

***N*-(4-Methoxyphenyl)thiourea**

Jeannie Bee-Jan Teh,^a
 Mari Sithambaram Karthikeyan,^b
 Hoong-Kun Fun,^{a*} P. S. Patil,^c
 Ibrahim Abdul Razak,^a
 Bantwal Shivarama Holla^b and
 S. M. Dharmaprakash^c

^aX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, ^bDepartment of P. G. Studies and Research in Chemistry, Mangalore University, Mangalagangothri, Mangalore 574 199, India, and ^cDepartment of Studies in Physics, Mangalore University, Mangalagangothri, Mangalore 574 199, India

Correspondence e-mail: hkfun@usm.my

Key indicators

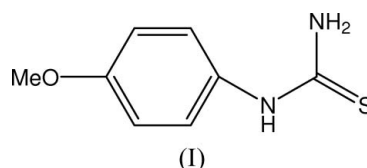
Single-crystal X-ray study
 $T = 100$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.029
 wR factor = 0.083
 Data-to-parameter ratio = 20.7

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

In the title compound, $\text{C}_8\text{H}_{10}\text{N}_2\text{OS}$, the dihedral angle between the benzene ring and the thiourea group is $59.23(4)^\circ$. Molecules are linked *via* $\text{N}-\text{H}\cdots\text{O}/\text{S}$ hydrogen bonds, forming a two-dimensional network.

Comment

The use of thiosemicarbazide in organic synthesis has become a classical strategy for the synthesis of several heterocycles. Their reactions with compounds containing $\text{C}=\text{O}$ and $\text{C}=\text{N}$ groups provide an elegant method for the preparation of biologically active compounds, *viz.* triazoles and thiazoles. For this reason, they have been the objective of several experimental and theoretical studies, aimed mainly at the determination of their crystal structures (Ali *et al.*, 2004; Cao *et al.*, 1996; Yamin & Yusof, 2003; Yusof & Yamin, 2004). We report here the synthesis and crystal structure of the title compound, (I).



The molecular structure of (I) is shown in Fig. 1. The dihedral angle between the benzene ring and the thiourea group is $59.23(4)^\circ$. The methoxy group at C6 is almost coplanar with the benzene ring (C1–C6), with a C7–O1–C6–C1 torsion angle of $4.83(14)^\circ$. The crystal structure is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{O}/\text{S}$ hydrogen bonds (Table 1), forming an infinite two-dimensional network parallel to the $(10\bar{1})$ plane (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 4-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*-(4-methoxyphenyl)-*N'*-benzoylthiourea, 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

Received 13 September 2006
 Accepted 21 September 2006

acid. The solid product was separated, washed with water and purified by recrystallization from ethanol to give crystals of (I) (yield 78%).

Crystal data

$C_8H_{10}N_2OS$
 $M_r = 182.24$
 Triclinic, $P\bar{1}$
 $a = 6.7328(2) \text{ \AA}$
 $b = 8.3813(2) \text{ \AA}$
 $c = 9.0571(2) \text{ \AA}$
 $\alpha = 63.779(1)^\circ$
 $\beta = 72.413(1)^\circ$
 $\gamma = 83.428(1)^\circ$
 $V = 436.96(2) \text{ \AA}^3$
 $Z = 2$
 $D_x = 1.385 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\mu = 0.32 \text{ mm}^{-1}$
 $T = 100.0(1) \text{ K}$
 Block, black
 $0.51 \times 0.43 \times 0.29 \text{ mm}$

Data collection

Brucker SMART APEX2 CCD diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\min} = 0.853, T_{\max} = 0.913$
 4876 measured reflections
 2531 independent reflections
 2432 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$
 $\theta_{\text{max}} = 30.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.083$
 $S = 1.11$
 2531 reflections
 122 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.1605P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.43 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.35 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1B\cdots S1^i$	0.88 (2)	2.420 (19)	3.2787 (10)	166 (2)
$N2-H2B\cdots O1^{ii}$	0.86 (2)	2.348 (19)	3.0512 (12)	139 (2)
$N2-H2C\cdots S1^{iii}$	0.88 (2)	2.511 (18)	3.3870 (10)	172 (2)

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $-x+1, -y+1, -z$; (iii) $-x+2, -y, -z+1$.

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

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/

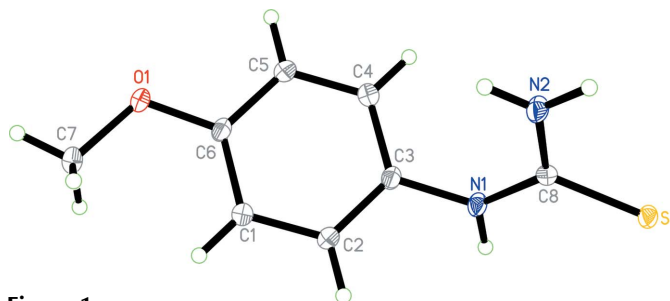


Figure 1 The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering.

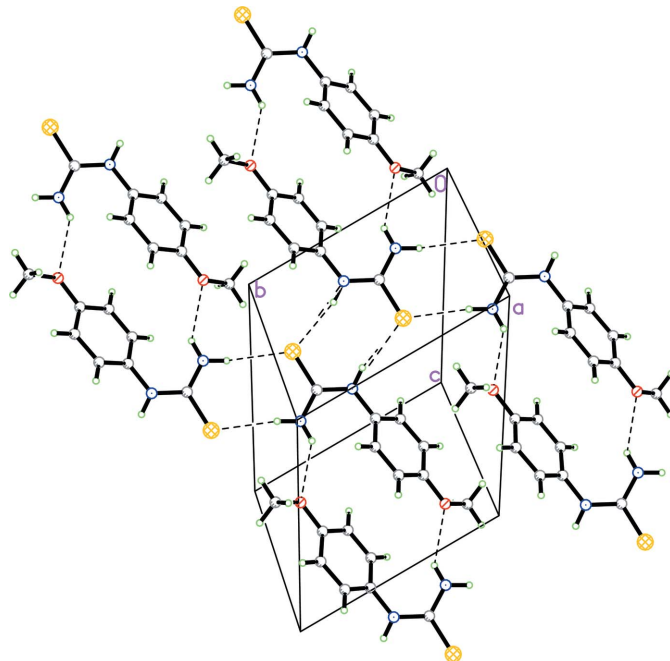


Figure 2 The crystal structure of (I). Dashed lines indicate hydrogen bonds.

A118. MSK is grateful to CSIR, New Delhi, for providing a Senior Research Fellowship.

References

Ali, H., Halim, S. N. A., Khamis, N. A., Yusof, M. S. & Yamin, B. M. (2004). *Acta Cryst.* **E60**, o1497–o1498.
 Bruker (2005). APEX2 (Version 1.27), SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
 Cao, Y., Zhao, B., Zhang, Y.-Q. & Zhang, D.-C. (1996). *Acta Cryst.* **C52**, 1772–1774.
 Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
 Sheldrick, G. M. (1998). SHELXTL. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
 Yamin, B. M. & Yusof, M. S. M. (2003). *Acta Cryst.* **E59**, o151–o152.
 Yusof, M. S. M. & Yamin, B. M. (2004). *Acta Cryst.* **E60**, o1687–o1688.