Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

5-Bromo-2-hydroxybenzaldehyde (4-phenyl-1,3-thiazol-2-yl)hydrazone

Genç Sadık,[a] Dege Necmi,[a]* Yılmaz Ibrahim,[b] Çukurovalı Alaaddin[b] and Muharrem Dinçer[a]

[a]Ondokuz Mayıs University, Arts and Sciences Faculty, Department of Physics, 55139 Samsun, Turkey, and [b]Fırat University, Faculty of Arts and Sciences, Department of Chemistry, 23119 Elazığ, Turkey

Correspondence e-mail: necmid@omu.edu.tr

Received 14 April 2004
Accepted 21 April 2004
Online 30 April 2004

Key indicators

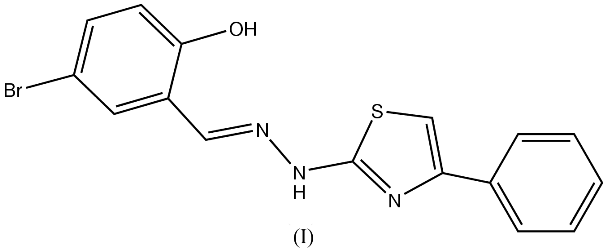
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.038
wR factor = 0.083
Data-to-parameter ratio = 13.3

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

In the title molecule, $C_{16}H_{12}BrN_3OS$, the thiazole ring makes an angle of 44.5 (2)° with the phenyl ring and an angle of 7.2 (2)° with the 5-bromosalicyaldehyde ring. The molecular structure is influenced by O—H···N and O—H···S intramolecular hydrogen bonds. In the crystal, the molecules pack as centrosymmetric dimers *via* intermolecular N—H···N hydrogen-bonds.

Comment

The chemistry of hydrazones has been intensively investigated in recent years, owing to their coordinating capability, pharmacological activity and antibacterial and antifungal properties, and their use in analytical chemistry as highly selective extractants (Domiano *et al.*, 1984; Sakamoto *et al.*, 1993; Li *et al.*, 1988). Many thiazole ring systems are of considerable importance because of their antibacterial and anti-inflammatory activity. Since a wide spectrum of activity is shown by the thiazole moiety, a large number of thiazoles substituted with different groups at various positions have been prepared (Giridhar *et al.*, 2001). We have now prepared the title thiazole-substituted hydrazone, (I), and present its crystal structure here.



(I)

A view of (I), with the atom-labelling scheme, is shown in Fig. 1. The molecule of (I) is non-planar. The mean plane through the thiazole ring makes an angle of 44.5 (2)° with the phenyl ring and an angle of 7.2 (2)° with the 5-bromosalicyaldehyde ring. The dihedral angle between the

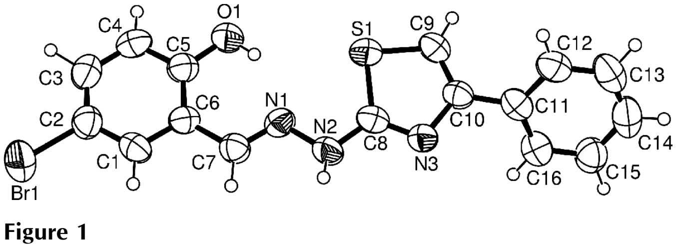


Figure 1
A drawing of (I), showing the atom-numbering scheme and with 50% probability displacement ellipsoids. H atoms are drawn as spheres of arbitrary radii.

DOI: 10.1107/S1600536804009705

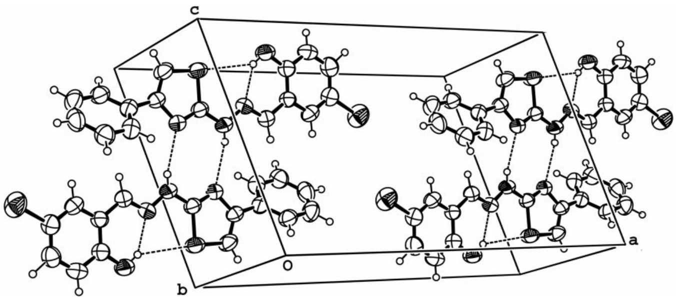


Figure 2
The crystal packing in (I), showing the N—H⋯N hydrogen-bonded dimers. (Hydrogen bonds are shown as dashed lines.)

