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# 1-(3-Methoxyphenyl)-3-(4-methylbenzoyl)thiourea

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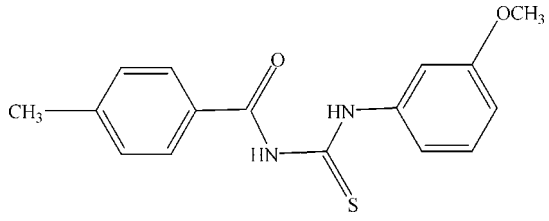
Key indicators: single-crystal X-ray study; $T$ = 120 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.045; $wR$ factor = 0.115; data-to-parameter ratio = 18.6.

In the structure of the title compound, $C_{16}H_{16}N_2O_2S$, the dihedral angle between the two aromatic ring planes is 48.3 (1)°. Intermolecular N—H···S hydrogen bonds link the molecules into dimers which are stacked along [100].

## Related literature

For related literature, see: Saeed & Flörke (2006, 2007).




## Experimental

### Crystal data

$C_{16}H_{16}N_2O_2S$
$M_r$ = 300.37
Monoclinic, $P2_1/n$
$a$ = 5.3031 (6) Å
$b$ = 21.159 (2) Å
$c$ = 13.2837 (14) Å
$\beta$ = 90.027 (15)°
$V$ = 1490.5 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.22 mm$^{-1}$
$T$ = 120 (2) K
0.52 × 0.10 × 0.09 mm

### Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min}$ = 0.893, $T_{max}$ = 0.980
12383 measured reflections
3535 independent reflections
2050 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.091

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.115
$S$ = 0.88
3535 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.33 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2···S1$^{i}$ | 0.88 | 2.89 | 3.7376 (19) | 163 |
| N1—H1···O1 | 0.88 | 1.89 | 2.621 (2) | 140 |

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

AS gratefully acknowledges a research grant from the Quaid-I-Azam University, Islamabad.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CS2042).

## References

Bruker (2002). *SMART* (Version 5.62), *SAINT* (Version 6.02), *SHELXTL* (Version 6.10) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.
Saeed, A. & Flörke, U. (2006). *Acta Cryst.* E**62**, o2924–o2925.
Saeed, A. & Flörke, U. (2007). *Acta Cryst.* E**63**, o1390–o1392.

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# supplementary materials

*Acta Cryst.* (2007). E**63**, o3695 [ **doi:10.1107/S1600536807037531** ]

# 1-(3-Methoxyphenyl)-3-(4-methylbenzoyl)thiourea

**A. Saeed and U. Flörke**

## Comment

The background to this study has been set out in the previous paper (Saeed & Flörke, 2007). In the molecular structure of the title compound, (I), (Fig. 1) the torsion angles C8—N2—C9—O1 of −5.4 (4)° and C9—N2—C8—N1 of 4.8 (3)° reflect the almost planar conformation of I with respect to the thiocarbonyl and carbonyl parts. The two aromatic rings form a dihedral angle of 48.30 (8)°, the associated N2—C9—C10—C15 torsion angle is −17.5 (3)°. All other bond parameters (Table 1) are typical for these thiourea compounds (Saeed & Flörke, 2006). Also typical is the intramolecular N1—H···O hydrogen bond. The crystal packing is determined by intermolecular N2—H···S hydrogen bonds, forming centrosymmetric dimers which are stacked along [100].

## Experimental

A solution of 4-methylbenzoyl chloride (1.75 g,10 mmol) in acetone (50 ml) was added dropwise to a suspension of potassium thiocyanate (0.97 g,10 mmol) in acetone (30 ml) and the reaction mixture was refluxed for 30 min. After cooling to room temperature, a solution of 3-methoxyaniline (10 mmol) in acetone (10 ml) was added and the resulting mixture refluxed for 2.0 h. The reaction mixture was poured into cold water when the thiourea was precipitated as a solid. Recrystallized from ethanol as colourless crystals (2.4 g, 8.0 mmol, 80%). m.p. 79°C. IR (KBr) cm-1: 3351 (free NH), 3200 (assoc. NH), 1667 (CO), 1610 (arom.), 1529 (thioureido I) 1325 II, 1160 III, 744, 762; 1H NMR (CDCl3) ?: 3.89 (3*H*, s, ArOCH3), 7.31–7.75 (aromatic), 9.19 (1*H*, s, broad, NH); 12.76 (1*H*, s, broad, NH); 13 C NMR (CDCl3) ? 55.6 (OCH3), 126.2 (4 CH), 129.0 (2 CH), 129.20 (2CH), 130.7 (C), 132.1 (C),134.8 (C) 142.3 (C), 168.1 (C=O), 178.4 (C=S). EIMS m/e: 300, 168.9,149, 119, 91, 64.9; Analysis calculated for $C_{16}H_{16}N_2O_2S$ C, 63.98; H, 5.37; N, 9.33; S, 10.67 found C, 64.01; H, 5.32; N, 9.10; S, 10.65

