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## Structure Reports

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**N-(4-Methylbenzoyl)-N'-(4-methylphenyl)thiourea**S. Aminah A. Razis,<sup>a</sup> M. Sukeri M. Yusof,<sup>a\*</sup> A. Maisara Kadir<sup>a</sup> and Bohari M. Yamin<sup>b</sup><sup>a</sup>Department of Chemical Sciences, Faculty of Science and Technology, Universiti Malaysia Terengganu, Mengabang Telipot, 21030 Kuala Terengganu, Malaysia, and<sup>b</sup>School of Chemical Sciences and Food Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

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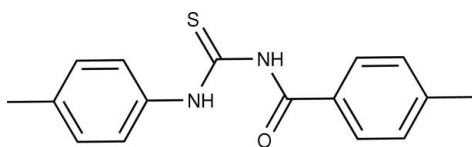
Received 1 October 2007; accepted 10 October 2007

Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.056;  $wR$  factor = 0.142; data-to-parameter ratio = 15.5.

The title compound,  $\text{C}_{16}\text{H}_{16}\text{N}_2\text{OS}$ , adopts a *trans-cis* configuration of the 4-methylbenzoyl and 4-methylphenyl groups, with respect to the thiono S atom across the thiourea C–N bonds. The dihedral angle between the two groups is  $10.36(8)^\circ$ . The structure is stabilized by intermolecular hydrogen bonds which form dimers. There are also intramolecular hydrogen bonds.

## Related literature

For related crystal structures, see: Yusof, Hamid *et al.* (2006); Yusof, Rahim & Yamin (2006).



## Experimental

## Crystal data

 $\text{C}_{16}\text{H}_{16}\text{N}_2\text{OS}$  $M_r = 284.37$ Monoclinic,  $P2_1/c$  $a = 12.191(2)$  Å $b = 6.4315(12)$  Å $c = 18.911(4)$  Å $\beta = 100.713(3)^\circ$  $V = 1456.9(5)$  Å<sup>3</sup> $Z = 4$ Mo  $K\alpha$  radiation $\mu = 0.22$  mm<sup>-1</sup> $T = 298(2)$  K $0.25 \times 0.22 \times 0.16$  mm

## Data collection

Bruker SMART APEX CCD area-detector diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 2000)

 $T_{\min} = 0.947$ ,  $T_{\max} = 0.966$ 

7857 measured reflections

2838 independent reflections

1942 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.034$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.056$  $wR(F^2) = 0.142$  $S = 1.03$ 

2838 reflections

183 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.19$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.13$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2}\cdots\text{O1}$	0.86	1.93	2.657 (3)	141
$\text{C11}-\text{H11}\cdots\text{S1}$	0.93	2.54	3.193 (3)	128
$\text{N1}-\text{H1}\cdots\text{S1}^{\text{i}}$	0.86	2.76	3.482 (2)	142

Symmetry code: (i)  $-x + 1, -y, -z + 2$ .

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); 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: BQ2038).

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**supplementary materials**

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## *N*-(4-Methylbenzoyl)-*N'*-(4-methylphenyl)thiourea

S. A. A. Razis, M. S. M. Yusof, A. M. Kadir and B. M. Yamin

### Comment

The title compound, (I), is isostructural to *N*-(4-methylbenzoyl)-*N'*-(4-nitrophenyl)thiourea (II), (Yusof, Hamid *et al.*, 2006), except that the nitro group at the phenyl ring is replaced by methyl group (Fig.1). The molecule maintains its *trans-cis* configuration with respect to the position of the 4-methylbenzoyl and 4-methylphenyl groups relative to the thiono S1 atom across their C—N bonds, respectively. The bond lengths and angles are in normal ranges and comparable to those in (II) and other thiourea derivatives (Yusof, Rahim & Yamin, 2006). The central carbonylthiourea (S1/N1/N2/C8/O1/C9) and both 4-methylphenyl groups [(C1—C6/C7) and (C10—C15/C16)] are all planar separately, with a maximum deviation of 0.039 (2)Å for atom N1 from the least-squares plane. The central carbonylthiourea fragment makes dihedral angles of 22.15 (8)° and 10.36 (8)° [24.67 (10) and 6.65 (10)° in (II)] with two rings [(C1—C6/C7) and (C10—C15/C16)], respectively. The two aryl rings are inclined to each other at an angle of 32.18 (10)° is larger compared in (II), 28.12 (12)°.

