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## Structure Reports

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## N-(2,2-Dimethylpropanoyl)-N'-(2-methoxyphenyl)thiourea

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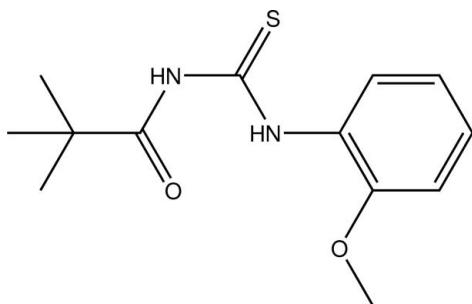
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Key indicators: single-crystal X-ray study;  $T = 273$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.068;  $wR$  factor = 0.147; data-to-parameter ratio = 17.0.

In the title compound,  $\text{C}_{13}\text{H}_{18}\text{N}_2\text{O}_2\text{S}$ , the carbonylthiourea fragment is nearly planar with an r.m.s. deviation of 0.0096 Å. The dihedral angle between carbonylthiourea group and the benzene ring is 19.16 (16)°. There are two intramolecular N—H···O hydrogen bonds, which lead to two pseudo-six-membered rings. Weak intramolecular C—H···S hydrogen bonding also occurs.

## Related literature

For related structures, see: Saeed & Flörke (2007); Yusof *et al.* (2008); Shoukat *et al.* (2007). For standard bond lengths, see: Allen *et al.* (1987).



## Experimental

## Crystal data

 $\text{C}_{13}\text{H}_{18}\text{N}_2\text{O}_2\text{S}$  $M_r = 266.35$ Orthorhombic,  $P2_12_12_1$  $a = 5.9181$  (10) Å $b = 13.492$  (2) Å $c = 17.592$  (3) Å $V = 1404.7$  (4) Å<sup>3</sup> $Z = 4$ Mo  $K\alpha$  radiation $\mu = 0.23$  mm<sup>-1</sup> $T = 273$  K $0.50 \times 0.14 \times 0.09$  mm

## Data collection

Bruker SMART APEX CCD area-detector diffractometer

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

 $T_{\min} = 0.895$ ,  $T_{\max} = 0.980$ 

8711 measured reflections

2763 independent reflections

2227 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.045$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.068$  $wR(F^2) = 0.147$  $S = 1.13$ 

2763 reflections

163 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.32$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.18$  e Å<sup>-3</sup>

Absolute structure: Flack (1983),

1141

Flack parameter: 0.63 (15)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2A}\cdots\text{O1}$	0.86	1.89	2.619 (4)	142
$\text{N2}-\text{H2A}\cdots\text{O2}$	0.86	2.17	2.575 (4)	109
$\text{C12}-\text{H12A}\cdots\text{S1}$	0.93	2.62	3.235 (4)	124

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PARST (Nardelli, 1995).

The authors wish to thank both Universiti Malaysia Terengganu and Universiti Kebangsaan Malaysia for the research facilities and the Ministry of Higher Education Malaysia for the research grant FRGS 59178.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2346).

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## supplementary materials

*Acta Cryst.* (2012). E68, o1129 [doi:10.1107/S1600536812010914]

***N*-(2,2-Dimethylpropanoyl)-*N'*-(2-methoxyphenyl)thiourea**

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**Comment**

The title compound is analogous to the previously reported, 1-(2-Nitrophenyl)-3-pivaloylthiourea (Saeed and Flörke, 2007) except that the nitro group is replaced with methoxy group (Fig. 1). The bond lengths are in normal ranges (Allen *et al.*, 1987) and comparable with other similar molecule reported (Yusof *et al.* 2008; Shoukat *et al.* 2007).

The carbonylthiourea (S1/N1/N2/O1/C4–C7) and phenyl fragments are essentially planar, with rms deviations of 0.0096 Å and 0.0064 Å, respectively. These two fragments inclined at each other at an angle of 19.16(0.16)°. There are two intramolecular hydrogen bonds, N2—H2A···O1 and N2—H2A···O2 leading to two pseudo-six membered rings. Weak C—H···S intramolecular H-bonding is also present. There is no intermolecular hydrogen bond in the crystal structure.

