Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(2-Methoxyphenyl)thiourea

Mohd Mustaqim Rosli,[a] Mari Sithambaram Karthikeyan,[b] Hoong-Kun Fun,[a]* Ibrahim Abdul Razak,[a] P. S. Patil,[c] Bantwal Shivarama Holla[b] and S. M. Dharmaprakash[c]

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, [b]Department of P. G. Studies and Research in Chemistry, Mangalore University, Mangalagangotri, Mangalore 574 199, India, and [c]Department of Studies in Physics, Mangalore University, Mangalagangotri, Mangalore 574 199, India

Correspondence e-mail: hkfun@usm.my

Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.001 Å
R factor = 0.026
wR factor = 0.079
Data-to-parameter ratio = 36.4

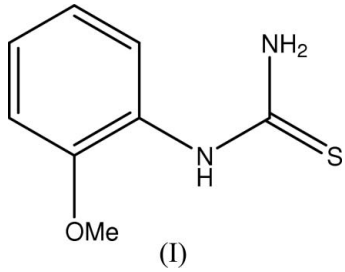
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 9 November 2006
Accepted 16 November 2006

In the title compound, $C_8H_{10}N_2OS$, the dihedral angle between the benzene ring and the thiourea group is 65.33 (2)°. Molecules are linked into infinite chains along the *a* axis by intermolecular N—H···O/S hydrogen bonds.

Comment

Phenylthiourea (PTU) has been used as a pesticide and exhibits both herbicidal and rodenticidal activity. The toxic effects of thiourea derivatives seem to arise from disturbing the carbohydrate metabolism and it has been shown that PTU causes chronic goitrogenic and other glandular problems in humans. Thiourea derivatives have also been screened as allergenic factors. Thiourea and its derivatives (including PTU) are used as corrosion inhibitors and as accelerators in the rubber industry. We present here the synthesis and crystal structure of the title thiourea compound, (I).



(I)

Bond lengths and angles in (I) show normal values (Allen *et al.*, 1987) and are comparable with those observed for *N*-(4-methoxyphenyl)thiourea (Teh *et al.*, 2006). The dihedral angle between the benzene ring and the thiourea group is 65.33 (2)°. The methoxy group attached at C1 is almost coplanar with the benzene ring (C1–C6), with a C8—O1—C1—C2 torsion angle of 5.88 (8)°. The crystal structure is stabilized by intermolecular N—H···O and N—H···S hydrogen bonds (Table 1), forming an infinite one-dimensional chain along the *a* axis (Fig. 2).

Experimental

Benzoyl chloride (11.6 ml, 0.1 mol) was added over a period of 5 min to a freshly prepared solution of ammonium isothiocyanate (11.6 g, 0.12 mol) in reagent grade acetone (150 ml) and the mixture was heated under reflux for *ca* 15 min. Heating was stopped and then 2-methoxyaniline (11.3 ml, 0.1 mol) in acetone (50 ml) was added as quickly as possible with vigorous refluxing. The mixture was then heated under reflux for 30 min and poured on to excess crushed ice with stirring. The resulting solid was collected and washed with water and then with a cold water–methanol mixture (1:1). This solid, *N*-

benzoyl-*N*-(2-methoxyphenyl)thiourea (19 g), was then added in one portion to a preheated (*ca* 353 K) solution of aqueous sodium hydroxide (50 ml, 5%) and stirred. The mixture was then poured on to an excess of ice containing aqueous hydrochloric acid (5%); the pH was adjusted to 8.0–8.5 with sodium carbonate to remove the benzoic acid. The solid product was separated, washed with water and purified by recrystallization from ethanol to give crystals of (I) (yield 80%).

