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N-(3-Iodophenyl)-*N*′-(2-methylbenzoyl)-thiourea

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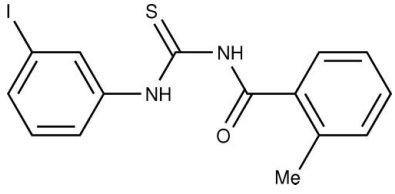
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.026; wR factor = 0.062; data-to-parameter ratio = 15.3.

The title compound, $C_{15}H_{13}IN_2OS$, adopts a *trans–cis* configuration of the 2-methylbenzoyl and 3-iodophenyl groups with respect to the thiono S atom across the thiourea C—N bonds. The dihedral angle between these two groups is 31.88 (9)°. The crystal structure is stabilized by intermolecular hydrogen bonds, forming dimers.

Related literature

For related crystal structures, see: Yusof *et al.* (2006); Razis, Yusof, Kadir & Yamin (2007); Razis, Yusof & Yamin (2007).



Experimental

Crystal data

$C_{15}H_{13}IN_2OS$
M_r = 396.23
Triclinic, $P\overline{1}$
a = 6.525 (3) Å
b = 10.249 (5) Å
c = 11.589 (5) Å
α = 78.093 (7)°
β = 87.915 (7)°
γ = 88.072 (7)°
V = 757.6 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.25 mm^{-1}
T = 298 (2) K
0.38 × 0.37 × 0.27 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.453, T_{max} = 0.547
7408 measured reflections
2800 independent reflections
2400 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.062
S = 1.04
2800 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.86	1.95	2.633 (3)	136
C11—H11···S1	0.93	2.55	3.155 (3)	123
N1—H1···S1^{i}	0.86	2.70	3.405 (3)	140

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2043).

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supplementary materials

Acta Cryst. (2007). E**63**, o4709 [**doi:10.1107/S1600536807057194**]

N-(3-Iodophenyl)-*N*'-(2-methylbenzoyl)thiourea

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Comment

The title compound, (I), adopts a *trans-cis* configuration with respect to the positions of the 2-methylbenzoyl and 3-iodophenyl groups relative to the thiono S atom, across the thiourea C—N bonds (Fig 1). The bond lengths and angles are in normal ranges and comparable to other thiourea derivatives (Yusof *et al.*, 2006; Razis, Yusof, Kadir & Yamin, 2007; Razis, Yusof & Yamin, 2007). The central thiourea (S1/N1/N2/C9), 2-methylphenyl (C1—C6/C7) and 3-iodophenyl (C10—C15/I1) groups are all planar, with a maximum deviation of 0.037 (3)Å for atom C15 from the least-squares plane. The central thiourea fragment makes dihedral angles of 48.30 (11)° and 19.82 (10)° with 2-methylphenyl and 3-iodophenyl groups, respectively. The two aryl rings are inclined to each other at an angle of 31.88 (9)°.

There are two intramolecular hydrogen bond, N2—H2···O1 and C11—H11···S1 (Table 1), forming two pseudo-six-membered rings, O1···H2—N2—C9—N1—C8—O1 and S1···H11—C11—C10—N2—C9—S1. In the crystal structure, the molecules are linked by intermolecular interaction, N—H···S (symmetry codes as in Table 1) to form dimers (Fig.2).

Experimental

To a stirring acetone solution (75 ml) of 2-methylbenzoyl chloride (2.0 g, 13 mmol) and ammoniumthiocyanate (0.98 g, 13 mmol), 3-iodoaniline (2.85 g, 13 mmol) in 40 ml of acetone was added dropwise. The solution mixture was refluxed for 1 h. The resulting solution was poured into a beaker containing some ice blocks. The white precipitate was filtered off and washed with distilled water and cold ethanol before dried under vacuum. Good quality crystals were obtained by recrystallization from DMSO.

Refinement

After their location in the difference map, all H-atoms were fixed geometrically at ideal positions and allowed to ride on the parent C or N atoms with C—H = 0.93–0.97Å and N—H = 0.86Å with U_{iso}(H)= 1.2 (CH_2 and NH) or 1.5U_{eq}(C)(CH_3).

Figures

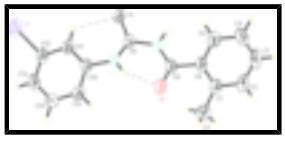

Fig. 1. : The molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate hydrogen bonds.

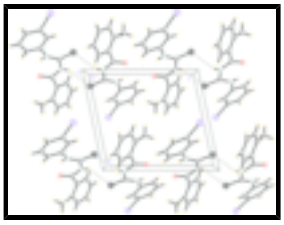

Fig. 2. : Packing diagram of compound,(I), viewed down the *a* axis. The dashed lines denote the N—H···S hydrogen bonds.