## Refinement

Hydrogen atoms were located in difference syntheses, constrained at idealized positions riding on the C (C—H = 0.95–0.99 Å) or N (N—H = 0.88 Å) atoms with isotropic displacement parameters $U_{iso}$(H) = 1.2U($C_{eq}$ / $N_{eq}$) and 1.5(methyl-C). Methyl H atoms were refined on the basis of rigid groups allowed to rotate but not tip.

## Figures

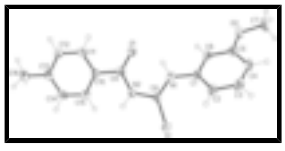

Fig. 1.

### 1-(3-Methoxyphenyl)-3-(4-methylbenzoyl)thiourea

*Crystal data*

| | |
|---|---|
| $C_{16}H_{16}N_2O_2S$ | $F_{000}$ = 632 |
| $M_r$ = 300.37 | $D_x$ = 1.339 Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation<br>$\lambda$ = 0.71073 Å |
| Hall symbol: -P 2yn | Cell parameters from 1407 reflections |
| $a$ = 5.3031 (6) Å | $\theta$ = 2.5–21.2º |
| $b$ = 21.159 (2) Å | $\mu$ = 0.22 mm$^{-1}$ |
| $c$ = 13.2837 (14) Å | $T$ = 120 (2) K |
| $\beta$ = 90.027 (15)º | Needle, colourless |
| $V$ = 1490.5 (3) Å$^3$ | 0.52 × 0.10 × 0.09 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX diffractometer | 3535 independent reflections |
| Radiation source: sealed tube | 2050 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.091 |
| $T$ = 120(2) K | $\theta_{max}$ = 27.9º |
| $\varphi$ and $\omega$ scans | $\theta_{min}$ = 1.8º |
| Absorption correction: multi-scan (SADABS; Bruker, 2002) | $h$ = −6→6 |
| $T_{min}$ = 0.893, $T_{max}$ = 0.980 | $k$ = −27→27 |
| 12383 measured reflections | $l$ = −17→16 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)]$ = 0.045 | H-atom parameters constrained |
| $wR(F^2)$ = 0.115 | $w = 1/[\sigma^2(F_o^2) + (0.0434P)^2]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 0.88 | $(\Delta/\sigma)_{max} < 0.001$ |
| 3535 reflections | $\Delta\rho_{max}$ = 0.32 e Å$^{-3}$ |
| 190 parameters | $\Delta\rho_{min}$ = −0.33 e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

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

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.32324 (14) | 1.01213 (3) | 0.85607 (5) | 0.0443 (2) |

