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1-(2-Chlorophenyl)-3-[(phenyl)(4-tolylimino)-methyl]thiourea

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Key indicators

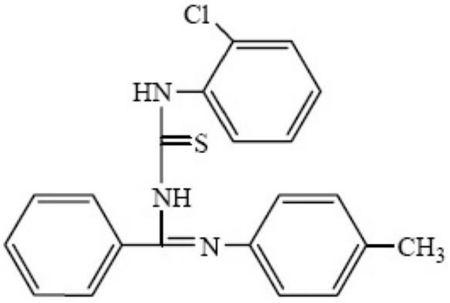
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.044
wR factor = 0.128
Data-to-parameter ratio = 12.4

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

In the crystal structure of the title compound, $C_{21}H_{18}ClN_3S$, there is an intramolecular N—H···N hydrogen bond and an intermolecular N—H···S hydrogen bond which associate the title molecules into a hydrogen-bonded dimer.

Comment

Thiourea derivatives are very important intermediates in organic syntheses, which have been widely utilized in medicinal chemistry (Smith *et al.*, 1996) and photochemistry (Dittami *et al.*, 1996; Jayanthi *et al.*, 1997). In order to study their photochemical behaviors, we have synthesized a series of new thiourea derivatives, and present here the crystal structure of one representative, *viz*. the title compound, (I).



(I)

The molecule of (I) shows a propeller-like structure (Fig. 1). The phenyl C1–C6 ring is disordered around the C1—C7 bond axis. There is an intramolecular N—H···N hydrogen bond (Table 2) which forms a six-membered ring located in the center of the molecule. Atoms N1, C7, N2, C8, N3 and H3 of the ring are coplanar, with a mean deviation of 0.015 Å. This mean plane makes dihedral angles of 129.5 (2) and 119.4 (3)° with the C9–C14 and C15–C20 benzene rings, respectively. The dihedral angle between the C9–C14 and C15–C20 rings is 58.1 (2)°. An intermolecular N—H···S hydrogen bond associates two neighbouring molecules into a molecular dimer (Fig. 2).

Experimental

The title compound was prepared by a slightly modified literature procedure (van der Nieuwendijk *et al.*, 2004). A solution of NaSCN (1.36 g, 16.8 mmol) in 50 ml acetone was added dropwise to a solution of benzimidoyl chloride (3 g, 13.8 mmol) in 70 ml 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 resulting precipitate (NaCl) was removed by filtration. The filtrate was stirred in an ice bath and a solution of 2-chlorobenzamine (1.78 g, 16.6 mmol) in 50 ml acetone was added dropwise. The solvent was removed by evaporation at room temperature and the residue was isolated by column chromatography using dichloromethane as eluent, which give the pure product (I). Single crystals suitable for X-ray analysis were obtained by crystallization from an acetonitrile solution.

Crystal data

$C_{21}H_{18}ClN_3S$
$M_r = 379.89$
Triclinic, $P\bar{1}$
$a = 7.2348$ (16) Å
$b = 11.429$ (2) Å
$c = 12.563$ (3) Å
$\alpha = 96.078$ (4)°
$\beta = 105.071$ (3)°
$\gamma = 94.283$ (4)°
$V = 991.7$ (4) Å^3
$Z = 2$
$D_x = 1.272$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.31$ mm^{-1}
$T = 294$ (2) K
Block, light yellow
0.20 × 0.16 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.941$, $T_{\max} = 0.958$
5059 measured reflections
3467 independent reflections
2463 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.016$
$\theta_{\max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.128$
$S = 1.05$
3467 reflections
279 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0606P)^2 + 0.2845P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.28$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···N1	0.77 (3)	1.97 (3)	2.630 (3)	143 (3)
N2—H2···S1^{i}	0.80 (3)	2.62 (3)	3.408 (2)	171 (3)

Symmetry code: (i) $-x + 2, -y + 1, -z + 1$.

All H atoms were initially located in a difference Fourier map. The C-bound atoms were then constrained to an ideal geometry (C—H = 0.93 or 0.96 Å), with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). The positional parameters of the amino H atoms were refined, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). The refined N—H distances are 0.77 (3) and 0.80 (3) Å. The C1–C6 phenyl ring is disordered over two positions around the C1—C7 bond axis. Least-squares refinements of the C1–C6 ring were carried out as a regular hexagon with fixed bond lengths of 1.39 Å. The occupancy factors of the disordered atoms were fixed at 0.5.

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*.

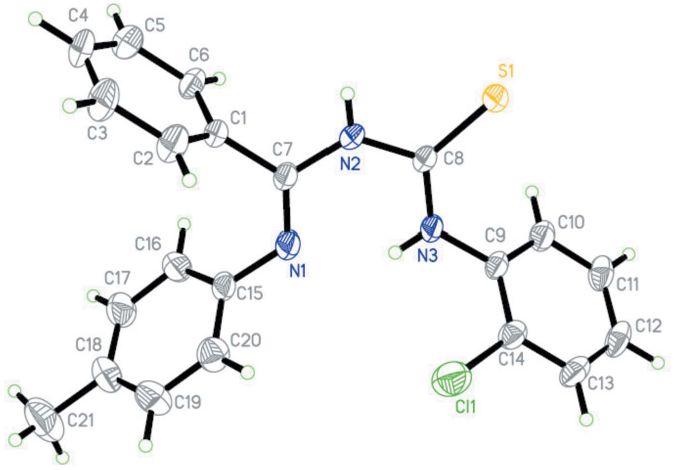


Figure 1
The structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. Only one component of the disordered phenyl group is shown.

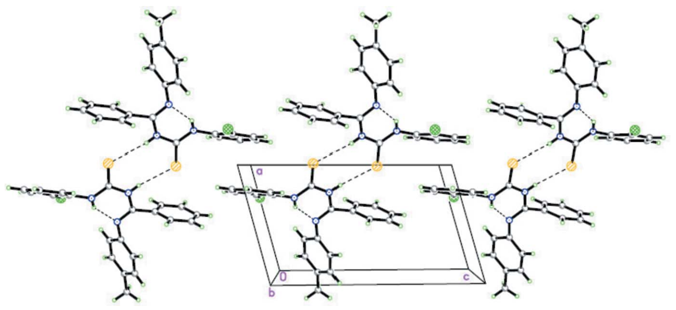


Figure 2
A view of the packing of (I) along the *b* axis, showing the inter- and intramolecular hydrogen bonds as dashed lines.

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