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

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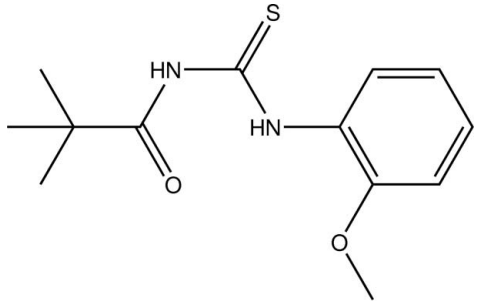
Key indicators: single-crystal X-ray study; $T$ = 273 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.068; $wR$ factor = 0.147; data-to-parameter ratio = 17.0.

In the title compound, $C_{13}H_{18}N_2O_2S$, 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*

$C_{13}H_{18}N_2O_2S$
$M_r$ = 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$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.23 mm$^{-1}$
$T$ = 273 K
0.50 × 0.14 × 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 $I > 2\sigma(I)$
$R_{int}$ = 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
$\Delta\rho_{max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$
Absolute structure: Flack (1983), 1141
Flack parameter: 0.63 (15)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2*A*···O1 | 0.86 | 1.89 | 2.619 (4) | 142 |
| N2—H2*A*···O2 | 0.86 | 2.17 | 2.575 (4) | 109 |
| C12—H12*A*···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).

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

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# supporting information

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

**Maisara A. Kadir, Bohari M. Yamin and M. Sukeri M. Yusof**

### S1. 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 replace 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 exist. There is no intermolecular hydrogen bond in the crystal structure.

### S2. 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.

### S3. 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 $U_{iso}$(H)= $xU_{eq}$(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.

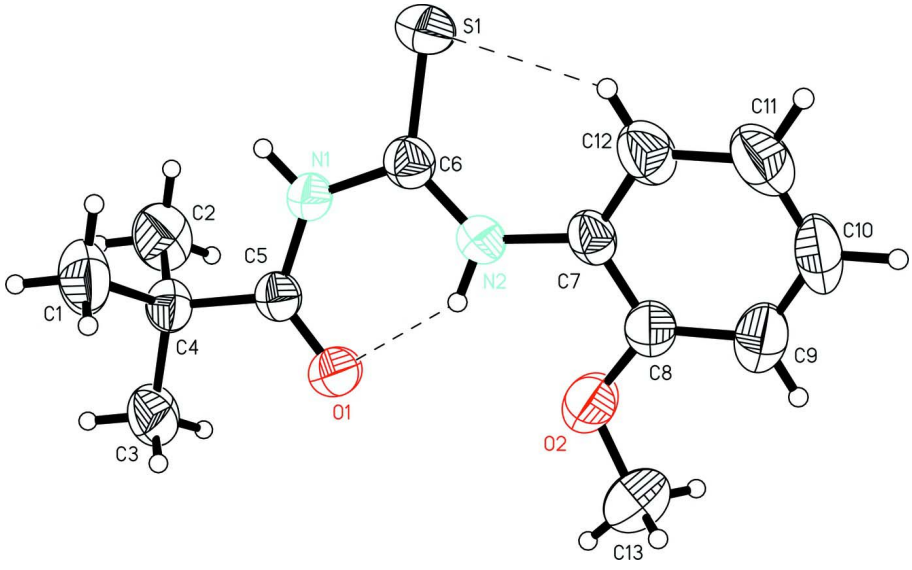


**Figure 1**

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$^3$
$Z = 4$
$F(000) = 568$
$D_x = 1.259$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 770 reflections
$\theta = 1.9$–$26.0°$
$\mu = 0.23$ mm$^{-1}$
$T = 273$ K
Slab, colourless
0.50 × 0.14 × 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$^{-1}$
$\omega$ 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 $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 26.0°$, $\theta_{min} = 1.9°$
$h = -7\rightarrow6$
$k = -16\rightarrow16$
$l = -20\rightarrow21$

*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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å$^{-3}$
$\Delta\rho_{min} = -0.18$ e Å$^{-3}$
Absolute structure: Flack (1983), 1141 Friedel pairs
Absolute structure 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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 (Å, º)*

| | | | |
|---|---|---|---|
| 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, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2*A*···O1 | 0.86 | 1.89 | 2.619 (4) | 142 |
| N2—H2*A*···O2 | 0.86 | 2.17 | 2.575 (4) | 109 |
| C12—H12*A*···S1 | 0.93 | 2.62 | 3.235 (4) | 124 |