5-bromosalicyaldehyde and phenyl rings is 51.6 (2)°.

Two types of intramolecular hydrogen bonds, O—H⋯N and O—H⋯S, are observed in the molecular structure of (I) (Table 2). These interactions constitute a pair of bifurcated-acceptor bonds generating an *S*(5) ring motif. In the crystal structure of (I), the molecules pack as centrosymmetric dimers *via* intermolecular N—H⋯N hydrogen-bonds (Table 2 and Fig. 2).

Experimental

To a suspension of 1-(2-hydroxy-5-bromobenzylidene)thiosemicarbazide (0.274 g, 1 mmol) in absolute ethanol, a solution of chloroacetophenone (0.199 g, 1 mmol) in ethanol was added gradually at *ca* 303–313 K with continuous stirring. After complete addition of the chloroacetophenone, the temperature of the mixture was raised to 323–328 K and kept at this value for 1 h. The mixture was cooled to room temperature and the solution was then made alkaline with an aqueous solution of NH_3 (5%). The precipitate which formed was filtered off, washed with aqueous NH_3 solution several times, dried in air and crystallized from aqueous ethanol (1:3) (yield 72%, m.p. 511 K). Analysis calculated for $C_{16}H_{12}BrN_3OS$ (374.26): C 51.35, H 3.23, N 11.23, S 8.57%; found: C 51.44, H 3.17, N 11.34, S 8.65%. Spectroscopic analysis: IR (KBr, ν_{max}, cm^{-1}): 3150 (—OH), 3105 (—NH—), 1619 (C═N), 1054 (C—O); ^1H NMR (DMSO-d_6, δ, p.p.m.): 6.89 (*s*, 1H, ═CH—, thiazole), 7.31–7.46 (*m*, 5H, aromatics), 7.76–7.89 (*m*, 3H, aromatics), 8.27 (*s*, 1H, azomethine), 10.42 (*s*, 1H, —NH—), 12.28 (*s*, 1H, —OH); ^{13}C NMR (DMSO-d_6, δ, p.p.m.): 124.53 (C_1), 130.28 (C_2), 112.60 (C_3), 134.39 (C_4), 120.21 (C_5), 156.81 (C_6), 138.86 (C_7), 169.66 (C_8), 105.37 (C_9), 152.38 (C_{10}), 136.34 (C_{11}), 127.21 (C_{12}), 129.37 (C_{13}), 129.49 (C_{14}).

Crystal data

$C_{16}H_{12}BrN_3OS$
$M_r = 374.26$
Monoclinic, $P2_1/c$
$a = 18.7025\ (15)$ Å
$b = 7.9183\ (8)$ Å
$c = 10.6063\ (12)$ Å
$\beta = 106.007\ (7)°$
$V = 1509.8\ (3)$ Å^3
$Z = 4$

$D_x = 1.646$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 4805 reflections
$\theta = 2.3–24.9°$
$\mu = 2.87$ mm^{-1}
$T = 293\ (2)$ K
Plate, colourless
0.20 × 0.15 × 0.04 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
$T_{min} = 0.535$, $T_{max} = 0.881$
15 222 measured reflections

2662 independent reflections
1179 reflections with $I > 2\sigma(I)$
$R_{int} = 0.134$
$\theta_{max} = 25.0°$
$h = -22 \rightarrow 22$
$k = -9 \rightarrow 9$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.083$
$S = 0.80$
2662 reflections
200 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0234P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0081 (6)

Table 1
Selected geometric parameters (Å, °).

