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

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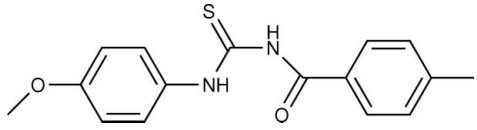
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.050; wR factor = 0.123; data-to-parameter ratio = 14.7.

In the title compound, $C_{16}H_{16}N_2O_2S$, one of the C—N bonds adopts a *transoid* configuration, whereas the other C—N bond is *cisoid* configured. The molecular conformation is stabilized by an intramolecular N—H···O hydrogen bond, and the crystal packing is stabilized by intermolecular N—H···S and C—H···O hydrogen bonds, forming chains parallel to the *a* axis.

Related literature

For related crystal structures of the title compound, see: Saeed & Flörke (2007); Yusof *et al.* (2006). For related literature, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{16}H_{16}N_2O_2S$
M_r = 300.37
Monoclinic, $P2_1/c$
a = 13.102 (3) Å
b = 6.4224 (17) Å
c = 18.292 (5) Å
β = 102.340 (4)°
V = 1503.6 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 298 (2) K
0.28 × 0.23 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.941, T_{max} = 0.974
7756 measured reflections
2794 independent reflections
2040 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.123
S = 1.03
2794 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1	0.86	1.95	2.644 (3)	138
N1—H1A···S1^{i}	0.86	2.64	3.463 (2)	160
C11—H11A···O1^{ii}	0.93	2.53	3.405 (3)	157

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

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: BT2523).

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supplementary materials

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

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Comment

The title compound is similar to 1-(3-methoxyphenyl)-3-(4-methylbenzoyl)thiourea, (Saeed & Flörke, 2007), except that the methoxy group at the *para* possition of the phenyl ring (Fig.1). The molecule maintains its *trans-cis* configuration with respect to the position of the 4-methylbenzoyl and 4-methoxyphenyl groups relative to the thiono S1 atom across their C—N bonds, respectively. The bond lengths and angles are in normal ranges (Allen *et al.*, 1987) and comparable with other thiourea derivatives (Yusof *et al.*, 2006). The central carbonylthiourea (S1/N1/N2/C7/O1/C8), 4-methylphenyl (C1—C6/C15) and 4-methoxyphenyl (C9—C14/C16/O2) groups are all planar, with a maximum deviation of 0.060 (2)Å for atom O2 from the least-squares plane of the 4-methoxyphenyl fragment. The central carbonylthiourea fragment makes dihedral angles of 29.21 (9)° and 56.21 (8)° with the 4-methylphenyl and 4-methoxyphenyl fragments, respectively. The two aryl rings are inclined to each other at an angle of 83.69 (9)°.

There is an intramolecular hydrogen bond, N2—H2A···O1 (Table 1), forming a six-membered ring (O1···H2A—N2—C8—N1—C7). In the crystal structure, the molecules are linked by intermolecular interactions, N—H···S and C—H···O (symmetry codes as in Table 1) forming chains parallel to the *a* axis (Fig. 2).

Experimental

To a stirring acetone solution (75 ml) of 4-methylbenzoyl, chloride (2.0 g, 13 mmol) and ammoniumthiocyanate (0.98 g, 13 mmol), *o*-anisidine (1.59 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 chloroform.

Refinement

After their location in a difference map, all H-atoms were fixed geometrically at ideal positions and allowed to ride on their 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

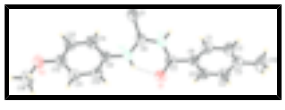

Fig. 1. Perspective view of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. The dashed line indicates a hydrogen bond.

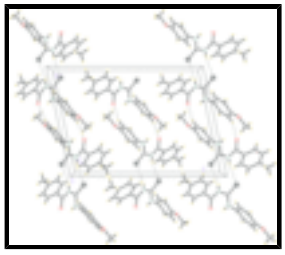

Fig. 2. Packing diagram of the title compound viewed down the *b* axis. The dashed lines denote the N—H···S and C—H···O hydrogen bonds.

