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N-[3-(Hydroxymethyl)phenyl]-*N*′-(4-methoxybenzoyl)thiourea

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.054
wR factor = 0.128
Data-to-parameter ratio = 15.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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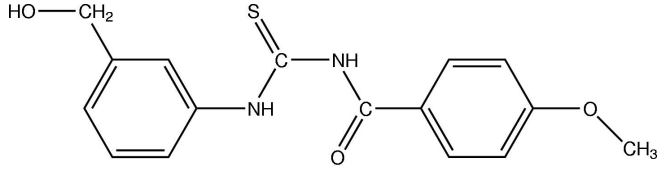
The 4-methoxybenzoyl and 3-(hydroxymethyl)phenyl groups in the title compound, $C_{16}H_{16}N_2O_3S$, are *trans* and *cis*, respectively, with respect to the thione group across the C—N bonds. The central carbonylthiourea moiety makes dihedral angles with the 4-methoxybenzoyl and 3-(hydroxymethyl)-phenyl fragments of 33.48 (7) and 45.96 (6)°, respectively. The molecules are stabilized by intermolecular N—H···O and O—H···S hydrogen bonds to form tetramers.

Comment

In the title compound, (I), the molecular structure and dimensions are similar to those in other benzoylthiourea derivatives, such as *N*-(2-chlorophenyl)-*N*′-(4-methoxybenzoyl)thiourea (Yusof & Yamin, 2004*a*), *N*-(4-methoxybenzoyl-*N*′-(*o*-tolyl)thiourea (Yusof & Yamin, 2004*b*) and *N*-(*p*-methoxybenzoyl)-*N*′-(*o*-methoxyphenyl)thiourea (Ali *et al.*, 2004). The molecule maintains its *trans-cis* configuration with respect to the position of the 4-methoxybenzoyl and 3-(hydroxymethyl)phenyl groups relative to the S atom across the thiourea C—N bonds.



(I)

The central carbonylthiourea moiety (S1/O1/N1/N2/C7/C8), the 4-methoxybenzoyl group (C1–C6/O2/C15) and the 3-(hydroxymethyl)phenyl (C9–C13/C16/O3) group are individually planar, the maximum deviation being 0.044 (2) Å for atom O2. The C8═S1, C8—N1 and C8—N2 bond lengths are 1.672 (2), 1.390 (2) and 1.333 (3) Å, respectively, comparable with those in *N*-(2-chlorophenyl)-*N*′-(4-methoxybenzoyl)-thiourea [C═S = 1.662 (2) Å, C8—N1 = 1.386 (3) Å and C8—N2 = 1.331 (3) Å; Yusof & Yamin, 2004*a*] and other

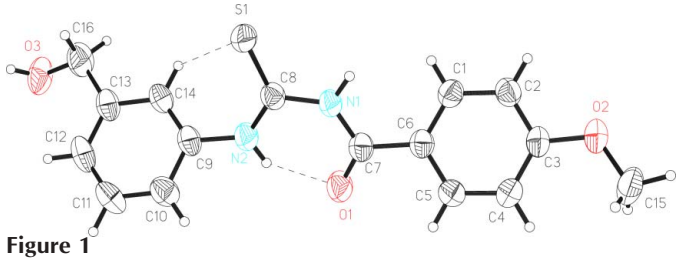


Figure 1
The molecular structure of the title compound, shown with 50% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines.

benzoylthiourea derivatives. The other bond lengths and angles are in normal ranges (Allen *et al.*, 1987). The dihedral angles between the central carbonylthiourea moiety and the 4-methoxybenzoyl and 3-(hydroxymethyl)phenyl fragments are 33.48 (7) and 45.96 (6)°, respectively. The two aryl groups are inclined at an angle of 11.99 (10)°. There are two intramolecular hydrogen bonds, *viz*. N2—H2···O1 and C14—H14···S1; as a result, two pseudo-six-membered rings, N2—H2*A*···O1—C7—N1—C8 and C14—H14*A*···S1—C8—N2—C9, respectively, are formed. In the crystal structure, the molecules are linked by intermolecular hydrogen bonds, *viz*. N1—H1···O3^{i} and O3—H3···S1^{ii} (symmetry codes as in Table 1), to form tetramers (Fig. 2).

