

***N*-Phenylethyl-*N'*-[3-(trifluoromethyl)phenyl]-thiourea**

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The title compound, $C_{16}H_{15}F_3N_2S$, is a biologically active anti-implantation agent. The dihedral angle between the phenyl and trifluoromethylphenyl rings is $15.9(2)^\circ$. The crystal structure is stabilized by intermolecular $N-H \cdots S$ hydrogen bonds, forming dimers.

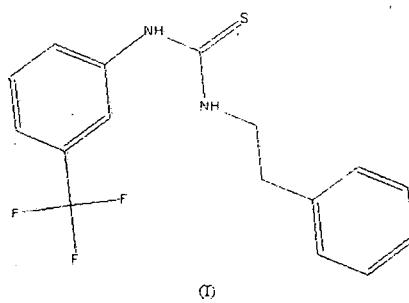
Comment

The design and synthesis of anti-fertility drugs leading to the inhibition of implantation has attracted considerable interest in the last few years. The title compound, (I), exhibits anti-implantation activity, and has been tested for uterotrophic and oestrogenic activity in rats (Nagarajan *et al.*, 1984).

Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
 Mean $\sigma(C-C) = 0.013\text{ \AA}$
 R factor = 0.089
 wR factor = 0.186
 Data-to-parameter ratio = 14.4

For details of how these key indicators were automatically derived from the article, see
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The molecular structure and a packing diagram for (I) are shown in Figs. 1 and 2, respectively. Atom C7 deviates from the plane of the attached aromatic ring by $-0.035(96)\text{ \AA}$. The torsion angles C1—N2—C8—N3, N2—C8—N3—C9, C8—N3—C9—C10 and N3—C9—C10—C11 are $6.33(66)$, $178.4(4)$, $-169.54(40)$ and $-64.64(52)^\circ$, respectively. The bond lengths C8—N2, C8—N3, C1—N2 and C9—N3 are $1.353(6)$, $1.324(6)$, $1.415(6)$ and $1.456(6)\text{ \AA}$, respectively, indicating that the electronic environments around these N atoms are different.

The crystal structure is stabilized by intermolecular $N-H \cdots S$ hydrogen bonds, forming dimers in the solid state. It is interesting that atom H3n does not participate in any hydrogen-bond formation. The approach of a neighbouring molecule is hindered because of the bulky aromatic groups which flank this N atom, thus eliminating formation of a possible hydrogen bond (Fig. 2).

Experimental

The title compound was synthesized according to the literature procedure of Nagarajan *et al.* (1984), and crystals of (I) were obtained from a solution in a mixture of dichloromethane and carbon tetrachloride, by slow evaporation at 278 K .

Crystal data

$C_{16}H_{15}F_3N_2S$
 $M_r = 324.37$
Triclinic, $P\bar{1}$
 $a = 6.978 (3) \text{ \AA}$
 $b = 7.298 (3) \text{ \AA}$
 $c = 16.064 (7) \text{ \AA}$
 $\alpha = 77.050 (6)$
 $\beta = 83.614 (6)$
 $\gamma = 86.078 (7)$
 $V = 791.5 (6) \text{ \AA}^3$

$Z = 2$
 $D_\lambda = 1.361 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 575
reflections
 $\theta = 2.4\text{--}24.7^\circ$
 $\mu = 0.23 \text{ mm}^{-1}$
 $T = 293 (2) \text{ K}$
Block, yellow
 $0.28 \times 0.20 \times 0.18 \text{ mm}$

Data collection

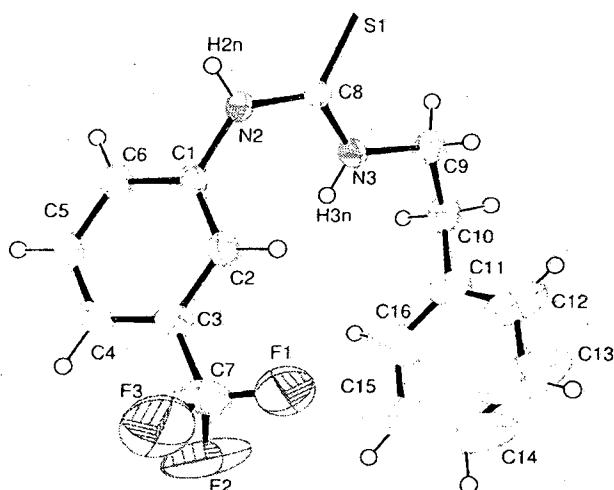
Bruker SMART APEX CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.911$, $T_{\max} = 0.959$
6240 measured reflections

2987 independent reflections
1805 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.048$
 $\theta_{\text{max}} = 26.4^\circ$
 $h = -8 \rightarrow 8$
 $k = -9 \rightarrow 9$
 $l = -19 \rightarrow 19$

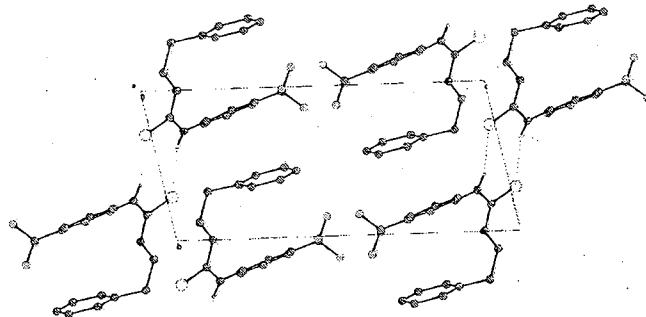
Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.089$
 $wR(F^2) = 0.186$
 $S = 1.14$
2987 reflections
207 parameters
H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_n)^2 + (0.1067P)^2 + 2.188P]$$
where $P = (F_n^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.41 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$

**Figure 1**

The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

**Figure 2**

A packing diagram for (I), viewed down the a axis. Dotted lines represent $N\text{--}H\cdots S$ hydrogen bonds.

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References

- Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
- Bruker (2000). SMART (Version 5.628) and SAINT (Version 6.02). Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Nagarajan, K., Tawarker, P. K., Kulkarni, C. L., Venkateswarlu, A., Prabhu, S. S. & Nayak, G. V. (1984). *Indian J. Chem. B*, **23**, 1243–1257.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). SHELXL97. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). CAMERON. Chemical Crystallography Laboratory, University of Oxford, England.

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

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
N2—H2n...S1 ⁱ	0.84 (6)	2.51 (6)	3.320 (5)	164 (6)

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

The amino H atoms were located in a difference Fourier map and refined isotropically. The methylene and aromatic H atoms were constrained to ride on their parent atoms, with $C\text{--}H = 0.93\text{--}0.97 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The phenyl ring exhibits probable disorder, as is indicated in the large displacement ellipsoids, but this does not in any way affect the nature of intermolecular interactions and also the molecular geometry and hence has not been dealt with.

Data collection: SMART (Bruker, 2000); cell refinement: SMART; data reduction: SAINT (Bruker, 2000); program(s) used to solve structure: S/R92 (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.*, 1996); software used to prepare material for publication: PLATON (Spek, 2003).

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