Crystal data

$C_8H_{10}N_2OS$
M_r = 182.24
Monoclinic, $P2_1/c$
a = 7.3389 (1) Å
b = 13.6979 (2) Å
c = 10.5491 (1) Å
β = 126.747 (1)°
V = 849.74 (2) Å^3
Z = 4
D_x = 1.425 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 100.0 (1) K
Block, colourless
0.61 × 0.35 × 0.20 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.823, T_{max} = 0.937
35595 measured reflections
4439 independent reflections
4202 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 37.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.079
S = 1.05
4439 reflections
122 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 0.1918P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*1···S1^{i}	0.85 (2)	2.515 (17)	3.3452 (7)	166 (1)
N2—H1*N*2···O1^{ii}	0.89 (1)	2.216 (13)	3.0903 (8)	169 (1)

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

N-bound H atoms were located in difference maps and refined isotropically. The remaining H atoms were positioned geometrically and treated as riding, with C—H = 0.93–0.96 Å and U_{iso}(H) = 1.2$_{eq}$(C) or 1.5$_{eq}$(methyl C).

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

The authors thank the Malaysian Government and Universiti Sains Malaysia for the Scientific Advancement Grant Allocation (SAGA) grant No. 304/PFIZIK/653003/A118. PSP and SMD are grateful to DRDO, the Government of India, for financial assistance.

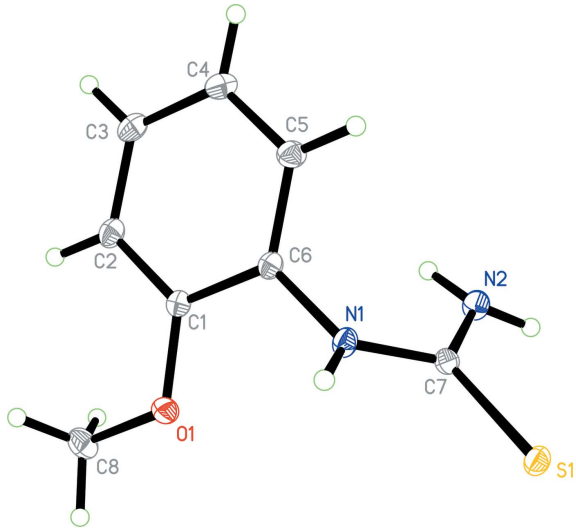


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering.

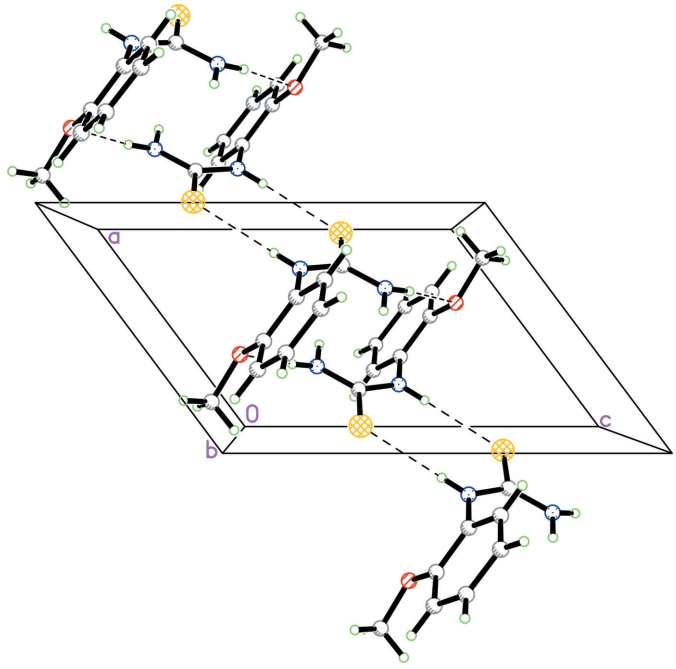


Figure 2
The packing of (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2005). *APEX2* (Version 1.27), *SAINT* (Version 7.12A) and *SADABS* (Version 2004/1). Bruker AXS Inc., Madison, Wisconsin, USA.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Teh, J. B.-J., Karthikeyan, M. S., Fun, H.-K., Patil, P. S., Razak, I. A., Holla, B. S. & Dharmaprakash, S. M. (2006). *Acta Cryst.* E**62**, o4693–o4694.