

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

1-Benzoyl-3-(4-hydroxyphenyl)thiourea

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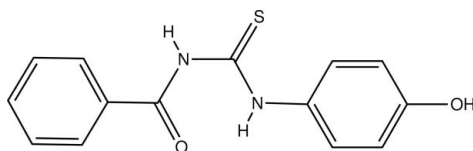
Received 8 October 2010; accepted 8 November 2010

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.031; wR factor = 0.085; data-to-parameter ratio = 13.6.

In the title compound, $\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_2\text{S}$, the aminophenol and the benzoyl groups adopt a *syn-anti* configuration with respect to the thiono $\text{C}=\text{S}$ group across the thiourea $\text{C}-\text{N}$. The dihedral angle between the mean planes of the benzoyl and hydroxyphenyl rings is $36.77(8)^\circ$. The molecules are stabilized by intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. In the crystal, weak intermolecular $\text{C}-\text{H}\cdots\text{O}$, $\text{O}-\text{H}\cdots\text{S}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into a chain along the c axis.

Related literature

For the preparation and chemical properties of related compounds, see: Zhang *et al.* (2001). For related structures, see: Abosadiya *et al.* (2007); Hung *et al.* (2010); Yamin & Yusof (2003). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

 $\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_2\text{S}$ $M_r = 272.33$ Orthorhombic, $P2_12_12_1$ $a = 5.5865(10)$ Å $b = 14.451(2)$ Å $c = 16.462(3)$ Å $V = 1329.0(4)$ Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.24$ mm⁻¹ $T = 298$ K $0.50 \times 0.48 \times 0.35$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer

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

 $T_{\min} = 0.886$, $T_{\max} = 0.919$

7608 measured reflections

2351 independent reflections

2143 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.018$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.031$ $wR(F^2) = 0.085$ $S = 1.05$

2351 reflections

173 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.13$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Absolute structure: Flack (1983),

958 Friedel pairs

Flack parameter: 0.09 (9)

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N}2-\text{H}2\text{B}\cdots\text{O}1$	0.86	1.95	2.631 (2)	135
$\text{N}1-\text{H}1\text{B}\cdots\text{O}2^{\text{i}}$	0.86	2.29	3.109 (2)	158
$\text{O}2-\text{H}2\text{C}\cdots\text{S}1^{\text{ii}}$	0.82	2.53	3.1533 (18)	134
$\text{C}1-\text{H}1\text{A}\cdots\text{O}2^{\text{i}}$	0.93	2.51	3.429 (3)	172
$\text{C}11-\text{H}11\text{A}\cdots\text{O}1^{\text{iii}}$	0.93	2.43	3.262 (2)	149

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

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL, PARST (Nardelli, 1995) and PLATON (Spek, 2009).

The authors thank Universiti Kebangsaan Malaysia for providing facilities and grants UKM-GUP-BTT-07-30-190 and UKM-OUP-TK-16-73/2010, the Libyan Government for providing a scholarship for AA and J.-C. Daran for his advice.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JJ2068).

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supplementary materials

Acta Cryst. (2010). E66, o3181 [doi:10.1107/S1600536810045988]

1-Benzoyl-3-(4-hydroxyphenyl)thiourea

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Comment

The title compound, I, is closely related to the previously reported *N*-benzoyl-*N'*-(3-hydroxyphenyl)thiourea (II) (Abosadiya *et al.*, 2007) compound. As in most benzoylthiourea derivatives of the type $R_1C(O)NHC(S)NHR_2$, the title compound has a *syn-anti* configuration for the hydroxyphenyl and benzoyl groups with respect to the thiono C=S bond across the thiourea C—N bond (Fig 1). The bond lengths and angles in the molecules are in normal ranges (Allen *et al.*, 1987) and comparable to those in (II). All C—N bond lengths are shorter than the normal value for C—N single bond, and the bond lengths C7—O1 and C8—S1 become longer than normal values for a double bond, which suggests the presence of delocalized π -electrons. For example, the C=S bond (1.671 (2) Å) and C—N bond lengths (C8—N1 = 1.388 (2) Å, C8—N2 = 1.328 (2) Å and C9—N2 = 1.428 (2) Å) in the title compound are longer than that observed in an unsubstituted phenyl ring (III - Yamin *et al.*, 2003) in which the C=S bond length (1.6567 (15) Å) and C—N bond lengths are 1.393 (2), 1.326 (2) and 1.408 (2) Å, respectively. This is due to donating effect of OH group in the *para* position, which contributes to an increase in the bond length.