| | | | | |
|---|---|---|---|---|
| O1 | 0.6061 (3) | 0.81054 (7) | 0.82493 (11) | 0.0427 (5) |
| O2 | 0.0758 (3) | 0.86111 (8) | 0.42661 (12) | 0.0484 (5) |
| N1 | 0.3031 (3) | 0.90015 (8) | 0.76221 (13) | 0.0325 (5) |
| H1 | 0.3542 | 0.8606 | 0.7636 | 0.039* |
| N2 | 0.5727 (3) | 0.90797 (8) | 0.89643 (13) | 0.0326 (5) |
| H2 | 0.6253 | 0.9309 | 0.9476 | 0.039* |
| C1 | 0.1346 (4) | 0.91542 (10) | 0.68283 (16) | 0.0302 (5) |
| C2 | −0.0701 (5) | 0.95549 (10) | 0.69401 (18) | 0.0371 (6) |
| H2A | −0.1001 | 0.9767 | 0.7559 | 0.044* |
| C3 | −0.2299 (5) | 0.96387 (11) | 0.6126 (2) | 0.0447 (7) |
| H3A | −0.3706 | 0.9913 | 0.6194 | 0.054* |
| C4 | −0.1907 (5) | 0.93341 (11) | 0.52169 (19) | 0.0421 (7) |
| H4A | −0.3028 | 0.9398 | 0.4668 | 0.050* |
| C5 | 0.0128 (5) | 0.89387 (10) | 0.51231 (17) | 0.0348 (6) |
| C6 | 0.1752 (4) | 0.88476 (9) | 0.59290 (17) | 0.0321 (6) |
| H6A | 0.3154 | 0.8572 | 0.5860 | 0.039* |
| C7 | −0.0996 (5) | 0.86212 (13) | 0.34504 (18) | 0.0579 (8) |
| H7A | −0.2651 | 0.8480 | 0.3689 | 0.087* |
| H7B | −0.0407 | 0.8338 | 0.2916 | 0.087* |
| H7C | −0.1132 | 0.9052 | 0.3187 | 0.087* |
| C8 | 0.3948 (4) | 0.93659 (10) | 0.83449 (16) | 0.0304 (5) |
| C9 | 0.6782 (5) | 0.84850 (10) | 0.88869 (16) | 0.0322 (5) |
| C10 | 0.8808 (4) | 0.83358 (9) | 0.96113 (15) | 0.0278 (5) |
| C11 | 0.9470 (5) | 0.77047 (10) | 0.97489 (16) | 0.0360 (6) |
| H11A | 0.8625 | 0.7384 | 0.9378 | 0.043* |
| C12 | 1.1343 (5) | 0.75421 (10) | 1.04196 (16) | 0.0362 (6) |
| H12A | 1.1783 | 0.7110 | 1.0500 | 0.043* |
| C13 | 1.2603 (4) | 0.79982 (10) | 1.09818 (15) | 0.0317 (5) |
| C14 | 1.1956 (5) | 0.86231 (10) | 1.08289 (17) | 0.0381 (6) |
| H14A | 1.2806 | 0.8943 | 1.1198 | 0.046* |
| C15 | 1.0105 (4) | 0.87923 (10) | 1.01519 (16) | 0.0342 (6) |
| H15A | 0.9714 | 0.9226 | 1.0055 | 0.041* |
| C16 | 1.4626 (5) | 0.78203 (11) | 1.17233 (17) | 0.0431 (6) |
| H16A | 1.6214 | 0.8024 | 1.1532 | 0.065* |
| H16B | 1.4848 | 0.7360 | 1.1722 | 0.065* |
| H16C | 1.4133 | 0.7960 | 1.2398 | 0.065* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0624 (5) | 0.0291 (3) | 0.0416 (4) | 0.0117 (3) | −0.0172 (3) | −0.0020 (3) |
| O1 | 0.0533 (12) | 0.0361 (9) | 0.0387 (10) | 0.0122 (8) | −0.0175 (9) | −0.0076 (7) |
| O2 | 0.0523 (13) | 0.0539 (11) | 0.0390 (10) | 0.0027 (9) | −0.0230 (9) | −0.0015 (8) |
| N1 | 0.0388 (13) | 0.0261 (9) | 0.0327 (11) | 0.0039 (9) | −0.0077 (9) | 0.0008 (8) |
| N2 | 0.0410 (13) | 0.0304 (10) | 0.0264 (10) | 0.0067 (9) | −0.0094 (9) | −0.0023 (8) |
| C1 | 0.0281 (14) | 0.0267 (11) | 0.0357 (13) | −0.0049 (10) | −0.0084 (11) | 0.0077 (10) |
| C2 | 0.0311 (15) | 0.0371 (13) | 0.0429 (15) | −0.0004 (11) | −0.0023 (12) | 0.0045 (11) |