**Experimental**

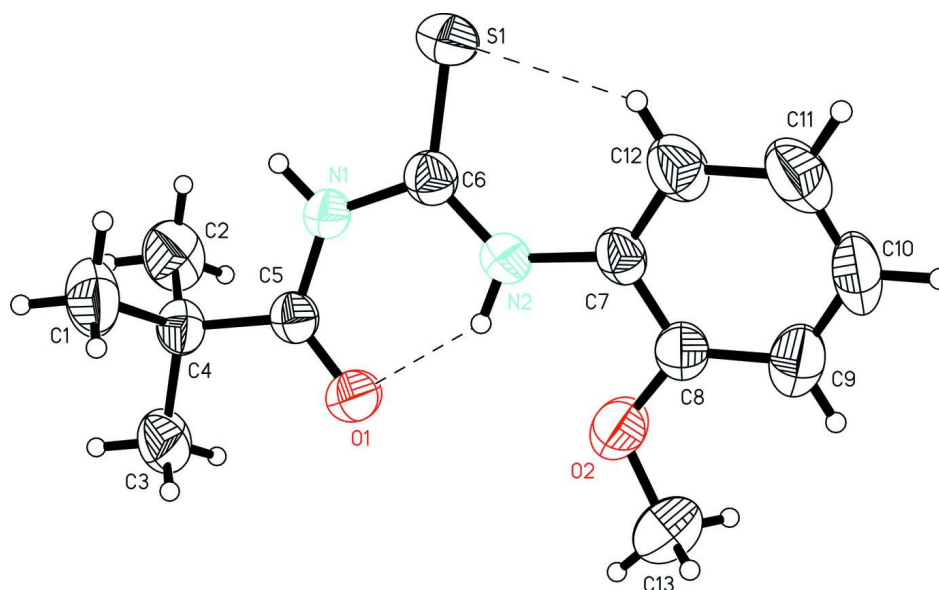
To a stirring acetone solution (75 ml) of pivaloyl chloride (5.0 g, 0.04 mol) and ammonium thiocyanate (3.15 g, 0.04 mol), 2-methoxyaniline (0.49 g, 0.04 mol) in 40 ml of acetone was added dropwise. The solution mixture was refluxed for 1 h. The resulting solution was poured into a beaker containing some ice blocks. The white precipitate was filtered off and washed with distilled water and cold ethanol before being dried under vacuum. Good quality crystals were obtained by recrystallization from DMF.

**Refinement**

H atoms on C were positioned geometrically with C—H 0.93, 0.96 Å, for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms with  $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$  where  $x=1.5$  for methyl H and  $x=1.2$  for aromatic H atoms. The H atom attached to oxygen atoms were located from the Fourier difference map and refined isotropically.

**Computing details**

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *PARST* (Nardelli, 1995).


**Figure 1**

The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level. Dashed lines show H-bondings.

### *N*-(2,2-Dimethylpropanoyl)-*N'*-(2-methoxyphenyl)thiourea

#### Crystal data

$C_{13}H_{18}N_2O_2S$

$M_r = 266.35$

Orthorhombic,  $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 5.9181$  (10) Å

$b = 13.492$  (2) Å

$c = 17.592$  (3) Å

$V = 1404.7$  (4) Å<sup>3</sup>

$Z = 4$

$F(000) = 568$

$D_x = 1.259$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 770 reflections

$\theta = 1.9$ – $26.0^\circ$

$\mu = 0.23$  mm<sup>-1</sup>

$T = 273$  K

Slab, colourless

$0.50 \times 0.14 \times 0.09$  mm

#### Data collection

Bruker SMART APEX CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 83.66 pixels mm<sup>-1</sup>

$\omega$  scan

Absorption correction: multi-scan

(*SADABS*; Bruker, 2000)

$T_{\min} = 0.895$ ,  $T_{\max} = 0.980$

8711 measured reflections

2763 independent reflections

2227 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$

$\theta_{\max} = 26.0^\circ$ ,  $\theta_{\min} = 1.9^\circ$

$h = -7 \rightarrow 6$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 21$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.068$

$wR(F^2) = 0.147$

$S = 1.13$

2763 reflections

163 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from  
neighbouring sites  
H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0655P)^2 + 0.2181P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.32 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$   
Absolute structure: Flack (1983), 1141 Friedel  
pairs  
Flack parameter: 0.63 (15)