N-(4-Methoxyphenyl)-N'-(4-methylbenzoyl)thiourea

Crystal data

$C_{16}H_{16}N_2O_2S$	F_{000} = 632
M_r = 300.37	D_x = 1.327 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 830 reflections
a = 13.102 (3) Å	θ = 1.5–25.5º
b = 6.4224 (17) Å	μ = 0.22 mm^{-1}
c = 18.292 (5) Å	T = 298 (2) K
β = 102.340 (4)º	Slab, colourless
V = 1503.6 (7) Å^3	0.28 × 0.23 × 0.12 mm
Z = 4	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2794 independent reflections
Radiation source: fine-focus sealed tube	2040 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.027
Detector resolution: 83.66 pixels mm^{-1}	θ_{max} = 25.5º
T = 298(2) K	θ_{min} = 1.5º
ω scans	h = −15→15
Absorption correction: multi-scan (SADABS; Bruker, 2000)	k = −7→7
T_{min} = 0.941, T_{max} = 0.974	l = −22→14
7756 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.050	H-atom parameters constrained
$wR(F^2)$ = 0.123	$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.4385P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$

2794 reflections	$\Delta\rho_{max} = 0.24$ e Å^{-3}
190 parameters	$\Delta\rho_{min} = -0.17$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}
S1	0.90844 (5)	0.64639 (12)	1.06464 (4)	0.0595 (2)
O1	0.67323 (13)	0.3771 (3)	0.86865 (10)	0.0631 (5)
N2	0.71400 (15)	0.6322 (3)	0.98468 (11)	0.0521 (5)
H2A	0.6708	0.5810	0.9469	0.063*
N1	0.83275 (14)	0.4150 (3)	0.94654 (10)	0.0457 (5)
H1A	0.8969	0.3762	0.9529	0.055*
C7	0.76414 (18)	0.3186 (4)	0.88890 (13)	0.0465 (6)
O2	0.54055 (14)	1.2210 (3)	1.14528 (11)	0.0659 (5)
C2	0.91657 (19)	−0.1598 (4)	0.85870 (13)	0.0501 (6)
H2B	0.9703	−0.2417	0.8855	0.060*
C6	0.80534 (17)	0.1382 (4)	0.85390 (12)	0.0426 (5)
C1	0.88511 (18)	0.0138 (4)	0.89214 (13)	0.0477 (6)
H1B	0.9179	0.0476	0.9410	0.057*
C8	0.81242 (18)	0.5665 (4)	0.99596 (13)	0.0453 (6)
C11	0.54404 (18)	0.8662 (4)	1.09945 (14)	0.0523 (6)
H11A	0.4877	0.8266	1.1197	0.063*
C9	0.67315 (17)	0.7806 (4)	1.02962 (13)	0.0456 (6)
C12	0.58214 (18)	1.0657 (4)	1.10923 (13)	0.0493 (6)
C3	0.8703 (2)	−0.2148 (4)	0.78647 (14)	0.0522 (6)
C14	0.7128 (2)	0.9803 (4)	1.04053 (15)	0.0569 (7)
H14A	0.7697	1.0191	1.0206	0.068*
C10	0.58969 (17)	0.7242 (4)	1.05940 (14)	0.0511 (6)
H10A	0.5635	0.5894	1.0526	0.061*
C13	0.6682 (2)	1.1218 (4)	1.08078 (15)	0.0571 (7)
H13A	0.6958	1.2553	1.0889	0.069*
C4	0.7899 (2)	−0.0895 (4)	0.74823 (14)	0.0586 (7)
H4A	0.7573	−0.1236	0.6994	0.070*
C5	0.75776 (19)	0.0834 (4)	0.78101 (14)	0.0538 (6)