| C3 | 0.0281 (16) | 0.0392 (14) | 0.0667 (19) | 0.0018 (12) | −0.0078 (14) | 0.0118 (13) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C4 | 0.0345 (16) | 0.0438 (14) | 0.0479 (17) | −0.0037 (12) | −0.0199 (13) | 0.0147 (12) |
| C5 | 0.0366 (16) | 0.0316 (12) | 0.0362 (15) | −0.0092 (11) | −0.0106 (12) | 0.0068 (10) |
| C6 | 0.0306 (15) | 0.0281 (11) | 0.0376 (14) | 0.0010 (10) | −0.0091 (11) | 0.0036 (10) |
| C7 | 0.060 (2) | 0.076 (2) | 0.0387 (16) | −0.0074 (15) | −0.0272 (15) | 0.0082 (13) |
| C8 | 0.0335 (15) | 0.0318 (12) | 0.0259 (12) | 0.0034 (10) | −0.0014 (11) | 0.0029 (9) |
| C9 | 0.0377 (15) | 0.0315 (12) | 0.0274 (13) | 0.0043 (11) | −0.0006 (11) | −0.0007 (10) |
| C10 | 0.0300 (14) | 0.0309 (12) | 0.0224 (12) | 0.0049 (10) | −0.0026 (10) | 0.0005 (9) |
| C11 | 0.0426 (16) | 0.0319 (12) | 0.0337 (14) | 0.0029 (11) | −0.0105 (12) | −0.0056 (10) |
| C12 | 0.0409 (16) | 0.0303 (12) | 0.0375 (14) | 0.0075 (11) | −0.0073 (12) | 0.0016 (10) |
| C13 | 0.0291 (14) | 0.0442 (13) | 0.0217 (12) | 0.0018 (11) | 0.0011 (10) | 0.0008 (10) |
| C14 | 0.0402 (16) | 0.0358 (13) | 0.0382 (14) | −0.0041 (11) | −0.0054 (12) | −0.0068 (11) |
| C15 | 0.0390 (16) | 0.0270 (12) | 0.0366 (14) | 0.0007 (10) | −0.0030 (12) | −0.0003 (10) |
| C16 | 0.0375 (16) | 0.0543 (16) | 0.0374 (15) | 0.0000 (12) | −0.0074 (12) | 0.0000 (12) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| S1—C8 | 1.668 (2) | C6—H6A | 0.9500 |
| O1—C9 | 1.228 (2) | C7—H7A | 0.9800 |
| O2—C5 | 1.374 (3) | C7—H7B | 0.9800 |
| O2—C7 | 1.428 (3) | C7—H7C | 0.9800 |
| N1—C8 | 1.324 (3) | C9—C10 | 1.477 (3) |
| N1—C1 | 1.419 (3) | C10—C15 | 1.386 (3) |
| N1—H1 | 0.8800 | C10—C11 | 1.393 (3) |
| N2—C9 | 1.381 (3) | C11—C12 | 1.378 (3) |
| N2—C8 | 1.391 (3) | C11—H11A | 0.9500 |
| N2—H2 | 0.8800 | C12—C13 | 1.391 (3) |
| C1—C6 | 1.376 (3) | C12—H12A | 0.9500 |
| C1—C2 | 1.385 (3) | C13—C14 | 1.381 (3) |
| C2—C3 | 1.385 (3) | C13—C16 | 1.504 (3) |
| C2—H2A | 0.9500 | C14—C15 | 1.379 (3) |
| C3—C4 | 1.385 (3) | C14—H14A | 0.9500 |
| C3—H3A | 0.9500 | C15—H15A | 0.9500 |
| C4—C5 | 1.371 (3) | C16—H16A | 0.9800 |
| C4—H4A | 0.9500 | C16—H16B | 0.9800 |
| C5—C6 | 1.387 (3) | C16—H16C | 0.9800 |
| C5—O2—C7 | 117.6 (2) | N1—C8—N2 | 115.12 (18) |
| C8—N1—C1 | 129.59 (18) | N1—C8—S1 | 126.83 (17) |
| C8—N1—H1 | 115.2 | N2—C8—S1 | 118.03 (16) |
| C1—N1—H1 | 115.2 | O1—C9—N2 | 121.4 (2) |
| C9—N2—C8 | 128.87 (18) | O1—C9—C10 | 122.4 (2) |
| C9—N2—H2 | 115.6 | N2—C9—C10 | 116.18 (19) |
| C8—N2—H2 | 115.6 | C15—C10—C11 | 118.4 (2) |
| C6—C1—C2 | 120.3 (2) | C15—C10—C9 | 123.34 (19) |
| C6—C1—N1 | 116.0 (2) | C11—C10—C9 | 118.28 (19) |
| C2—C1—N1 | 123.6 (2) | C12—C11—C10 | 120.4 (2) |
| C3—C2—C1 | 118.3 (2) | C12—C11—H11A | 119.8 |
| C3—C2—H2A | 120.9 | C10—C11—H11A | 119.8 |
| C1—C2—H2A | 120.9 | C11—C12—C13 | 121.3 (2) |
| C2—C3—C4 | 122.0 (2) | C11—C12—H12A | 119.3 |