N-(3-Iodophenyl)-N'-(2-methylbenzoyl)thiourea

Crystal data

$C_{15}H_{13}IN_2OS$	$Z = 2$
$M_r = 396.23$	$F_{000} = 388$
Triclinic, $P\overline{1}$	$D_x = 1.737$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.525$ (3) Å	Cell parameters from 907 reflections
$b = 10.249$ (5) Å	$\theta = 1.8$–25.5º
$c = 11.589$ (5) Å	$\mu = 2.25$ mm^{-1}
$\alpha = 78.093$ (7)º	$T = 298$ (2) K
$\beta = 87.915$ (7)º	Block, colourless
$\gamma = 88.072$ (7)º	0.38 × 0.37 × 0.27 mm
$V = 757.6$ (6) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2800 independent reflections
Radiation source: fine-focus sealed tube	2400 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.022$
Detector resolution: 83.66 pixels mm^{-1}	$\theta_{max} = 25.5$º
$T = 298$(2) K	$\theta_{min} = 1.8$º
ω scan	$h = -7 \rightarrow 7$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$k = -12 \rightarrow 12$
$T_{min} = 0.453$, $T_{max} = 0.547$	$l = -14 \rightarrow 14$
7408 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.026$	$w = 1/[\sigma^2(F_o^2) + (0.0209P)^2 + 0.5551P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.062$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.53$ e Å^{-3}

2800 reflections

183 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

$\Delta\rho_{min} = -0.65$ e Å^{-3}

Extinction correction: SHELXL, $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0547 (17)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.19499 (4)	1.47444 (3)	0.114013 (19)	0.07956 (15)
S1	0.83545 (13)	1.16065 (8)	0.03516 (6)	0.0592 (2)
O1	1.0786 (3)	0.9391 (2)	0.38571 (16)	0.0594 (5)
N1	1.0762 (3)	1.0086 (2)	0.18631 (19)	0.0459 (5)
H1	1.1421	1.0003	0.1225	0.055*
N2	0.8230 (3)	1.1210 (2)	0.27187 (18)	0.0459 (5)
H2	0.8927	1.0910	0.3338	0.055*
C1	1.3370 (4)	0.7106 (3)	0.3381 (2)	0.0490 (6)
C2	1.5064 (5)	0.6312 (3)	0.3186 (3)	0.0612 (8)
H2A	1.5171	0.5443	0.3621	0.073*
C3	1.6583 (5)	0.6775 (4)	0.2370 (3)	0.0691 (9)
H3	1.7702	0.6223	0.2259	0.083*
C4	1.6453 (5)	0.8052 (4)	0.1719 (3)	0.0653 (8)
H4	1.7484	0.8368	0.1167	0.078*
C5	1.4802 (4)	0.8863 (3)	0.1883 (3)	0.0541 (7)
H5	1.4713	0.9727	0.1438	0.065*
C6	1.3259 (4)	0.8400 (3)	0.2712 (2)	0.0431 (6)
C7	1.1734 (5)	0.6515 (3)	0.4259 (3)	0.0661 (8)
H7A	1.1919	0.5563	0.4447	0.099*
H7B	1.0410	0.6743	0.3928	0.099*
H7C	1.1827	0.6863	0.4963	0.099*
C8	1.1509 (4)	0.9319 (3)	0.2886 (2)	0.0445 (6)
C9	0.9086 (4)	1.0979 (2)	0.1716 (2)	0.0423 (6)
C10	0.6356 (4)	1.1872 (2)	0.2924 (2)	0.0401 (5)
C11	0.5345 (4)	1.2804 (3)	0.2082 (2)	0.0475 (6)
H11	0.5914	1.3075	0.1327	0.057*
C12	0.3472 (4)	1.3323 (3)	0.2393 (2)	0.0468 (6)