The benzoyl ring [C1/C2/C3/C4/C5/C6/C7/O1] (A), hydroxyphenyl ring [N2/C9/C10/C11/C12/C13/C14/O2] (B) and thiourea [(S1/N1/N2/C8)] (C) fragments are essentially planar with maximum deviations from their mean planes of 0.045 (2) Å for atom O1 (A), 0.010 (2) Å for atom C10 (B) and 0.008 (2) Å for atom C8 (C), respectively. The dihedral angle between the mean planes of A and B is 36.77 (8)°. The crystal structure is stabilized by an intramolecular N2—H2B \cdots O1 hydrogen bond which forms a six-membered ring (N2/H2B/O1/C7/N1/C8) commonly observed in this class of ligands. In addition, the molecules are linked by weak intermolecular C11—H \cdots O1, C1—H \cdots O2, O2—H \cdots S1 and N1—H \cdots O2 hydrogen bonds (Table 2), resulting in a one-dimensional chain along the *c*-axis (Fig 2).

Experimental

The title compound was first synthesized by (Zhang *et al.*, 2001) under the condition of solid-liquid phase transfer catalysis using polyethylene glycol as catalyst, however, a much simpler method was used to synthesize the title compound. The reaction scheme involved a reaction of benzoyl chloride (10 mmol) with ammonium thiocyanate (10 mmol) in dry acetone. The product, benzoyl isothiocyanate was reacted with 4-hydroxy aniline (10 mmol) to give the title compound with a 56% yield. A slow evaporation of ethanolic solution of the compound gave light brown crystals suitable for X-ray diffraction.

Refinement

All the non hydrogen atom were refined anisotropically. the hydrogen positions were calculated to give an idealized geometry fixed to ride on their respective atoms, with $U_{iso}=1.2U_{eq}$ (C) for aromatic (CH = 0.93 Å), $U_{iso}=1.2U_{eq}$ (N) for N (NH = 0.86 Å) and $U_{iso}=1.5U_{eq}$ (O) for OH (OH = 0.82 Å).

Figures

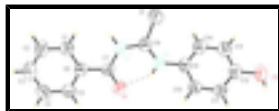


Fig. 1. The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level. Dashed lines indicate intramolecular N—H...O hydrogen bonds.

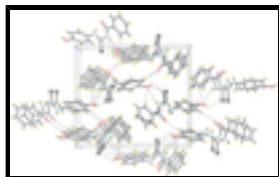


Fig. 2. Crystal packing of (I) viewed down the *a* axis. Dashed lines indicate weak C—H...O, C—H...O, O—H...S and N—H...O hydrogen bonds.

1-Benzoyl-3-(4-hydroxyphenyl)thiourea

Crystal data

$C_{14}H_{12}N_2O_2S$

$M_r = 272.33$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 5.5865$ (10) Å

$b = 14.451$ (2) Å

$c = 16.462$ (3) Å

$V = 1329.0$ (4) Å³

$Z = 4$

$F(000) = 568$

$D_x = 1.361$ Mg m⁻³

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

Cell parameters from 1024 reflections

$\theta = 1.9$ – 25.0°

$\mu = 0.24$ mm⁻¹

$T = 298$ K

Block, brown

$0.50 \times 0.48 \times 0.35$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

ω scans

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

$T_{\min} = 0.886$, $T_{\max} = 0.919$

7608 measured reflections

2351 independent reflections

2143 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 1.9^\circ$

$h = -6 \rightarrow 6$

$k = -15 \rightarrow 17$

$l = -19 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.031$

$wR(F^2) = 0.085$

$S = 1.05$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 0.1346P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

