C5	0.29828 (12)	0.25755 (12)	0.65467 (7)	0.0473 (4)
C5*a*	0.31675 (12)	0.34153 (11)	0.61553 (7)	0.0450 (4)
C6	0.41400 (13)	0.38592 (13)	0.61394 (8)	0.0564 (4)
C7	0.43416 (14)	0.46333 (14)	0.57589 (8)	0.0667 (5)
C8	0.35769 (15)	0.49890 (14)	0.53877 (8)	0.0670 (5)
C9	0.26072 (14)	0.45843 (13)	0.54055 (7)	0.0586 (5)
C9*a*	0.23793 (12)	0.38034 (11)	0.57929 (7)	0.0473 (4)
C1′	0.21374 (13)	0.09823 (12)	0.68846 (7)	0.0530 (4)
C2′	0.2944 (2)	0.01413 (13)	0.68940 (8)	0.0656 (5)
C21′	0.3033 (2)	−0.0281 (2)	0.62569 (9)	0.0859 (7)
C22′	0.3998 (2)	0.0500 (2)	0.71081 (10)	0.0874 (7)
C23′	0.2550 (2)	−0.0674 (2)	0.73148 (10)	0.1019 (8)
O1′	0.20741 (8)	0.13609 (8)	0.74953 (5)	0.0542 (3)
C3′	0.11859 (14)	0.17934 (13)	0.76603 (8)	0.0556 (4)
O3′	0.04901 (10)	0.19375 (12)	0.73132 (6)	0.0838 (4)
C1*p*	0.11671 (12)	0.20635 (12)	0.83028 (7)	0.0525 (4)
C2*p*	0.19944 (15)	0.19121 (15)	0.86803 (8)	0.0650 (5)
C3*p*	0.1938 (2)	0.2198 (2)	0.92749 (9)	0.0769 (6)
C4*p*	0.1059 (2)	0.2614 (2)	0.94931 (9)	0.0792 (6)
C5*p*	0.0224 (2)	0.2751 (2)	0.91264 (11)	0.0865 (7)
C6*p*	0.0279 (2)	0.2483 (2)	0.85303 (9)	0.0733 (6)

Table 12. *Selected geometric parameters* (Å, °) *for* (6)

C1—C9*a*	1.493 (2)	C4—C5	1.344 (2)
C1—N2	1.494 (2)	C5—C5*a*	1.447 (2)
N2—N3	1.252 (2)	C5*a*—C9*a*	1.402 (2)
N3—C4	1.413 (2)		
C9*a*—C1—N2	105.89 (13)	C4—C5—C5*a*	126.58 (14)
N3—N2—C1	117.77 (12)	C9*a*—C5*a*—C5	121.10 (14)
N2—N3—C4	123.99 (14)	C5*a*—C9*a*—C1	117.42 (14)
C5—C4—N3	123.20 (14)		
C9*a*—C1—N2—N3	78.8 (2)	C4—C5—C5*a*—C9*a*	29.6 (2)
C1—N2—N3—C4	−7.7 (2)	C5—C5*a*—C9*a*—C1	3.7 (2)
N2—N3—C4—C5	−46.1 (2)	N2—C1—C9*a*—C5*a*	−70.5 (2)
N3—C4—C5—C5*a*	13.5 (3)		

H atoms were placed in fixed calculated positions with $U_{\rm iso} = 1.5 \times U_{\rm eq}({\rm C})$ for methyl H atoms and $U_{\rm iso} = 1.2 \times U_{\rm eq}({\rm C})$ for others.

Data collection and cell refinement: *STADI*4 (Stoe & Cie, 1995*a*) for (4), otherwise *DIF*4 (Stoe & Cie, 1990*a*). Data reduction: *X-DATRED* (Stoe & Cie, 1995*b*) for (4), otherwise *REDU*4 (Stoe & Cie, 1990*b*). Program used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1992).

We thank the SERC for provision of a four-circle diffractometer and for a studentship (to MH).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1139). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Blake, A. J., Harding, M. & Sharp, J. T. (1994). *J. Chem. Soc. Perkin Trans.* 1, pp. 3149–3161.
Clegg, W. (1981). *Acta Cryst.* A**37**, 22–28.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–108.
Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.
Sheldrick, G. M. (1992). *SHELXTL/PC.* Version 4.3. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures.* Univ. of Göttingen, Germany.
Stoe & Cie (1990*a*). *DIF*4. *Diffractometer Control Program.* Version 7.09/DOS. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1990*b*). *REDU*4. *Data Reduction Program.* Version 7.03/DOS. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1995*a*). *STADI*4. *Data Collection Program for Windows.* Version 1.04. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1995*b*). *X-RED. Data Reduction Program for Windows.* Version 1.04. Stoe & Cie, Darmstadt, Germany.

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1-Cyclohexyl-3-(2-tolyl)thiourea

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Abstract

The cyclohexane ring in the title compound, $C_{14}H_{20}N_2S$, is in a chair conformation. There are N—H···S hydrogen bonds present in the structure.