H5A	0.7037	0.1647	0.7543	0.065*
C16	0.4569 (2)	1.1650 (5)	1.18001 (16)	0.0692 (8)
H16A	0.4344	1.2854	1.2034	0.104*
H16B	0.4803	1.0599	1.2171	0.104*
H16C	0.3997	1.1119	1.1429	0.104*
C15	0.9056 (3)	−0.4045 (4)	0.75038 (16)	0.0740 (9)
H15A	0.8649	−0.4184	0.7003	0.111*
H15B	0.8963	−0.5259	0.7789	0.111*
H15C	0.9781	−0.3902	0.7489	0.111*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0489 (4)	0.0750 (5)	0.0519 (4)	0.0112 (3)	0.0043 (3)	−0.0143 (3)
O1	0.0453 (10)	0.0689 (12)	0.0716 (12)	0.0082 (9)	0.0046 (9)	−0.0141 (10)
N2	0.0422 (11)	0.0587 (13)	0.0542 (12)	0.0071 (10)	0.0075 (9)	−0.0106 (10)
N1	0.0384 (10)	0.0489 (12)	0.0501 (12)	0.0053 (9)	0.0100 (9)	−0.0046 (9)
C7	0.0428 (14)	0.0485 (15)	0.0486 (14)	0.0007 (11)	0.0106 (11)	0.0032 (11)
O2	0.0657 (12)	0.0576 (11)	0.0799 (13)	0.0010 (9)	0.0282 (10)	−0.0156 (10)
C2	0.0573 (15)	0.0431 (15)	0.0508 (15)	0.0046 (11)	0.0138 (12)	0.0058 (12)
C6	0.0416 (12)	0.0457 (13)	0.0416 (13)	−0.0036 (11)	0.0114 (10)	−0.0012 (11)
C1	0.0539 (14)	0.0458 (14)	0.0431 (13)	−0.0016 (12)	0.0096 (11)	0.0006 (11)
C8	0.0463 (14)	0.0447 (13)	0.0463 (14)	0.0060 (11)	0.0131 (11)	0.0034 (11)
C11	0.0416 (13)	0.0561 (16)	0.0601 (16)	−0.0023 (12)	0.0128 (11)	−0.0063 (13)
C9	0.0408 (13)	0.0489 (14)	0.0457 (13)	0.0076 (11)	0.0065 (10)	−0.0019 (11)
C12	0.0477 (14)	0.0489 (15)	0.0501 (14)	0.0065 (11)	0.0078 (11)	−0.0053 (12)
C3	0.0679 (17)	0.0464 (14)	0.0473 (14)	−0.0030 (12)	0.0235 (13)	0.0021 (12)
C14	0.0540 (15)	0.0521 (16)	0.0707 (18)	0.0034 (13)	0.0269 (13)	0.0072 (13)
C10	0.0400 (13)	0.0488 (14)	0.0633 (16)	−0.0016 (11)	0.0080 (12)	−0.0078 (12)
C13	0.0571 (16)	0.0434 (15)	0.0734 (18)	−0.0009 (12)	0.0198 (14)	−0.0014 (13)
C4	0.0664 (17)	0.0686 (18)	0.0409 (14)	−0.0049 (14)	0.0113 (13)	−0.0101 (13)
C5	0.0491 (14)	0.0628 (17)	0.0472 (14)	0.0044 (12)	0.0049 (11)	0.0007 (12)
C16	0.0666 (18)	0.0727 (19)	0.0731 (19)	0.0172 (15)	0.0256 (15)	−0.0053 (15)
C15	0.110 (2)	0.0560 (18)	0.0621 (18)	0.0064 (16)	0.0318 (17)	−0.0060 (14)

Geometric parameters (Å, °)

S1—C8	1.658 (2)	C11—H11A	0.9300
O1—C7	1.228 (3)	C9—C10	1.370 (3)
N2—C8	1.330 (3)	C9—C14	1.382 (3)
N2—C9	1.434 (3)	C12—C13	1.387 (3)
N2—H2A	0.8600	C3—C4	1.390 (4)
N1—C7	1.377 (3)	C3—C15	1.505 (3)
N1—C8	1.392 (3)	C14—C13	1.376 (3)
N1—H1A	0.8600	C14—H14A	0.9300
C7—C6	1.480 (3)	C10—H10A	0.9300
O2—C12	1.372 (3)	C13—H13A	0.9300
O2—C16	1.425 (3)	C4—C5	1.370 (3)
C2—C1	1.376 (3)	C4—H4A	0.9300