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 4-methylbenzoyl chloride (2.0 g, 13 mmol) and ammoniumthiocyanate (0.98 g, 13 mmol), *o*-toluidine (1.39 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 acetone.

### 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  $U_{\text{iso}}(\text{H}) = 1.2$  (CH<sub>2</sub> and NH) or 1.5  $U_{\text{eq}}(\text{C})(\text{CH}_3)$ .

### Figures

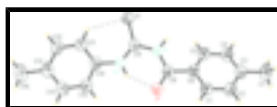


Fig. 1. : The molecule of (I) showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate hydrogen bonds.

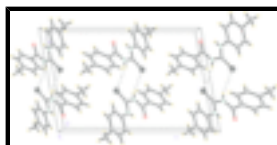


Fig. 2. : Packing diagram of compound,(I), viewed down the *b* axis. The dashed lines denote the N—H···S hydrogen bonds.

## *N*-(4-Methylbenzoyl)-*N'*-(4-methylphenyl)thiourea

### *Crystal data*

$C_{16}H_{16}N_2OS$	$F_{000} = 600$
$M_r = 284.37$	$D_x = 1.296 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
Hall symbol: -P 2ybc	$\lambda = 0.71073 \text{ \AA}$
$a = 12.191 (2) \text{ \AA}$	Cell parameters from 693 reflections
$b = 6.4315 (12) \text{ \AA}$	$\theta = 1.7\text{--}25.9^\circ$
$c = 18.911 (4) \text{ \AA}$	$\mu = 0.22 \text{ mm}^{-1}$
$\beta = 100.713 (3)^\circ$	$T = 298 (2) \text{ K}$
$V = 1456.9 (5) \text{ \AA}^3$	Block, colourless
$Z = 4$	$0.25 \times 0.22 \times 0.16 \text{ mm}$

