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N-(*p*-Methoxybenzoyl)-*N*′-(*o*-methoxyphenyl)thiourea

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.105
Data-to-parameter ratio = 15.7

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

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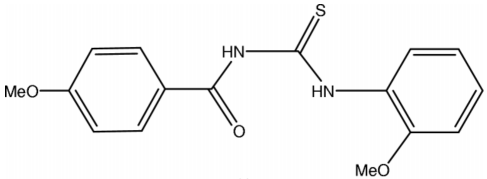
In the title compound, $C_{16}H_{16}N_2O_3S$, the *o*-methoxyphenyl and *p*-methoxybenzoyl groups are *cis* and *trans*, respectively, with respect to the S atom across the C—N bonds. The dihedral angle between the central NCSN thiourea fragment and the *o*-methoxyphenyl group is 63.66 (8)°, while that with the *p*-methoxybenzoyl group is 40.30 (7)°. The molecules are stabilized by N—H⋯S and C—H⋯O intermolecular hydrogen-bonding interactions, forming zigzag polymeric chains along the *a* axis.

Comment

Some substituted thiourea compounds have found application in the rubber and dye industries (Ramadas *et al.*, 1993), as well as exhibiting potential as biologically active compounds (Baruah *et al.*, 2002). In the title compound, (I), substituted aryl groups are attached to each of the terminal N atoms of the thiourea moiety (Fig. 1). The molecule is discrete, with the *o*-methoxyphenyl and *p*-methoxybenzoyl groups located *cis* and *trans*, respectively, to the S atom across the C8—N2 and C8—N1 bonds.



(I)

The structure (Fig. 1) and bond dimensions of (I) (Table 1) are in agreement with other substituted benzoylthioureas, such as *N*′-benzoyl-*N*-*p*-bromophenylthiourea (Yamin & Yusof, 2003*a*), *N*′-benzoyl-*N*-phenylthiourea (Yamin & Yusof, 2003*b*) and *N*-benzoyl-*N*′-(3,4-dimethylphenyl)thiourea (Shanmuga Sundara Raj *et al.*, 1999). The central thiourea [S1/N1/N2/C7/C8/C9; maximum deviation at atom N1 0.052 (1) Å] and *o*-methoxyphenyl [O3/N2/C9–C14; maximum deviation at C9 −0.033 (2) Å] fragments are each planar. The presence of a methoxy group at the *ortho* position causes the dihedral angle between the *o*-methoxyphenyl (C9–C14/O3/C16) and the central thiourea (S1/N1/N2/C7/C8/C9) fragments to increase from 7.52 (9)° in the unsubstituted *N*′-benzoyl-*N*-phenylthiourea (Yamin & Yusof, 2003*b*) to 63.66 (8)° in (I). Similarly, the dihedral angle of 40.30 (7)° between the *p*-methoxybenzoyl and the central thiourea fragments is larger than that in *N*′-benzoyl-*N*-phenylthiourea [28.78 (9)°], while the inclination between the aryl groups of 29.28 (9)° is comparable with the dihedral angle observed in *N*-phenyl-*N*′-benzoylthiourea [33.3 (1)°; Yamin & Yusof, 2003*b*].

There is one intramolecular hydrogen bond in (I), N2—H2*A*⋯O1 (Table 2), which forms the six-membered pseudoring (N1—C8—N2—H2*A*···O1—C7). In the crystal structure, the molecules are linked by intermolecular hydrogen-bonding interactions, N1—H1*A*···S1^{i} and C14—H14···O2^{ii} [symmetry codes: (i) $1 - x, 1 - y, -z$; (ii) $2 - x, 1 - y, -z$] (Table 2) to form zigzag polymeric chains along the *a* axis (Fig. 2).

Experimental

A solution of *o*-methoxyaniline (0.50 g, 4.07 mmol) in acetone (50 ml) was added dropwise to an acetone solution (50 ml) containing an equimolar quantity of *p*-methoxybenzoyl thiocyanate (0.78 g, 4.07 mmol) in a round-bottomed flask. The solution was refluxed for *ca* 1 h and then cooled in ice. The white precipitate which formed was filtered off and washed with distilled water–ethanol and then dried under vacuum. The solid was filtered and recrystallized from pyridine (m.p. 434–435 K). Analysis, found: C 60.43, H 4.80, N 8.62%; calculated for $C_{16}H_{16}N_2O_3S$: C 60.74, H 5.10, N 8.85%.