Comment

Unsymmetrical thioureas are found to exhibit agrochemical properties and are commercially interesting compounds (Sarkis & Faisal, 1985; Alder, 1989). The title compound, (I), was synthesized in view of its agrochemical importance and the details of the synthesis have been published elsewhere (Ramadas, Srinivasan & Janarthanan, 1993). There is not much in the literature on the crystal structure determination of disubstituted thioureas and so this prompted us to perform an X-ray investigation on this series of compounds. Also, this forms part of our studies on the structural and insecticidal activities of this class of compounds.

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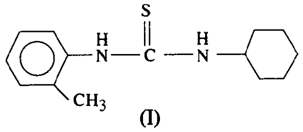

(I)

Fig. 1 shows a displacement ellipsoid plot of the molecule with the numbering scheme used. The distances N7—C8 [1.334 (2) Å] and N9—C8 [1.349 (2) Å] are shorter than their usual values, but common for thioureas (Allen *et al.*, 1987). All other bond lengths and angles in this structure are as expected. The dihedral angles between the plane of the thiourea moiety and the best plane through the phenyl ring and that through the cyclohexane ring are 102.88 (5) and 122.26 (6)°, respectively. The cyclohexane ring is in a chair conformation. Both the N atoms are involved in intermolecular N—H···S hydrogen bonds: N7—H7 0.82 (2), N7···S^{i} 3.481 (2), H7···S^{i} 2.72 (2) Å and N7—H7···S^{i} 156 (2)°; N9—H9 0.81 (2), N9···S^{i} 3.393 (1), H9···S^{i} 2.61 (2) Å and N9—H9···S^{i} 163 (2)° [symmetry code: (i) $1-x$, $y-\frac{1}{2}$, $\frac{1}{2}-z$].

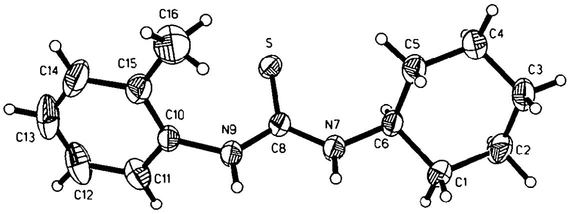

Fig. 1. Structure of (I) showing 50% probability displacement ellipsoids.

Experimental

The synthesis of compound (I) is reported elsewhere (Ramadas, Srinivasan & Janarthanan, 1993). Single crystals were obtained from an ethanol solution.

Crystal data

$C_{14}H_{20}N_2S$
$M_r = 248.38$
Monoclinic
$P2_1/c$
$a = 8.153\,(1)$ Å
$b = 8.482\,(1)$ Å
$c = 20.638\,(2)$ Å
$\beta = 92.45\,(1)°$
$V = 1425.9\,(3)$ Å^3
$Z = 4$
$D_x = 1.157$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8–25°$
$\mu = 0.209$ mm^{-1}
$T = 293\,(2)$ K
Needle
0.58 × 0.52 × 0.50 mm
Transparent, colourless

Data collection

Siemens *P*4 four-circle diffractometer
$\theta–2\theta$ scans
Absorption correction: none

$R_{\rm int} = 0.0175$
$\theta_{\rm max} = 27.50°$
$h = -1 \rightarrow 9$
$k = -1 \rightarrow 11$
$l = -26 \rightarrow 26$

4376 measured reflections
3204 independent reflections
2275 observed reflections [$I > 2\sigma(I)$]

3 standard reflections monitored every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0383$
$wR(F^2) = 0.1065$
$S = 1.064$
3204 reflections
234 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0717P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.164$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.192$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$U_{\rm eq}$
S	0.34234 (5)	0.06892 (5)	0.18215 (2)	0.05185 (15)
C1	0.2563 (3)	−0.1139 (3)	0.38544 (8)	0.0660 (5)
C2	0.1126 (3)	−0.0521 (3)	0.42328 (9)	0.0774 (6)
C3	−0.0507 (3)	−0.1066 (3)	0.39547 (10)	0.0723 (5)
C4	−0.0720 (3)	−0.0644 (3)	0.32442 (10)	0.0710 (5)
C5	0.0689 (2)	−0.1344 (3)	0.28688 (9)	0.0652 (5)
C6	0.2325 (2)	−0.0751 (2)	0.31383 (7)	0.0489 (4)
N7	0.3684 (2)	−0.1409 (2)	0.27850 (6)	0.0572 (4)
C8	0.4186 (2)	−0.0921 (2)	0.22119 (7)	0.0465 (3)
N9	0.5386 (2)	−0.1805 (2)	0.19659 (6)	0.0578 (4)
C10	0.6157 (2)	−0.1519 (2)	0.13713 (7)	0.0490 (4)
C11	0.7709 (2)	−0.0891 (2)	0.13953 (12)	0.0699 (5)
C12	0.8539 (3)	−0.0662 (3)	0.0838 (2)	0.0960 (8)
C13	0.7820 (4)	−0.1044 (3)	0.0260 (2)	0.1029 (10)
C14	0.6268 (4)	−0.1666 (3)	0.02279 (10)	0.0943 (8)
C15	0.5403 (2)	−0.1952 (2)	0.07865 (8)	0.0653 (5)
C16	0.3729 (4)	−0.2670 (5)	0.0741 (2)	0.1112 (10)

