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1-[(Phenyl)(*p*-tolylimino)methyl]-3-(*p*-tolyl)thiourea

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In the title molecule, $C_{22}H_{21}N_3S$, an intramolecular N—H···N hydrogen bond appears to influence the overall conformation. In the crystal structure, pairs of molecules are linked into centrosymmetric dimers *via* N—H···S hydrogen bonds.

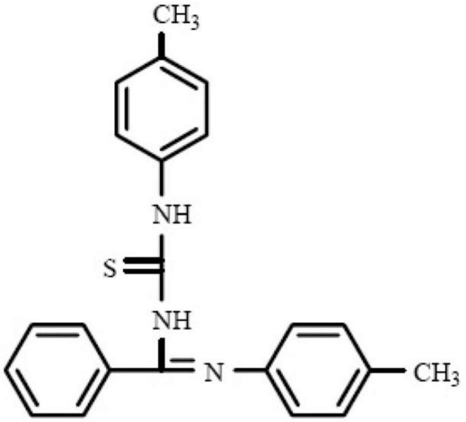
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.127
Data-to-parameter ratio = 14.5

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

Comment

Thiourea derivatives have been widely used in many fields due to their biological activity (Crank *et al.*, 1973; Landreau *et al.*, 2003). Since our group has been working in the area of the photochemistry of some heterocycles (Zhang *et al.*, 2000), we became interested in the synthesis of thiourea derivatives in order to study their characteristics with respect to their photochemistry. Here, the preparation and the crystal structure of 1-[(phenyl)(*p*-tolylimino)methyl]-3-(*p*-tolyl)thiourea (TPTMT), (I), are described.



(I)

An intramolecular hydrogen bond (Table 1 and Fig. 1) appears to influence the conformation of the molecule. The resulting six-membered ring (C7/N1/C8/N2/N3/H2) is essentially planar, with an r.m.s. deviation for the fitted atoms of 0.043 Å. The C1–C6 phenyl ring and the C9–C14 and C16–C21 benzene rings make dihedral angles of 74.1 (3), 40.0 (3) and 79.2 (2)°, respectively, with the C7/N1/C8/N2/N3/H2 ring. In the crystal structure, pairs of molecules form centrosymmetric dimers *via* intermolecular N—H···S hydrogen bonds (Table 1 and Fig. 2).

Experimental

The title compound was prepared by a slightly modified literature procedure (van der Nieuwendijk *et al.*, 2004). A solution of 1.36 g (16.8 mmol) NaSCN in 50 ml acetone was added dropwise to a solution of 3.17 g (13.8 mmol) *N*-*p*-tolylbenzimidoyl chloride in 70 ml

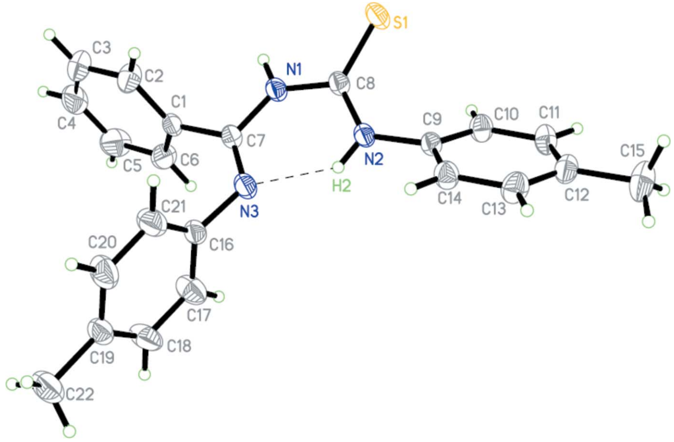


Figure 1
The molecular structure of (I), showing displacement ellipsoids drawn at the 30% probability level and H atoms drawn as small spheres. The intramolecular hydrogen bond is shown as a dashed line.

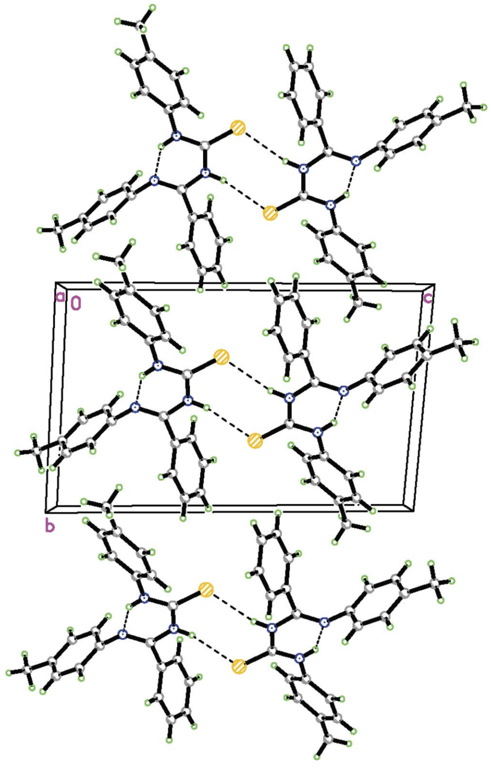


Figure 2
Partial packing plot showing three hydrogen-bonded (dashed lines) dimers.

acetone in an ethanol cooling bath at 258 K. The reaction mixture was continuously stirred in the bath until the temperature rose to 273 K. The precipitate (NaCl) was removed by filtration. The filtrate was stirred in an ice bath and a solution of 1.48 g (13.8 mmol) *p*-toluidine in 50 ml acetone was added dropwise. After warming to room temperature, the solvent was removed by evaporation and the residue was recrystallized from acetonitrile to give the pure product. Single crystals of the title compound suitable for X-ray analysis were recrystallized from acetonitrile.

Crystal data

$C_{22}H_{21}N_3S$
$M_r = 359.49$
Triclinic, $P\bar{1}$
$a = 6.0370$ (12) Å
$b = 10.372$ (2) Å
$c = 16.286$ (3) Å
$\alpha = 92.473$ (3)°
$\beta = 94.729$ (4)°
$\gamma = 106.100$ (4)°
$V = 974.1$ (3) Å^3
$Z = 2$
$D_x = 1.226$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.18$ mm^{-1}
$T = 294$ (2) K
Block, light yellow
0.20 × 0.14 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.948$, $T_{\max} = 0.983$
4945 measured reflections
3411 independent reflections
2342 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.022$
$\theta_{\max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.127$
$S = 1.03$
3411 reflections
236 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2 + 0.2903P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.24$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···S1$^{\rm i}$	0.86	2.53	3.370 (2)	167
N2—H2···N3	0.86	1.95	2.661 (3)	139

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

All H atoms were initially located in a difference Fourier map but were then constrained to an ideal geometry, with methyl C—H = 0.96 Å, aromatic C—H = 0.93 Å, N—H = 0.86 Å and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C,N) or $1.5U_{\rm eq}$(methyl C).

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

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