*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  $F^2 > \sigma(F^2)$  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	0.1312 (2)	0.79180 (7)	0.57763 (7)	0.0693 (4)
O1	0.3640 (5)	0.47260 (17)	0.56305 (13)	0.0559 (7)
O2	0.7537 (5)	0.5571 (2)	0.67865 (15)	0.0662 (8)
C6	0.2391 (6)	0.6781 (2)	0.58348 (19)	0.0442 (8)
N2	0.3935 (5)	0.6446 (2)	0.63138 (15)	0.0453 (7)
H2A	0.4306	0.5836	0.6243	0.054*
N1	0.1602 (5)	0.60835 (18)	0.53095 (15)	0.0427 (7)
H1A	0.0573	0.6290	0.5003	0.051*
C5	0.2250 (6)	0.5112 (2)	0.52178 (18)	0.0384 (8)
C8	0.7025 (7)	0.6429 (3)	0.71748 (19)	0.0474 (9)
C9	0.8272 (8)	0.6805 (3)	0.7766 (2)	0.0623 (11)
H9A	0.9588	0.6489	0.7924	0.075*
C4	0.1096 (6)	0.4544 (2)	0.45732 (18)	0.0445 (8)
C7	0.5083 (7)	0.6910 (2)	0.69201 (17)	0.0434 (9)
C3	0.2134 (8)	0.3508 (3)	0.4531 (2)	0.0682 (12)
H3A	0.1901	0.3171	0.5005	0.102*
H3B	0.3724	0.3562	0.4432	0.102*
H3C	0.1427	0.3140	0.4128	0.102*
C2	0.1488 (9)	0.5089 (3)	0.3823 (2)	0.0726 (13)
H2B	0.0831	0.5738	0.3850	0.109*
H2C	0.0798	0.4724	0.3416	0.109*
H2D	0.3082	0.5146	0.3731	0.109*
C12	0.4381 (8)	0.7757 (3)	0.7293 (2)	0.0599 (12)
H12A	0.3061	0.8075	0.7142	0.072*
C11	0.5629 (10)	0.8133 (3)	0.7888 (2)	0.0731 (15)
H11A	0.5165	0.8711	0.8129	0.088*
C10	0.7544 (9)	0.7660 (3)	0.8125 (2)	0.0714 (14)
H10A	0.8365	0.7915	0.8532	0.086*
C13	0.9241 (8)	0.4949 (3)	0.7089 (2)	0.0744 (13)
H13A	0.9422	0.4379	0.6769	0.112*
H13B	0.8817	0.4740	0.7591	0.112*

H13C	1.0642	0.5307	0.7113	0.112*
C1	-0.1417 (7)	0.4454 (3)	0.4742 (3)	0.0734 (12)
H1B	-0.2072	0.5103	0.4777	0.110*
H1C	-0.1626	0.4110	0.5215	0.110*
H1D	-0.2141	0.4090	0.4341	0.110*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0848 (8)	0.0424 (5)	0.0806 (7)	0.0145 (6)	-0.0263 (7)	-0.0102 (5)
O1	0.0650 (17)	0.0465 (13)	0.0562 (14)	0.0105 (13)	-0.0175 (14)	-0.0061 (11)
O2	0.068 (2)	0.0701 (18)	0.0609 (15)	0.0127 (16)	-0.0192 (15)	-0.0080 (14)
C6	0.045 (2)	0.0429 (18)	0.0448 (18)	-0.0036 (16)	0.0009 (18)	0.0016 (15)
N2	0.0535 (19)	0.0360 (14)	0.0465 (16)	0.0025 (14)	-0.0089 (15)	-0.0053 (12)
N1	0.0445 (17)	0.0396 (15)	0.0440 (15)	0.0033 (13)	-0.0109 (14)	-0.0057 (12)
C5	0.0366 (19)	0.0403 (17)	0.0385 (17)	-0.0044 (15)	0.0033 (15)	0.0015 (14)
C8	0.054 (2)	0.048 (2)	0.0405 (18)	-0.0111 (18)	-0.0028 (17)	0.0069 (16)
C9	0.061 (3)	0.074 (3)	0.051 (2)	-0.020 (2)	-0.010 (2)	0.008 (2)
C4	0.039 (2)	0.0484 (19)	0.0462 (19)	-0.0020 (17)	0.0003 (16)	-0.0068 (15)
C7	0.057 (2)	0.0386 (18)	0.0351 (18)	-0.0119 (18)	-0.0021 (17)	0.0025 (14)
C3	0.074 (3)	0.057 (2)	0.074 (3)	0.002 (2)	-0.016 (2)	-0.027 (2)
C2	0.100 (4)	0.078 (3)	0.039 (2)	-0.011 (3)	-0.004 (2)	-0.0070 (19)
C12	0.085 (3)	0.050 (2)	0.045 (2)	-0.007 (2)	-0.003 (2)	-0.0055 (17)
C11	0.119 (5)	0.050 (2)	0.050 (2)	-0.015 (3)	-0.002 (3)	-0.0072 (18)
C10	0.100 (4)	0.068 (3)	0.046 (2)	-0.041 (3)	-0.016 (3)	0.000 (2)
C13	0.063 (3)	0.088 (3)	0.073 (3)	0.012 (2)	-0.007 (2)	0.008 (2)
C1	0.051 (3)	0.080 (3)	0.090 (3)	-0.014 (2)	-0.002 (2)	-0.021 (2)