Experimental

(3-Aminophenyl)methanol (0.25 g, 2.03 mmol) in acetone (50 ml) was added dropwise to a two-necked round-bottomed flask containing 4-methoxybenzoyl isothiocyanate (0.39 g, 2.03 mmol) in acetone (50 ml). The solution was refluxed for 2 h and the filtrate was then cooled in ice. The yellow precipitate was filtered off and washed with distilled water. Recrystallization from chloroform yielded single crystals suitable for X-ray analysis (m.p. 438–439 K). Analysis calculated for $C_{16}H_{16}N_2O_3S$: C 60.7, H 5.09, N 8.8, S 10.1, O 15.3%; found: C 59.9, H 5.15, N 8.5, S 9.7, O 14.9%.

Crystal data

$C_{16}H_{16}N_2O_3S$
M_r = 316.37
Monoclinic, $C2/c$
a = 17.559 (7) Å
b = 8.959 (4) Å
c = 19.566 (8) Å
β = 97.591 (9)°
V = 3051 (2) Å^3
Z = 8
D_x = 1.377 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 864 reflections
θ = 2.1–26.5°
μ = 0.23 mm^{-1}
T = 298 (2) K
Block, colourless
0.40 × 0.29 × 0.26 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SABABS*; Sheldrick, 1996)
T_{min} = 0.915, T_{max} = 0.944
8291 measured reflections
3144 independent reflections
2828 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 26.5°
$h = -22 \rightarrow 22$
$k = -11 \rightarrow 11$
$l = -24 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.128
S = 1.19
3144 reflections
201 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 2.0214P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.86	1.90	2.615 (3)	140
C14—H14···S1	0.93	2.87	3.279 (3)	108
N1—H1···O3^{i}	0.86	2.14	2.971 (3)	162
O3—H3···S1^{ii}	0.82	2.63	3.401 (3)	158

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

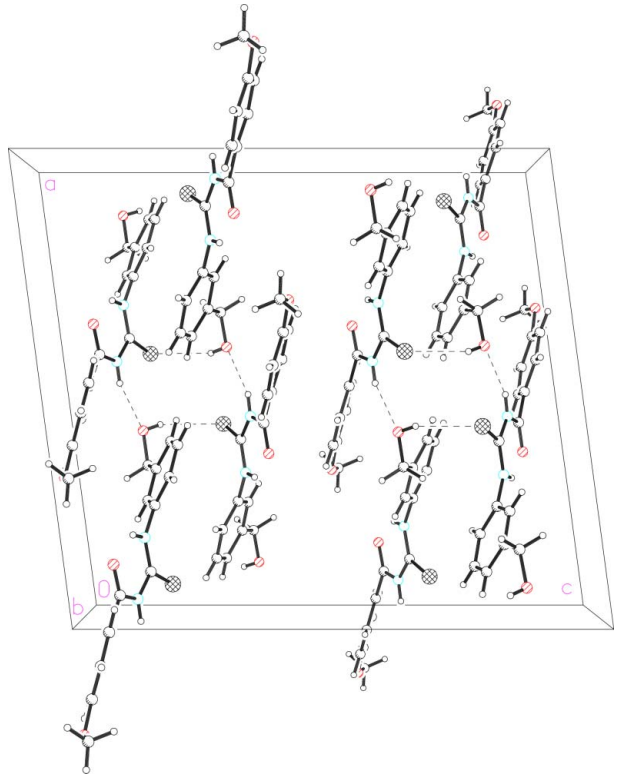


Figure 2
Packing diagram of the title complex, viewed down the *b* axis. The dashed lines denote N—H···O and O—H···S hydrogen bonds.

After their location in a difference map, all H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and U_{iso}(H) = 1.2U_{eq}(C) for CH and CH_2 groups, 1.5U_{eq}(C) for CH_3 groups and 1.2U_{eq}(N) for NH groups.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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