C13	0.2607 (4)	1.2946 (3)	0.3506 (3)	0.0533 (7)
H13	0.1324	1.3285	0.3690	0.064*
C14	0.3669 (5)	1.2061 (3)	0.4344 (3)	0.0573 (7)
H14	0.3122	1.1821	0.5107	0.069*
C15	0.5538 (4)	1.1526 (3)	0.4060 (2)	0.0484 (6)
H15	0.6252	1.0933	0.4633	0.058*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0782 (2)	0.0951 (2)	0.06210 (17)	0.05246 (14)	−0.02494 (11)	−0.01330 (12)
S1	0.0753 (5)	0.0578 (4)	0.0386 (4)	0.0319 (4)	−0.0002 (3)	−0.0017 (3)
O1	0.0597 (12)	0.0749 (13)	0.0417 (11)	0.0334 (10)	−0.0061 (9)	−0.0127 (9)
N1	0.0398 (12)	0.0527 (13)	0.0425 (11)	0.0168 (10)	0.0022 (9)	−0.0066 (10)
N2	0.0425 (12)	0.0552 (13)	0.0381 (11)	0.0214 (10)	−0.0064 (9)	−0.0080 (10)
C1	0.0430 (15)	0.0522 (15)	0.0535 (15)	0.0105 (12)	−0.0084 (12)	−0.0156 (12)
C2	0.0553 (18)	0.0521 (17)	0.077 (2)	0.0206 (14)	−0.0121 (16)	−0.0167 (15)
C3	0.0469 (18)	0.078 (2)	0.088 (2)	0.0262 (16)	−0.0087 (17)	−0.0327 (19)
C4	0.0390 (16)	0.085 (2)	0.073 (2)	0.0083 (15)	0.0039 (14)	−0.0222 (18)
C5	0.0428 (16)	0.0591 (17)	0.0593 (17)	0.0055 (13)	−0.0032 (13)	−0.0108 (14)
C6	0.0340 (13)	0.0500 (14)	0.0467 (14)	0.0101 (11)	−0.0058 (11)	−0.0140 (12)
C7	0.063 (2)	0.0580 (18)	0.072 (2)	0.0049 (15)	0.0038 (16)	−0.0030 (16)
C8	0.0377 (14)	0.0488 (15)	0.0469 (15)	0.0107 (11)	−0.0050 (11)	−0.0112 (12)
C9	0.0414 (14)	0.0399 (13)	0.0430 (14)	0.0080 (11)	−0.0009 (11)	−0.0041 (11)
C10	0.0383 (13)	0.0415 (13)	0.0411 (13)	0.0104 (10)	−0.0047 (10)	−0.0114 (10)
C11	0.0479 (15)	0.0541 (15)	0.0381 (13)	0.0188 (12)	−0.0025 (11)	−0.0065 (11)
C12	0.0453 (15)	0.0483 (14)	0.0482 (15)	0.0175 (12)	−0.0121 (12)	−0.0145 (12)
C13	0.0418 (15)	0.0550 (16)	0.0636 (18)	0.0128 (13)	0.0035 (13)	−0.0164 (14)
C14	0.0611 (19)	0.0558 (17)	0.0504 (16)	0.0109 (14)	0.0128 (14)	−0.0048 (13)
C15	0.0550 (16)	0.0457 (14)	0.0411 (14)	0.0152 (12)	−0.0018 (12)	−0.0038 (11)

Geometric parameters (Å, °)

I1—C12	2.086 (3)	C4—H4	0.9300
S1—C9	1.660 (3)	C5—C6	1.391 (4)
O1—C8	1.220 (3)	C5—H5	0.9300
N1—C8	1.376 (3)	C6—C8	1.490 (3)
N1—C9	1.395 (3)	C7—H7A	0.9600
N1—H1	0.8600	C7—H7B	0.9600
N2—C9	1.332 (3)	C7—H7C	0.9600
N2—C10	1.415 (3)	C10—C15	1.383 (4)
N2—H2	0.8600	C10—C11	1.386 (3)
C1—C2	1.390 (4)	C11—C12	1.382 (4)
C1—C6	1.392 (4)	C11—H11	0.9300
C1—C7	1.500 (4)	C12—C13	1.374 (4)
C2—C3	1.371 (5)	C13—C14	1.374 (4)
C2—H2A	0.9300	C13—H13	0.9300
C3—C4	1.371 (5)	C14—C15	1.377 (4)
C3—H3	0.9300	C14—H14	0.9300