2351 reflections	$\Delta\rho_{\max} = 0.13 \text{ e } \text{\AA}^{-3}$
173 parameters	$\Delta\rho_{\min} = -0.14 \text{ e } \text{\AA}^{-3}$
0 restraints	Absolute structure: Flack (1983), 958 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.09 (9)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	1.13932 (14)	-0.04354 (4)	0.71251 (4)	0.0812 (2)
O1	0.7785 (3)	0.23667 (10)	0.72476 (9)	0.0719 (4)
N1	0.8692 (3)	0.09206 (10)	0.76731 (8)	0.0518 (4)
H1B	0.8429	0.0530	0.8057	0.062*
N2	1.0996 (3)	0.12902 (11)	0.65596 (9)	0.0565 (4)
H2B	1.0469	0.1841	0.6646	0.068*
C1	0.5090 (4)	0.10914 (14)	0.89160 (13)	0.0632 (5)
H1A	0.6077	0.0573	0.8928	0.076*
C2	0.3268 (5)	0.11715 (16)	0.94751 (14)	0.0712 (6)
H2A	0.3032	0.0712	0.9862	0.085*
C3	0.1812 (4)	0.19276 (18)	0.94584 (14)	0.0726 (6)
H3A	0.0581	0.1983	0.9836	0.087*
C4	0.2149 (4)	0.26068 (17)	0.88890 (14)	0.0734 (6)
H4A	0.1140	0.3119	0.8879	0.088*
C5	0.3978 (4)	0.25336 (14)	0.83317 (12)	0.0632 (5)
H5A	0.4208	0.3000	0.7950	0.076*
C6	0.5475 (3)	0.17701 (12)	0.83367 (10)	0.0487 (4)
C7	0.7395 (4)	0.17240 (12)	0.77127 (10)	0.0512 (4)
C8	1.0373 (4)	0.06508 (13)	0.70994 (11)	0.0527 (4)
C9	1.2439 (4)	0.11670 (13)	0.58521 (11)	0.0504 (4)
C10	1.1577 (4)	0.15191 (12)	0.51275 (11)	0.0520 (5)
H10A	1.0118	0.1829	0.5119	0.062*
C11	1.2858 (4)	0.14146 (12)	0.44198 (11)	0.0518 (5)
H11A	1.2257	0.1643	0.3933	0.062*
C12	1.5042 (3)	0.09692 (12)	0.44377 (12)	0.0515 (5)
C13	1.5928 (4)	0.06247 (16)	0.51603 (11)	0.0596 (5)
H13A	1.7399	0.0323	0.5170	0.072*