Crystal data

$C_{16}H_{16}N_2O_3S$
$M_r = 316.37$
Triclinic, $P\bar{1}$
$a = 7.832$ (1) Å
$b = 9.151$ (2) Å
$c = 12.201$ (3) Å
$\alpha = 92.68$ (4)°
$\beta = 104.56$ (4)°
$\gamma = 113.45$ (3)°
$V = 765.8$ (3) Å^3
$Z = 2$
$D_x = 1.372$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 3149 reflections
$\theta = 1.8$–26.5°
$\mu = 0.23$ mm^{-1}
$T = 273$ (2) K
Block, colourless
0.49 × 0.48 × 0.40 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.898$, $T_{max} = 0.915$
8107 measured reflections
3149 independent reflections
2927 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 26.5°$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.105$
$S = 1.06$
3149 reflections
201 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.2153P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C8	1.6681 (15)	O2—C3	1.3587 (18)
O1—C7	1.2225 (18)		
C7—N1—C8	127.74 (12)	N1—C7—C6	116.56 (12)
C8—N2—C9	126.65 (12)	N2—C8—N1	115.77 (12)
O1—C7—N1	121.87 (13)	N2—C8—S1	125.20 (11)
O1—C7—C6	121.56 (13)	N1—C8—S1	119.03 (10)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1	0.86	1.93	2.630 (2)	137
N1—H1*A*···S1^{i}	0.86	2.70	3.518 (2)	159
C14—H14···O2^{ii}	0.93	2.52	3.431 (2)	167

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

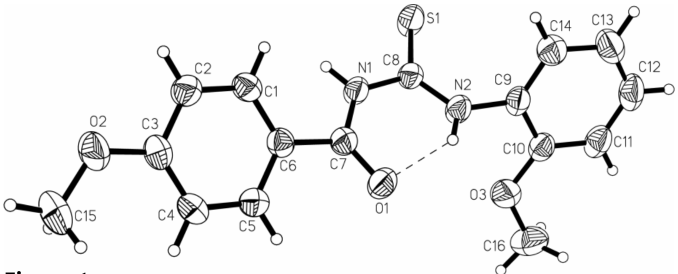


Figure 1
The molecular structure of (I), with 50% probability displacement ellipsoids. The dashed line indicates the intramolecular hydrogen bond.

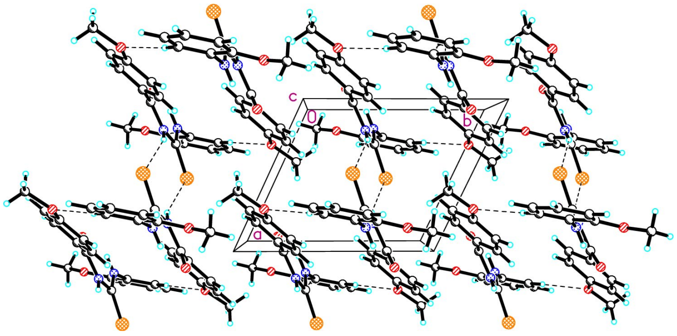


Figure 2
A packing diagram for (I), viewed along the *a* axis. Dashed lines indicate the N1—H1*A*···S1 and C14—H14···O2 hydrogen bonds.

After their location in a difference map, all H atoms were positioned geometrically and allowed to ride on their parent C atoms, with C—H = 0.93–0.96 Å and N—H = 0.86 Å, and with $U_{iso}(H) = 1.2$ or $1.5U_{eq}$(C,N).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); 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).

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References

Baruah, H. L., Reactor, C. M., Monier, S. & Bierbach U. (2002). *Biochem. Pharmacol.* **64**, 191–200.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Ramadas, K., Srinivasan, N. & Janarthanan, N. (1993). *Tetrahedron Lett.* **34**, 6447–6450.
Shanmuga Sundara Raj, S., Puviarasan, K., Velmurugan, D., Jayanthi, G. & Fun, H.-K. (1999). *Acta Cryst.* C**55**, 1318–1320.
Sheldrick, G. M. (1996). *SADABS.* University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL.* Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Yamin, M. Y. & Yusof, M. S. M. (2003*a*). *Acta Cryst.* E**59**, o340–o341.
Yamin, M. Y. & Yusof, M. S. M. (2003*b*). *Acta Cryst.* E**59**, o151–o152.

o1498 Hapipah Ali *et al.* • $C_{16}H_{16}N_2O_3S$ *Acta Cryst.* (2004). E**60**, o1497–o1498