C4—C5	1.370 (4)	C15—H15	0.9300
C8—N1—C9	129.1 (2)	C1—C7—H7C	109.5
C8—N1—H1	115.5	H7A—C7—H7C	109.5
C9—N1—H1	115.5	H7B—C7—H7C	109.5
C9—N2—C10	130.1 (2)	O1—C8—N1	122.2 (2)
C9—N2—H2	114.9	O1—C8—C6	123.0 (2)
C10—N2—H2	114.9	N1—C8—C6	114.8 (2)
C2—C1—C6	117.6 (3)	N2—C9—N1	114.6 (2)
C2—C1—C7	118.8 (3)	N2—C9—S1	127.5 (2)
C6—C1—C7	123.6 (2)	N1—C9—S1	117.97 (19)
C3—C2—C1	121.8 (3)	C15—C10—C11	120.1 (2)
C3—C2—H2A	119.1	C15—C10—N2	115.2 (2)
C1—C2—H2A	119.1	C11—C10—N2	124.7 (2)
C4—C3—C2	120.0 (3)	C12—C11—C10	118.4 (2)
C4—C3—H3	120.0	C12—C11—H11	120.8
C2—C3—H3	120.0	C10—C11—H11	120.8
C5—C4—C3	119.9 (3)	C13—C12—C11	121.9 (2)
C5—C4—H4	120.0	C13—C12—I1	119.29 (19)
C3—C4—H4	120.0	C11—C12—I1	118.8 (2)
C4—C5—C6	120.4 (3)	C12—C13—C14	118.9 (3)
C4—C5—H5	119.8	C12—C13—H13	120.5
C6—C5—H5	119.8	C14—C13—H13	120.5
C5—C6—C1	120.4 (2)	C13—C14—C15	120.5 (3)
C5—C6—C8	118.9 (2)	C13—C14—H14	119.8
C1—C6—C8	120.7 (2)	C15—C14—H14	119.8
C1—C7—H7A	109.5	C14—C15—C10	120.1 (2)
C1—C7—H7B	109.5	C14—C15—H15	120.0
H7A—C7—H7B	109.5	C10—C15—H15	120.0
C6—C1—C2—C3	0.1 (5)	C10—N2—C9—N1	−168.7 (2)
C7—C1—C2—C3	178.0 (3)	C10—N2—C9—S1	10.0 (4)
C1—C2—C3—C4	−0.1 (5)	C8—N1—C9—N2	7.3 (4)
C2—C3—C4—C5	−0.1 (5)	C8—N1—C9—S1	−171.6 (2)
C3—C4—C5—C6	0.3 (5)	C9—N2—C10—C15	156.7 (3)
C4—C5—C6—C1	−0.3 (4)	C9—N2—C10—C11	−23.2 (4)
C4—C5—C6—C8	178.7 (3)	C15—C10—C11—C12	−3.1 (4)
C2—C1—C6—C5	0.1 (4)	N2—C10—C11—C12	176.9 (2)
C7—C1—C6—C5	−177.7 (3)	C10—C11—C12—C13	0.5 (4)
C2—C1—C6—C8	−178.8 (3)	C10—C11—C12—I1	179.3 (2)
C7—C1—C6—C8	3.4 (4)	C11—C12—C13—C14	2.2 (4)
C9—N1—C8—O1	−3.9 (5)	I1—C12—C13—C14	−176.7 (2)
C9—N1—C8—C6	176.1 (2)	C12—C13—C14—C15	−2.2 (5)
C5—C6—C8—O1	−138.0 (3)	C13—C14—C15—C10	−0.4 (5)
C1—C6—C8—O1	40.9 (4)	C11—C10—C15—C14	3.1 (4)
C5—C6—C8—N1	42.0 (3)	N2—C10—C15—C14	−176.9 (3)
C1—C6—C8—N1	−139.1 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

N2—H2···O1	0.86	1.95	2.633 (3)	136
C11—H11···S1	0.93	2.55	3.155 (3)	123
N1—H1···S1^{i}	0.86	2.70	3.405 (3)	140

Symmetry codes: (i) −*x*+2, −*y*+2, −*z*.

Fig. 1

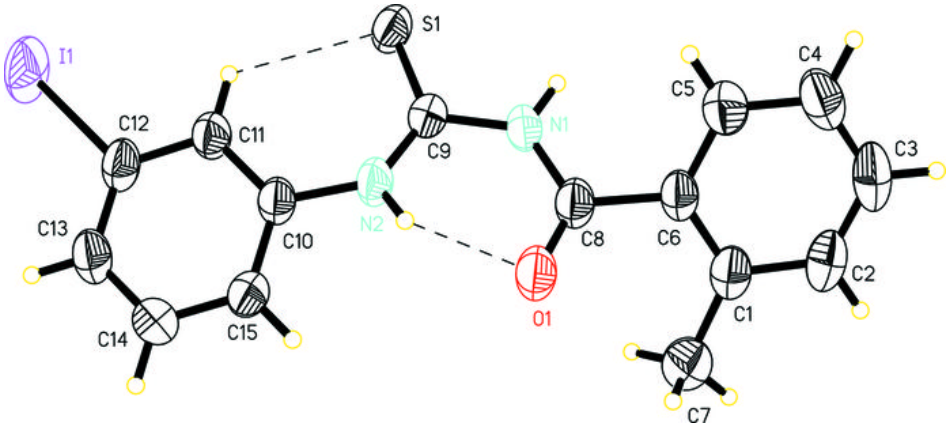

Fig. 2