supplementary materials

C14	1.4626 (4)	0.07290 (16)	0.58682 (12)	0.0594 (5)
H14A	1.5229	0.0503	0.6356	0.071*
O2	1.6406 (3)	0.08472 (10)	0.37487 (9)	0.0682 (4)
H2C	1.5717	0.1079	0.3359	0.102*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.1110 (5)	0.0497 (3)	0.0828 (4)	0.0182 (3)	0.0277 (4)	0.0010 (3)
O1	0.1018 (11)	0.0536 (8)	0.0603 (8)	0.0184 (8)	0.0223 (8)	0.0179 (6)
N1	0.0712 (10)	0.0432 (8)	0.0410 (7)	0.0061 (8)	0.0053 (7)	0.0030 (6)
N2	0.0712 (10)	0.0480 (8)	0.0504 (8)	0.0014 (8)	0.0101 (8)	-0.0027 (7)
C1	0.0732 (14)	0.0513 (11)	0.0649 (12)	0.0051 (10)	0.0142 (10)	0.0055 (9)
C2	0.0783 (15)	0.0630 (13)	0.0723 (13)	-0.0108 (12)	0.0191 (12)	0.0028 (11)
C3	0.0575 (13)	0.0890 (17)	0.0713 (13)	-0.0058 (12)	0.0105 (11)	-0.0166 (13)
C4	0.0649 (14)	0.0824 (16)	0.0728 (14)	0.0219 (12)	0.0005 (12)	-0.0090 (12)
C5	0.0755 (14)	0.0596 (12)	0.0546 (11)	0.0119 (11)	-0.0026 (11)	0.0023 (9)
C6	0.0573 (11)	0.0472 (10)	0.0416 (8)	0.0009 (8)	-0.0045 (8)	-0.0040 (7)
C7	0.0669 (11)	0.0461 (10)	0.0406 (9)	0.0026 (9)	-0.0032 (8)	0.0018 (7)
C8	0.0627 (11)	0.0508 (10)	0.0446 (9)	-0.0008 (8)	-0.0022 (8)	-0.0056 (8)
C9	0.0560 (11)	0.0481 (9)	0.0471 (9)	-0.0079 (9)	0.0045 (8)	-0.0071 (8)
C10	0.0548 (11)	0.0411 (9)	0.0600 (11)	0.0009 (9)	0.0083 (9)	0.0050 (8)
C11	0.0618 (12)	0.0426 (10)	0.0509 (10)	-0.0032 (8)	0.0049 (9)	0.0051 (8)
C12	0.0543 (11)	0.0470 (10)	0.0531 (10)	-0.0119 (9)	0.0061 (8)	-0.0105 (8)
C13	0.0464 (10)	0.0724 (13)	0.0602 (11)	-0.0001 (10)	-0.0044 (9)	-0.0115 (10)
C14	0.0522 (11)	0.0780 (13)	0.0480 (10)	-0.0026 (10)	-0.0083 (8)	-0.0081 (9)
O2	0.0727 (10)	0.0735 (9)	0.0583 (8)	0.0002 (8)	0.0169 (7)	-0.0080 (7)

Geometric parameters (\AA , $^\circ$)

S1—C8	1.670 (2)	C4—H4A	0.9300
O1—C7	1.223 (2)	C5—C6	1.385 (3)
N1—C7	1.370 (2)	C5—H5A	0.9300
N1—C8	1.388 (2)	C6—C7	1.486 (3)
N1—H1B	0.8600	C9—C14	1.376 (3)
N2—C8	1.328 (2)	C9—C10	1.384 (3)
N2—C9	1.428 (2)	C10—C11	1.376 (3)
N2—H2B	0.8600	C10—H10A	0.9300
C1—C2	1.377 (3)	C11—C12	1.379 (3)
C1—C6	1.385 (3)	C11—H11A	0.9300
C1—H1A	0.9300	C12—O2	1.378 (2)
C2—C3	1.362 (3)	C12—C13	1.381 (3)
C2—H2A	0.9300	C13—C14	1.382 (3)
C3—C4	1.370 (3)	C13—H13A	0.9300
C3—H3A	0.9300	C14—H14A	0.9300
C4—C5	1.377 (3)	O2—H2C	0.8200
C7—N1—C8	128.94 (15)	O1—C7—C6	121.81 (17)
C7—N1—H1B	115.5	N1—C7—C6	116.91 (15)