### *Data collection*

Bruker SMART APEX CCD area-detector diffractometer	2838 independent reflections
Radiation source: fine-focus sealed tube	1942 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.034$
Detector resolution: $83.66 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 25.9^\circ$
$T = 298(2) \text{ K}$	$\theta_{\text{min}} = 1.7^\circ$
$\omega$ scans	$h = -12 \rightarrow 15$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$k = -7 \rightarrow 7$
$T_{\text{min}} = 0.947$ , $T_{\text{max}} = 0.966$	$l = -22 \rightarrow 23$
7857 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
$R[F^2 > 2\sigma(F^2)] = 0.056$	H-atom parameters constrained
$wR(F^2) = 0.142$	$w = 1/[\sigma^2(F_o^2) + (0.067P)^2 + 0.2946P]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
2838 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
183 parameters	$\Delta\rho_{\text{max}} = 0.19 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.13 \text{ e \AA}^{-3}$
	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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.59737 (6)	0.13899 (12)	1.08812 (4)	0.0657 (3)
O1	0.84014 (16)	-0.0690 (3)	0.94662 (11)	0.0680 (6)
N1	0.68101 (17)	-0.0707 (3)	0.99280 (11)	0.0490 (5)
H1	0.6181	-0.1338	0.9895	0.059*
N2	0.79159 (16)	0.1988 (3)	1.04329 (11)	0.0486 (5)
H2	0.8354	0.1463	1.0172	0.058*
C1	0.7480 (2)	-0.3785 (4)	0.84506 (15)	0.0565 (7)
H1A	0.8003	-0.2902	0.8309	0.068*
C2	0.7117 (2)	-0.5499 (4)	0.80403 (15)	0.0597 (7)
H2A	0.7399	-0.5759	0.7625	0.072*
C3	0.6340 (2)	-0.6845 (4)	0.82327 (13)	0.0495 (6)
C4	0.5938 (2)	-0.6419 (4)	0.88529 (14)	0.0534 (7)
H4	0.5412	-0.7303	0.8991	0.064*
C5	0.6303 (2)	-0.4701 (4)	0.92731 (13)	0.0501 (7)
H5	0.6027	-0.4448	0.9692	0.060*
C6	0.7078 (2)	-0.3359 (4)	0.90707 (13)	0.0448 (6)
C7	0.5932 (3)	-0.8713 (4)	0.77817 (15)	0.0727 (9)
H7A	0.5510	-0.9595	0.8042	0.109*
H7B	0.6560	-0.9470	0.7674	0.109*
H7C	0.5466	-0.8268	0.7341	0.109*
C8	0.7503 (2)	-0.1490 (4)	0.94979 (13)	0.0473 (6)
C9	0.6967 (2)	0.0950 (4)	1.04097 (13)	0.0449 (6)
C10	0.8334 (2)	0.3817 (4)	1.08134 (13)	0.0449 (6)
C11	0.7742 (2)	0.5094 (4)	1.11914 (15)	0.0584 (7)
H11	0.7009	0.4773	1.1222	0.070*