C2—C3	1.377 (3)	C5—H5A	0.9300
C2—H2B	0.9300	C16—H16A	0.9600
C6—C1	1.381 (3)	C16—H16B	0.9600
C6—C5	1.391 (3)	C16—H16C	0.9600
C1—H1B	0.9300	C15—H15A	0.9600
C11—C12	1.373 (3)	C15—H15B	0.9600
C11—C10	1.383 (3)	C15—H15C	0.9600
C8—N2—C9	126.2 (2)	C2—C3—C4	117.7 (2)
C8—N2—H2A	116.9	C2—C3—C15	121.0 (2)
C9—N2—H2A	116.9	C4—C3—C15	121.3 (2)
C7—N1—C8	128.82 (19)	C13—C14—C9	120.0 (2)
C7—N1—H1A	115.6	C13—C14—H14A	120.0
C8—N1—H1A	115.6	C9—C14—H14A	120.0
O1—C7—N1	121.7 (2)	C9—C10—C11	120.6 (2)
O1—C7—C6	121.9 (2)	C9—C10—H10A	119.7
N1—C7—C6	116.4 (2)	C11—C10—H10A	119.7
C12—O2—C16	116.9 (2)	C14—C13—C12	120.1 (2)
C1—C2—C3	121.5 (2)	C14—C13—H13A	119.9
C1—C2—H2B	119.3	C12—C13—H13A	119.9
C3—C2—H2B	119.3	C5—C4—C3	121.3 (2)
C1—C6—C5	118.3 (2)	C5—C4—H4A	119.3
C1—C6—C7	122.7 (2)	C3—C4—H4A	119.3
C5—C6—C7	118.9 (2)	C4—C5—C6	120.5 (2)
C2—C1—C6	120.6 (2)	C4—C5—H5A	119.7
C2—C1—H1B	119.7	C6—C5—H5A	119.7
C6—C1—H1B	119.7	O2—C16—H16A	109.5
N2—C8—N1	115.8 (2)	O2—C16—H16B	109.5
N2—C8—S1	124.93 (18)	H16A—C16—H16B	109.5
N1—C8—S1	119.27 (17)	O2—C16—H16C	109.5
C12—C11—C10	119.9 (2)	H16A—C16—H16C	109.5
C12—C11—H11A	120.1	H16B—C16—H16C	109.5
C10—C11—H11A	120.1	C3—C15—H15A	109.5
C10—C9—C14	119.7 (2)	C3—C15—H15B	109.5
C10—C9—N2	118.7 (2)	H15A—C15—H15B	109.5
C14—C9—N2	121.6 (2)	C3—C15—H15C	109.5
O2—C12—C11	124.8 (2)	H15A—C15—H15C	109.5
O2—C12—C13	115.5 (2)	H15B—C15—H15C	109.5
C11—C12—C13	119.7 (2)		
C8—N1—C7—O1	8.6 (4)	C10—C11—C12—O2	177.3 (2)
C8—N1—C7—C6	−170.1 (2)	C10—C11—C12—C13	−1.9 (4)
O1—C7—C6—C1	−150.9 (2)	C1—C2—C3—C4	−0.4 (4)
N1—C7—C6—C1	27.8 (3)	C1—C2—C3—C15	179.8 (2)
O1—C7—C6—C5	25.0 (3)	C10—C9—C14—C13	−0.3 (4)
N1—C7—C6—C5	−156.3 (2)	N2—C9—C14—C13	176.7 (2)
C3—C2—C1—C6	0.2 (4)	C14—C9—C10—C11	0.8 (4)
C5—C6—C1—C2	0.1 (3)	N2—C9—C10—C11	−176.3 (2)
C7—C6—C1—C2	176.0 (2)	C12—C11—C10—C9	0.3 (4)
C9—N2—C8—N1	177.5 (2)	C9—C14—C13—C12	−1.3 (4)

C9—N2—C8—S1	−0.7 (3)	O2—C12—C13—C14	−176.9 (2)
C7—N1—C8—N2	−2.3 (3)	C11—C12—C13—C14	2.4 (4)
C7—N1—C8—S1	175.92 (18)	C2—C3—C4—C5	0.2 (4)
C8—N2—C9—C10	−124.5 (3)	C15—C3—C4—C5	−180.0 (2)
C8—N2—C9—C14	58.5 (3)	C3—C4—C5—C6	0.1 (4)
C16—O2—C12—C11	5.2 (4)	C1—C6—C5—C4	−0.3 (4)
C16—O2—C12—C13	−175.5 (2)	C7—C6—C5—C4	−176.3 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1	0.86	1.95	2.644 (3)	138
N1—H1A···S1^{i}	0.86	2.64	3.463 (2)	160
C11—H11A···O1^{ii}	0.93	2.53	3.405 (3)	157

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+2; (ii) −*x*+1, −*y*+1, −*z*+2.

Fig. 1

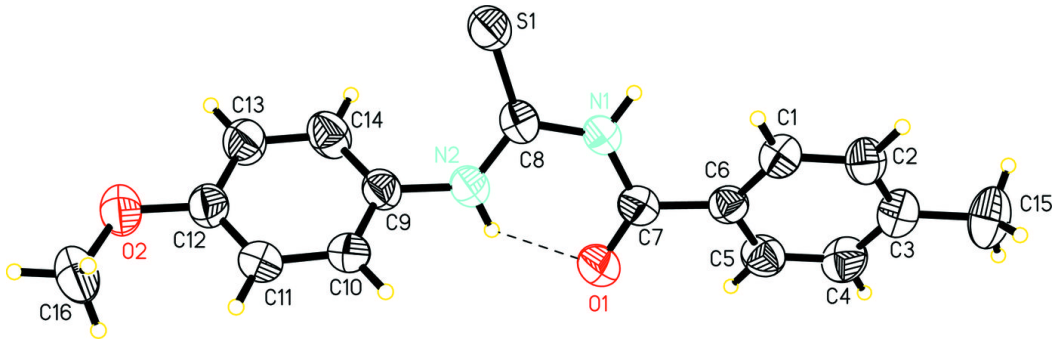

Fig. 2