| | | | |
|---|---|---|---|
| C2—C3—H3A | 119.0 | C13—C12—H12A | 119.3 |
| C4—C3—H3A | 119.0 | C14—C13—C12 | 117.8 (2) |
| C5—C4—C3 | 118.8 (2) | C14—C13—C16 | 120.9 (2) |
| C5—C4—H4A | 120.6 | C12—C13—C16 | 121.4 (2) |
| C3—C4—H4A | 120.6 | C15—C14—C13 | 121.4 (2) |
| C4—C5—O2 | 125.0 (2) | C15—C14—H14A | 119.3 |
| C4—C5—C6 | 120.2 (2) | C13—C14—H14A | 119.3 |
| O2—C5—C6 | 114.7 (2) | C14—C15—C10 | 120.7 (2) |
| C1—C6—C5 | 120.5 (2) | C14—C15—H15A | 119.7 |
| C1—C6—H6A | 119.8 | C10—C15—H15A | 119.7 |
| C5—C6—H6A | 119.8 | C13—C16—H16A | 109.5 |
| O2—C7—H7A | 109.5 | C13—C16—H16B | 109.5 |
| O2—C7—H7B | 109.5 | H16A—C16—H16B | 109.5 |
| H7A—C7—H7B | 109.5 | C13—C16—H16C | 109.5 |
| O2—C7—H7C | 109.5 | H16A—C16—H16C | 109.5 |
| H7A—C7—H7C | 109.5 | H16B—C16—H16C | 109.5 |
| H7B—C7—H7C | 109.5 | | |
| C8—N1—C1—C6 | 144.9 (2) | C9—N2—C8—S1 | −173.97 (18) |
| C8—N1—C1—C2 | −39.0 (3) | C8—N2—C9—O1 | −5.4 (4) |
| C6—C1—C2—C3 | −0.3 (3) | C8—N2—C9—C10 | 174.6 (2) |
| N1—C1—C2—C3 | −176.2 (2) | O1—C9—C10—C15 | 162.5 (2) |
| C1—C2—C3—C4 | 0.2 (3) | N2—C9—C10—C15 | −17.5 (3) |
| C2—C3—C4—C5 | −0.1 (4) | O1—C9—C10—C11 | −16.7 (3) |
| C3—C4—C5—O2 | −179.4 (2) | N2—C9—C10—C11 | 163.3 (2) |
| C3—C4—C5—C6 | 0.1 (3) | C15—C10—C11—C12 | 1.0 (3) |
| C7—O2—C5—C4 | −8.0 (3) | C9—C10—C11—C12 | −179.7 (2) |
| C7—O2—C5—C6 | 172.4 (2) | C10—C11—C12—C13 | 0.6 (4) |
| C2—C1—C6—C5 | 0.4 (3) | C11—C12—C13—C14 | −1.5 (4) |
| N1—C1—C6—C5 | 176.56 (19) | C11—C12—C13—C16 | 179.2 (2) |
| C4—C5—C6—C1 | −0.3 (3) | C12—C13—C14—C15 | 0.8 (4) |
| O2—C5—C6—C1 | 179.32 (18) | C16—C13—C14—C15 | −180.0 (2) |
| C1—N1—C8—N2 | −175.1 (2) | C13—C14—C15—C10 | 0.9 (4) |
| C1—N1—C8—S1 | 3.5 (4) | C11—C10—C15—C14 | −1.7 (3) |
| C9—N2—C8—N1 | 4.8 (3) | C9—C10—C15—C14 | 179.1 (2) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2···S1$^{i}$ | 0.88 | 2.89 | 3.7376 (19) | 163 |
| N1—H1···O1 | 0.88 | 1.89 | 2.621 (2) | 140 |

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

**Fig. 1**