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

S1—C6	1.665 (3)	C7—C12	1.382 (5)
O1—C5	1.215 (4)	C3—H3A	0.9600
O2—C8	1.378 (5)	C3—H3B	0.9600
O2—C13	1.416 (5)	C3—H3C	0.9600
C6—N2	1.323 (4)	C2—H2B	0.9600
C6—N1	1.399 (4)	C2—H2C	0.9600
N2—C7	1.411 (4)	C2—H2D	0.9600
N2—H2A	0.8600	C12—C11	1.378 (6)
N1—C5	1.375 (4)	C12—H12A	0.9300
N1—H1A	0.8600	C11—C10	1.366 (7)
C5—C4	1.530 (5)	C11—H11A	0.9300
C8—C9	1.373 (5)	C10—H10A	0.9300
C8—C7	1.394 (5)	C13—H13A	0.9600
C9—C10	1.385 (6)	C13—H13B	0.9600
C9—H9A	0.9300	C13—H13C	0.9600
C4—C1	1.522 (5)	C1—H1B	0.9600
C4—C2	1.529 (5)	C1—H1C	0.9600
C4—C3	1.529 (5)	C1—H1D	0.9600
C8—O2—C13	117.9 (3)	C4—C3—H3C	109.5

N2—C6—N1	114.9 (3)	H3A—C3—H3C	109.5
N2—C6—S1	128.3 (3)	H3B—C3—H3C	109.5
N1—C6—S1	116.8 (3)	C4—C2—H2B	109.5
C6—N2—C7	131.5 (3)	C4—C2—H2C	109.5
C6—N2—H2A	114.3	H2B—C2—H2C	109.5
C7—N2—H2A	114.3	C4—C2—H2D	109.5
C5—N1—C6	128.7 (3)	H2B—C2—H2D	109.5
C5—N1—H1A	115.7	H2C—C2—H2D	109.5
C6—N1—H1A	115.7	C11—C12—C7	120.2 (4)
O1—C5—N1	121.9 (3)	C11—C12—H12A	119.9
O1—C5—C4	122.0 (3)	C7—C12—H12A	119.9
N1—C5—C4	116.1 (3)	C10—C11—C12	120.4 (4)
C9—C8—O2	124.6 (4)	C10—C11—H11A	119.8
C9—C8—C7	121.0 (4)	C12—C11—H11A	119.8
O2—C8—C7	114.4 (3)	C11—C10—C9	120.5 (4)
C8—C9—C10	119.1 (4)	C11—C10—H10A	119.7
C8—C9—H9A	120.5	C9—C10—H10A	119.7
C10—C9—H9A	120.5	O2—C13—H13A	109.5
C1—C4—C2	110.8 (4)	O2—C13—H13B	109.5
C1—C4—C3	109.2 (3)	H13A—C13—H13B	109.5
C2—C4—C3	109.6 (3)	O2—C13—H13C	109.5
C1—C4—C5	109.4 (3)	H13A—C13—H13C	109.5
C2—C4—C5	109.4 (3)	H13B—C13—H13C	109.5
C3—C4—C5	108.4 (3)	C4—C1—H1B	109.5
C12—C7—C8	118.7 (3)	C4—C1—H1C	109.5
C12—C7—N2	125.5 (4)	H1B—C1—H1C	109.5
C8—C7—N2	115.7 (3)	C4—C1—H1D	109.5
C4—C3—H3A	109.5	H1B—C1—H1D	109.5
C4—C3—H3B	109.5	H1C—C1—H1D	109.5
H3A—C3—H3B	109.5		
N1—C6—N2—C7	-178.8 (3)	O1—C5—C4—C3	-4.2 (5)
S1—C6—N2—C7	2.0 (6)	N1—C5—C4—C3	176.6 (3)
N2—C6—N1—C5	-1.5 (5)	C9—C8—C7—C12	-2.3 (5)
S1—C6—N1—C5	177.9 (3)	O2—C8—C7—C12	177.6 (3)
C6—N1—C5—O1	2.2 (5)	C9—C8—C7—N2	-179.4 (3)
C6—N1—C5—C4	-178.6 (3)	O2—C8—C7—N2	0.5 (4)
C13—O2—C8—C9	10.6 (5)	C6—N2—C7—C12	19.9 (6)
C13—O2—C8—C7	-169.3 (3)	C6—N2—C7—C8	-163.3 (3)
O2—C8—C9—C10	-178.1 (3)	C8—C7—C12—C11	2.2 (6)
C7—C8—C9—C10	1.7 (5)	N2—C7—C12—C11	178.9 (3)
O1—C5—C4—C1	114.7 (4)	C7—C12—C11—C10	-1.4 (6)
N1—C5—C4—C1	-64.5 (4)	C12—C11—C10—C9	0.8 (6)
O1—C5—C4—C2	-123.7 (4)	C8—C9—C10—C11	-1.0 (6)
N1—C5—C4—C2	57.1 (4)		

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—H2A...O1	0.86	1.89	2.619 (4)	142

## supplementary materials

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N2—H2A···O2	0.86	2.17	2.575 (4)	109
C12—H12A···S1	0.93	2.62	3.235 (4)	124

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