C8—N1—H1B	115.5	N2—C8—N1	115.91 (16)
C8—N2—C9	127.36 (16)	N2—C8—S1	125.54 (15)
C8—N2—H2B	116.3	N1—C8—S1	118.53 (13)
C9—N2—H2B	116.3	C14—C9—C10	119.66 (17)
C2—C1—C6	121.0 (2)	C14—C9—N2	122.90 (17)
C2—C1—H1A	119.5	C10—C9—N2	117.43 (17)
C6—C1—H1A	119.5	C11—C10—C9	120.56 (18)
C3—C2—C1	119.7 (2)	C11—C10—H10A	119.7
C3—C2—H2A	120.2	C9—C10—H10A	119.7
C1—C2—H2A	120.2	C10—C11—C12	119.57 (18)
C2—C3—C4	120.4 (2)	C10—C11—H11A	120.2
C2—C3—H3A	119.8	C12—C11—H11A	120.2
C4—C3—H3A	119.8	O2—C12—C11	122.08 (18)
C3—C4—C5	120.2 (2)	O2—C12—C13	117.68 (18)
C3—C4—H4A	119.9	C11—C12—C13	120.24 (18)
C5—C4—H4A	119.9	C12—C13—C14	119.87 (19)
C4—C5—C6	120.4 (2)	C12—C13—H13A	120.1
C4—C5—H5A	119.8	C14—C13—H13A	120.1
C6—C5—H5A	119.8	C9—C14—C13	120.08 (19)
C1—C6—C5	118.32 (18)	C9—C14—H14A	120.0
C1—C6—C7	123.80 (17)	C13—C14—H14A	120.0
C5—C6—C7	117.87 (17)	C12—O2—H2C	109.5
O1—C7—N1	121.28 (18)		
C6—C1—C2—C3	0.2 (3)	C9—N2—C8—S1	7.0 (3)
C1—C2—C3—C4	0.1 (4)	C7—N1—C8—N2	7.5 (3)
C2—C3—C4—C5	-0.5 (4)	C7—N1—C8—S1	-171.15 (16)
C3—C4—C5—C6	0.6 (3)	C8—N2—C9—C14	-49.9 (3)
C2—C1—C6—C5	-0.1 (3)	C8—N2—C9—C10	130.9 (2)
C2—C1—C6—C7	-179.5 (2)	C14—C9—C10—C11	1.6 (3)
C4—C5—C6—C1	-0.3 (3)	N2—C9—C10—C11	-179.13 (16)
C4—C5—C6—C7	179.15 (19)	C9—C10—C11—C12	-1.2 (3)
C8—N1—C7—O1	-7.2 (3)	C10—C11—C12—O2	-179.80 (17)
C8—N1—C7—C6	171.87 (17)	C10—C11—C12—C13	0.4 (3)
C1—C6—C7—O1	-174.8 (2)	O2—C12—C13—C14	-179.95 (18)
C5—C6—C7—O1	5.8 (3)	C11—C12—C13—C14	-0.2 (3)
C1—C6—C7—N1	6.1 (3)	C10—C9—C14—C13	-1.4 (3)
C5—C6—C7—N1	-173.29 (17)	N2—C9—C14—C13	179.44 (19)
C9—N2—C8—N1	-171.58 (17)	C12—C13—C14—C9	0.7 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2B \cdots O1	0.86	1.95	2.631 (2)	135
N1—H1B \cdots O2 ⁱ	0.86	2.29	3.109 (2)	158
O2—H2C \cdots S1 ⁱⁱ	0.82	2.53	3.1533 (18)	134
C1—H1A \cdots O2 ⁱ	0.93	2.51	3.429 (3)	172
C11—H11A \cdots O1 ⁱⁱⁱ	0.93	2.43	3.262 (2)	149

Symmetry codes: (i) $-x+5/2, -y, z+1/2$; (ii) $-x+5/2, -y, z-1/2$; (iii) $x+1/2, -y+1/2, -z+1$.

Fig. 1

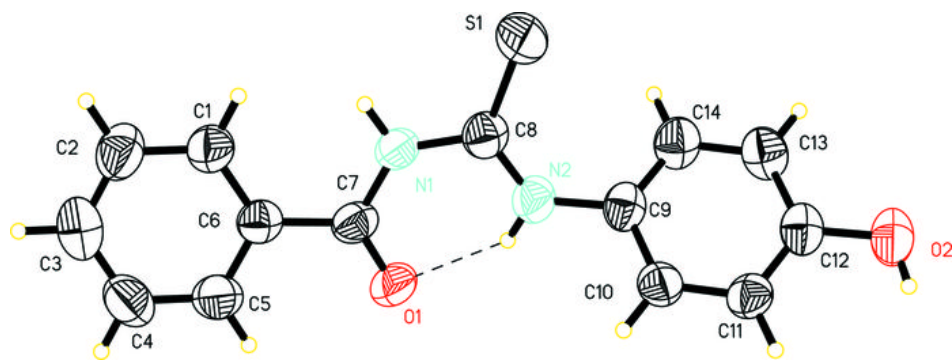


Fig. 2

