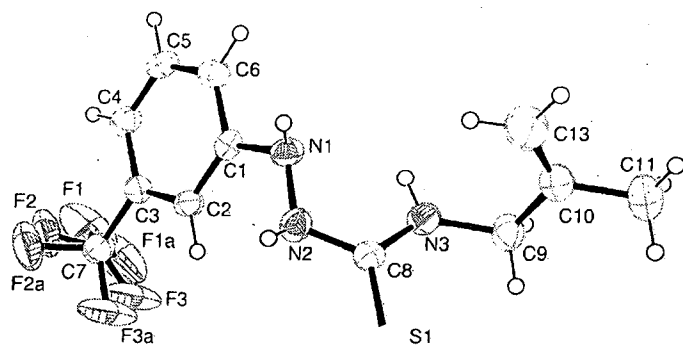


Figure 1
View of (I), showing 40% probability displacement ellipsoids and both components of the disordered trifluoromethyl group.

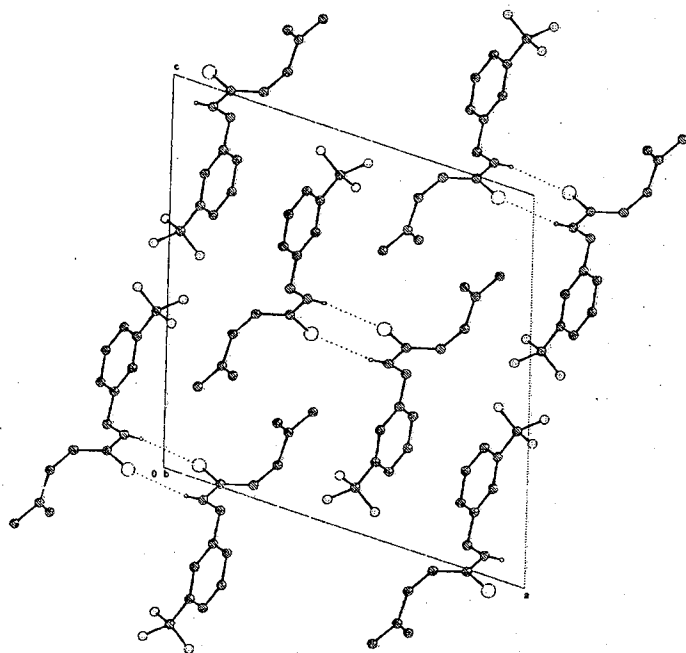


Figure 2
Packing diagram, viewed down the *b* axis. Dotted lines represent the dimer-forming N—H...S hydrogen bonds. Only the major component of the disordered trifluoromethyl group is shown. H atoms not involved in this hydrogen bond have been omitted.

Data collection

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

Refinement

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

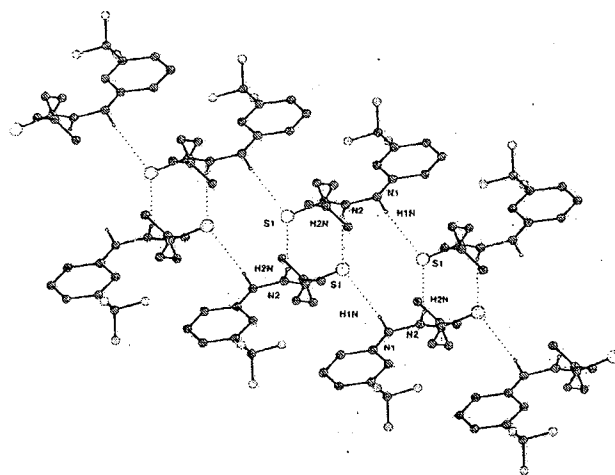


Figure 3
The polymeric chain consisting of alternate hydrogen-bonded tetramers and dimers with only the major components of the disordered trifluoromethyl groups shown.

Table 1
Hydrogen-bonding geometry (\AA , $^\circ$).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N2—H2N...S1 ⁱ	0.70 (5)	2.69 (5)	3.366 (5)	162 (5)
N1—H1N...S1 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 [$C-H = 0.96 \text{ \AA}$ and $U_{iso} = 1.5U_{eq}(C)$], 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) \AA .

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1993); program(s) used to refine structure: SHELXL97 (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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