Br1—C2	1.894 (4)	N1—N2	1.386 (4)
S1—C9	1.713 (4)	N2—C8	1.374 (5)
S1—C8	1.716 (4)	N3—C8	1.318 (5)
O1—C5	1.368 (5)	N3—C10	1.412 (5)
N1—C7	1.288 (5)	C10—C11	1.473 (6)
O1—C5—C6	123.2 (4)	C10—C9—S1	111.5 (3)
O1—C5—C4	115.9 (4)	C9—C10—N3	115.4 (4)
C5—C6—C7	123.1 (4)	C9—C10—C11	124.5 (4)
C1—C6—C7	118.0 (4)	N3—C10—C11	120.1 (4)
N1—C7—C6	119.2 (4)	C16—C11—C12	118.3 (4)
N3—C8—N2	123.3 (4)	C16—C11—C10	121.6 (4)
N3—C8—S1	116.8 (3)	C12—C11—C10	120.1 (5)
N2—C8—S1	119.9 (3)		
C7—N1—N2—C8	−176.8 (4)	N1—N2—C8—S1	2.2 (5)
N2—N1—C7—C6	−179.3 (4)	C9—C10—C11—C16	−132.7 (5)
C5—C6—C7—N1	1.9 (7)	N3—C10—C11—C16	44.1 (6)
C1—C6—C7—N1	−177.9 (4)	C9—C10—C11—C12	44.4 (6)
N1—N2—C8—N3	−178.5 (4)	N3—C10—C11—C12	−138.8 (4)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N2—H2*A*⋯N3^i	0.86	2.25	3.039 (5)	153
O1—H1*A*⋯S1	0.82	2.85	3.523 (3)	141
O1—H1*A*⋯N1	0.82	1.96	2.636 (4)	140

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

The reflections were very weak due to the thinness of the crystal. No further precaution was available to increase the intensities. The reflections were very weak due to the thinness of the crystal. H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms, with O—H, N—H and C—H distances of 0.82, 0.86 and 0.93 Å, respectively. The U_{iso}(H) values were set equal to $1.5U_{eq}$(O) for the hydroxyl H atom and $1.2U_{eq}$(parent atom) for the other H atoms.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *PLATON* (Spek, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

Domiano, P., Pelizzi, C. & Predieri, G. (1984). *Polyhedron*, **3**, 281–286.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Giridhar, T., Reddy, R. B., Prasanna, B. & Mouli, G. V. P. C. (2001). *Indian J. Chem. Soc. Sect. B*, **40**, 1279–1560.

Li, X. R., Sun, Z. M. & Chang, J. C. (1988). *Synth. React. Inorg. Met. Org. Chem.* **18**, 657–665.

Sakamoto, H., Goto, H., Yokoshima, M., Dobashi, M., Ishikawa, J., Doi, K. & Otomo, M. (1993). *Bull. Chem. Soc. Jpn*, **66**, 2907–2914.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Spek, A. L. (1997). *PLATON*. University of Utrecht, The Netherlands.

Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED*32 (Version 1.04). Stoe & Cie, Darmstadt, Germany.

Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

5-Bromo-2-hydroxybenzaldehyde (4-phenyl-1,3-thiazol-2-yl)hydrazone. Erratum

Sadık Genç,[a] Necmi Dege,[a]* IbrahimYılmaz Yılmaz,[b] Alaaddin Çukurovalı[b] and Muharrem Dinçer[a]

[a]Ondokuz Mayıs University, Arts and Sciences Faculty, Department of Physics, 55139 Samsun, Turkey, and [b]Fırat University, Faculty of Arts and Sciences, Department of Chemistry, 23119 Elazığ, Turkey

Correspondence e-mail: necmid@omu.edu.tr

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.038
wR factor = 0.083
Data-to-parameter ratio = 13.3

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

In the paper by Sadik [*Acta Cryst.* (2004), E**60**, o889–o891], the forenames and surnames of the first four authors are given in the incorrect order. The correct names are given opposite.

e10 Sadık Genç *et al.* • $C_{16}H_{12}BrN_3OS$

DOI: 10.1107/S1600536804011493