C12	0.8244 (2)	0.6855 (4)	1.15247 (15)	0.0602 (7)
H12	0.7840	0.7690	1.1786	0.072*
C13	0.9316 (2)	0.7415 (4)	1.14847 (14)	0.0544 (7)
C14	0.9888 (2)	0.6136 (4)	1.11015 (15)	0.0617 (8)
H14	1.0617	0.6476	1.1065	0.074*
C15	0.9414 (2)	0.4356 (4)	1.07668 (14)	0.0548 (7)
H15	0.9823	0.3519	1.0510	0.066*
C16	0.9857 (3)	0.9309 (5)	1.18721 (17)	0.0805 (10)

## supplementary materials

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H16A	0.9300	1.0352	1.1891	0.121*
H16B	1.0409	0.9848	1.1619	0.121*
H16C	1.0206	0.8933	1.2353	0.121*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0614 (5)	0.0779 (6)	0.0643 (5)	-0.0172 (4)	0.0283 (4)	-0.0237 (4)
O1	0.0564 (12)	0.0650 (12)	0.0889 (14)	-0.0144 (10)	0.0302 (11)	-0.0285 (11)
N1	0.0489 (13)	0.0450 (12)	0.0554 (13)	-0.0083 (10)	0.0156 (10)	-0.0085 (10)
N2	0.0452 (12)	0.0474 (12)	0.0553 (13)	-0.0022 (10)	0.0149 (10)	-0.0120 (10)
C1	0.0582 (17)	0.0533 (16)	0.0614 (17)	-0.0063 (13)	0.0202 (14)	-0.0059 (14)
C2	0.073 (2)	0.0571 (17)	0.0522 (16)	-0.0044 (15)	0.0188 (14)	-0.0098 (14)
C3	0.0616 (17)	0.0395 (14)	0.0446 (14)	0.0017 (12)	0.0025 (12)	0.0025 (11)
C4	0.0641 (18)	0.0410 (14)	0.0561 (16)	-0.0064 (13)	0.0132 (14)	0.0040 (13)
C5	0.0627 (18)	0.0434 (15)	0.0464 (14)	0.0010 (12)	0.0162 (13)	0.0008 (12)
C6	0.0471 (15)	0.0398 (13)	0.0473 (14)	0.0032 (11)	0.0085 (11)	-0.0018 (11)
C7	0.103 (3)	0.0542 (18)	0.0598 (18)	-0.0137 (16)	0.0113 (17)	-0.0091 (14)
C8	0.0473 (15)	0.0448 (14)	0.0506 (15)	-0.0025 (12)	0.0114 (12)	-0.0041 (12)
C9	0.0471 (15)	0.0428 (14)	0.0438 (14)	-0.0023 (11)	0.0060 (11)	-0.0021 (11)
C10	0.0488 (15)	0.0422 (14)	0.0422 (14)	-0.0004 (11)	0.0044 (11)	-0.0028 (11)
C11	0.0535 (17)	0.0552 (17)	0.0682 (18)	0.0002 (13)	0.0160 (14)	-0.0141 (14)
C12	0.070 (2)	0.0499 (16)	0.0617 (18)	0.0018 (14)	0.0150 (15)	-0.0135 (14)
C13	0.071 (2)	0.0419 (15)	0.0456 (15)	-0.0040 (14)	-0.0004 (13)	0.0008 (12)
C14	0.0545 (17)	0.0600 (18)	0.0694 (19)	-0.0130 (14)	0.0080 (15)	-0.0029 (15)
C15	0.0545 (17)	0.0492 (16)	0.0624 (17)	-0.0016 (13)	0.0156 (14)	-0.0079 (13)
C16	0.102 (3)	0.0566 (19)	0.077 (2)	-0.0188 (17)	0.0010 (19)	-0.0088 (16)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