Table 2. *Selected geometric parameters* (Å, °)

S—C8	1.691 (2)	C8—N9	1.349 (2)
C1—C6	1.519 (2)	N9—C10	1.423 (2)
C1—C2	1.529 (3)	C10—C11	1.372 (3)
C2—C3	1.500 (3)	C10—C15	1.381 (2)
C3—C4	1.512 (3)	C11—C12	1.373 (3)
C4—C5	1.532 (3)	C12—C13	1.347 (4)
C5—C6	1.510 (3)	C13—C14	1.370 (4)
C6—N7	1.463 (2)	C14—C15	1.398 (3)
N7—C8	1.334 (2)	C15—C16	1.494 (3)
C6—C1—C2	110.7 (2)	C8—N9—C10	125.75 (14)
C3—C2—C1	112.7 (2)	C11—C10—C15	120.7 (2)
C2—C3—C4	111.2 (2)	C11—C10—N9	118.4 (2)
C3—C4—C5	109.9 (2)	C15—C10—N9	120.78 (15)
C6—C5—C4	110.8 (2)	C10—C11—C12	120.8 (2)
N7—C6—C5	111.42 (14)	C13—C12—C11	119.8 (2)
N7—C6—C1	109.53 (14)	C12—C13—C14	120.1 (2)
C5—C6—C1	111.1 (2)	C13—C14—C15	121.7 (2)
C8—N7—C6	126.22 (15)	C10—C15—C14	116.9 (2)
N7—C8—N9	114.59 (14)	C10—C15—C16	122.4 (2)
N7—C8—S	123.57 (12)	C14—C15—C16	120.7 (2)
N9—C8—S	121.83 (11)		

All H atoms were located from difference Fourier maps.

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93. Geometric calculations: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AS1160). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Alder, A. (1989). *Chem. Abstr.* **111**, 173788b.

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, O. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.

Fait, J. (1991). *XSCANS Users Manual.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Ramadas, K., Srinivasan, N. & Janarthanan, N. (1993). *Tetrahedron Lett.* **34**, 6447–6450.

Sarkis, G. Y. & Faisal, E. D. (1985). *J. Heterocycl. Chem.* **22**, 137–140.

Sheldrick, G. M. (1990*a*). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1990*b*). *SHELXTL/PC Users Manual.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* Univ. of Göttingen, Germany.

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2-(4-Hydroxyphenyl)benzothiazole

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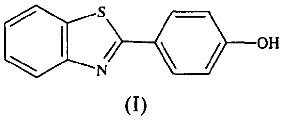
Abstract

The planar thiazole ring of the title compound, $C_{13}H_9NOS$, makes an angle of 1.39 (7)° with the benzene plane. The phenyl ring is not coplanar with the benzothiazole moiety; the dihedral angle between the thiazole and phenyl rings is 18.49 (6)°.

Comment

The thiazole ring of the title compound, (I), prepared in our laboratory, is planar and makes an angle of 1.39 (7)° with the benzene plane. The phenyl ring is twisted from the benzothiazole moiety about the C(1)—C(7) bond; the torsion angle C(6)—C(1)—C(7)—S is 19.4 (3)°.



(I)

A comparison between the title structure and those of known thiazole derivatives (Caranoni & Reboul, 1982; Prout & Miao, 1982; Bolognesi, Catellani, Destri & Porzio, 1987*a*,*b*) reveals no significant differences, either in bond lengths or in bond angles. A close intermolecular contact [2.759 (8) Å] exists between the N and $O(1-x, \frac{1}{2}+y, \frac{3}{2}-z)$ atoms.

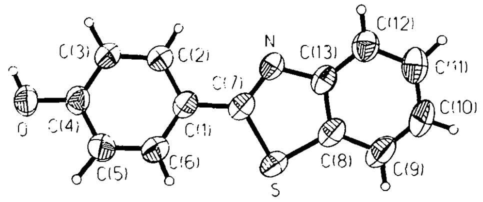


Fig. 1. View of the title compound with atomic numbering. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

Crystal data

$C_{13}H_9NOS$
$M_r = 227.3$
Orthorhombic
Pbca
$a = 13.188\ (2)$ Å
$b = 7.722\ (1)$ Å
$c = 21.178\ (3)$ Å
$V = 2156.7\ (6)$ Å^3
$Z = 8$
$D_x = 1.400$ Mg m^{-3}
$D_m = 1.391$ Mg m^{-3}
D_m measured by flotation

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 50 reflections
$\theta = 7.5–17.5°$
$\mu = 0.274$ mm^{-1}
$T = 298$ K
Plate
0.8 × 0.2 × 0.1 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$2\theta/\theta$ scans
Absorption correction: refined from ΔF (Ugozzoli, 1987)
$T_{\min} = 0.797$, $T_{\max} = 0.986$
2373 measured reflections
2373 independent reflections
1781 observed reflections [$F > 4\sigma(F)$]

$\theta_{\max} = 27.5°$
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 27$
2 standard reflections monitored every 100 reflections
intensity decay: not significant

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