S1—C9	1.656 (2)	C6—C8	1.487 (3)
O1—C8	1.222 (3)	C7—H7A	0.9600
N1—C8	1.372 (3)	C7—H7B	0.9600
N1—C9	1.392 (3)	C7—H7C	0.9600
N1—H1	0.8600	C10—C11	1.377 (3)
N2—C9	1.329 (3)	C10—C15	1.380 (3)
N2—C10	1.423 (3)	C11—C12	1.382 (4)
N2—H2	0.8600	C11—H11	0.9300
C1—C2	1.373 (4)	C12—C13	1.371 (4)
C1—C6	1.380 (3)	C12—H12	0.9300
C1—H1A	0.9300	C13—C14	1.370 (4)
C2—C3	1.381 (4)	C13—C16	1.509 (4)
C2—H2A	0.9300	C14—C15	1.382 (4)
C3—C4	1.380 (3)	C14—H14	0.9300
C3—C7	1.503 (3)	C15—H15	0.9300
C4—C5	1.385 (3)	C16—H16A	0.9600
C4—H4	0.9300	C16—H16B	0.9600
C5—C6	1.385 (3)	C16—H16C	0.9600
C5—H5	0.9300		

C8—N1—C9	130.1 (2)	O1—C8—N1	122.1 (2)
C8—N1—H1	115.0	O1—C8—C6	122.6 (2)
C9—N1—H1	115.0	N1—C8—C6	115.4 (2)
C9—N2—C10	131.3 (2)	N2—C9—N1	114.8 (2)
C9—N2—H2	114.4	N2—C9—S1	127.98 (19)
C10—N2—H2	114.4	N1—C9—S1	117.23 (18)
C2—C1—C6	120.7 (2)	C11—C10—C15	118.8 (2)
C2—C1—H1A	119.6	C11—C10—N2	125.6 (2)
C6—C1—H1A	119.6	C15—C10—N2	115.6 (2)
C1—C2—C3	121.3 (3)	C10—C11—C12	119.7 (3)
C1—C2—H2A	119.4	C10—C11—H11	120.2
C3—C2—H2A	119.4	C12—C11—H11	120.2
C4—C3—C2	118.0 (2)	C13—C12—C11	122.4 (3)
C4—C3—C7	120.6 (2)	C13—C12—H12	118.8
C2—C3—C7	121.5 (2)	C11—C12—H12	118.8
C3—C4—C5	121.3 (2)	C14—C13—C12	117.1 (3)
C3—C4—H4	119.4	C14—C13—C16	121.5 (3)
C5—C4—H4	119.4	C12—C13—C16	121.4 (3)
C4—C5—C6	120.1 (2)	C13—C14—C15	121.9 (3)
C4—C5—H5	120.0	C13—C14—H14	119.0
C6—C5—H5	120.0	C15—C14—H14	119.0
C1—C6—C5	118.7 (2)	C10—C15—C14	120.1 (2)
C1—C6—C8	118.6 (2)	C10—C15—H15	119.9
C5—C6—C8	122.7 (2)	C14—C15—H15	119.9
C3—C7—H7A	109.5	C13—C16—H16A	109.5
C3—C7—H7B	109.5	C13—C16—H16B	109.5
H7A—C7—H7B	109.5	H16A—C16—H16B	109.5
C3—C7—H7C	109.5	C13—C16—H16C	109.5
H7A—C7—H7C	109.5	H16A—C16—H16C	109.5
H7B—C7—H7C	109.5	H16B—C16—H16C	109.5
C6—C1—C2—C3	0.1 (4)	C10—N2—C9—N1	175.3 (2)
C1—C2—C3—C4	-0.1 (4)	C10—N2—C9—S1	-4.9 (4)
C1—C2—C3—C7	179.5 (3)	C8—N1—C9—N2	3.3 (4)
C2—C3—C4—C5	-0.2 (4)	C8—N1—C9—S1	-176.5 (2)
C7—C3—C4—C5	-179.8 (2)	C9—N2—C10—C11	-8.7 (4)
C3—C4—C5—C6	0.6 (4)	C9—N2—C10—C15	173.8 (2)
C2—C1—C6—C5	0.3 (4)	C15—C10—C11—C12	-1.2 (4)
C2—C1—C6—C8	179.7 (2)	N2—C10—C11—C12	-178.6 (2)
C4—C5—C6—C1	-0.6 (4)	C10—C11—C12—C13	1.2 (4)
C4—C5—C6—C8	-180.0 (2)	C11—C12—C13—C14	-0.6 (4)
C9—N1—C8—O1	-3.3 (4)	C11—C12—C13—C16	-178.1 (3)
C9—N1—C8—C6	177.2 (2)	C12—C13—C14—C15	0.1 (4)
C1—C6—C8—O1	-23.2 (4)	C16—C13—C14—C15	177.6 (3)
C5—C6—C8—O1	156.2 (3)	C11—C10—C15—C14	0.7 (4)
C1—C6—C8—N1	156.3 (2)	N2—C10—C15—C14	178.3 (2)
C5—C6—C8—N1	-24.3 (3)	C13—C14—C15—C10	-0.1 (4)

## supplementary materials

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### Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N2—H2···O1	0.86	1.93	2.657 (3)	141
C11—H11···S1	0.93	2.54	3.193 (3)	128
N1—H1···S1 <sup>i</sup>	0.86	2.76	3.482 (2)	142

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



Fig. 1

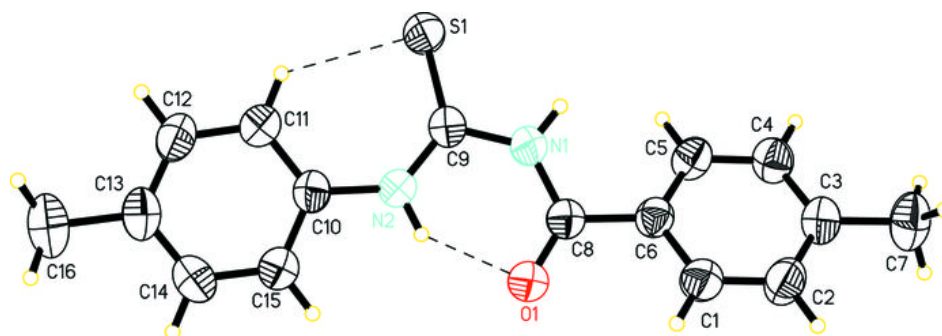


Fig. 2

