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## Structure Reports

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# Benzaldehyde thiosemicarbazone monohydrate

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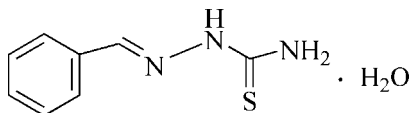
Received 2 July 2008; accepted 20 July 2008

 Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.046;  $wR$  factor = 0.106; data-to-parameter ratio = 14.9.

In the title compound,  $\text{C}_8\text{H}_9\text{N}_3\text{S}\cdot\text{H}_2\text{O}$ , intramolecular  $\text{N}-\text{H}\cdots\text{N}$  hydrogen bonding contributes to the molecular conformation. Water molecules are involved in intermolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{O}-\text{H}\cdots\text{S}$  hydrogen bonds, which link the molecules into ribbons extended along the  $a$  axis. Weak intermolecular  $\text{N}-\text{H}\cdots\text{S}$  hydrogen bonds link these ribbons into layers parallel to the  $ab$  plane with the phenyl rings pointing up and down.

## Related literature

For related crystal structures, see Beraldo *et al.* (2004); Bondock *et al.* (2007); Jing *et al.* (2006).



## Experimental

### Crystal data

 $\text{C}_8\text{H}_9\text{N}_3\text{S}\cdot\text{H}_2\text{O}$ 
 $M_r = 197.26$ 

 Orthorhombic,  $P2_12_12_1$ 
 $a = 6.1685$  (10) Å

 $b = 7.6733$  (12) Å

 $c = 21.131$  (2) Å

 $V = 1000.2$  (2) Å<sup>3</sup>
 $Z = 4$ 

 Mo  $K\alpha$  radiation

 $\mu = 0.29$  mm<sup>-1</sup>
 $T = 298$  (2) K

 $0.49 \times 0.30 \times 0.28$  mm

### Data collection

Bruker SMART CCD area-detector diffractometer

 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)

 $T_{\min} = 0.871$ ,  $T_{\max} = 0.923$ 

4749 measured reflections

1764 independent reflections

 1438 reflections with  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.065$ 

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.045$ 
 $wR(F^2) = 0.105$ 
 $S = 1.07$ 

1764 reflections

118 parameters

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.24$  e Å<sup>-3</sup>
 $\Delta\rho_{\min} = -0.17$  e Å<sup>-3</sup>

Absolute structure: Flack (1983),

689 Friedel pairs

 Flack parameter:  $-0.05$  (13)

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N3}-\text{H3A}\cdots\text{N1}$	0.86	2.26	2.613 (4)	105
$\text{N2}-\text{H2}\cdots\text{O1}^{\text{i}}$	0.86	1.95	2.805 (3)	171
$\text{N3}-\text{H3B}\cdots\text{S1}^{\text{ii}}$	0.86	2.57	3.423 (3)	170
$\text{O1}-\text{H1A}\cdots\text{S1}$	0.85	2.45	3.276 (2)	164
$\text{O1}-\text{H1B}\cdots\text{S1}^{\text{i}}$	0.85	2.44	3.284 (2)	172

 Symmetry codes: (i)  $x + \frac{1}{2}, -y + \frac{3}{2}, -z + 1$ ; (ii)  $x - \frac{1}{2}, -y + \frac{1}{2}, -z + 1$ .

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINTE* (Siemens, 1996); data reduction: *SAINTE*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2428).

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## supporting information

*Acta Cryst.* (2008). E64, o1597 [doi:10.1107/S1600536808022769]

**Benzaldehyde thiosemicarbazone monohydrate****Sheng-Jiu Gu and Kai-Mei Zhu****S1. Comment**

Aryl-hydrazones, such as semicarbazones, thiosemicarbazones and guanyl hydrazones, exhibit strong biological activity. Therefore, they are important for drug design (Beraldo *et al.*, 2004), organocatalysis and for the preparation of heterocyclic rings (Bondock *et al.*, 2007). In this paper, we present the title compound, (I).

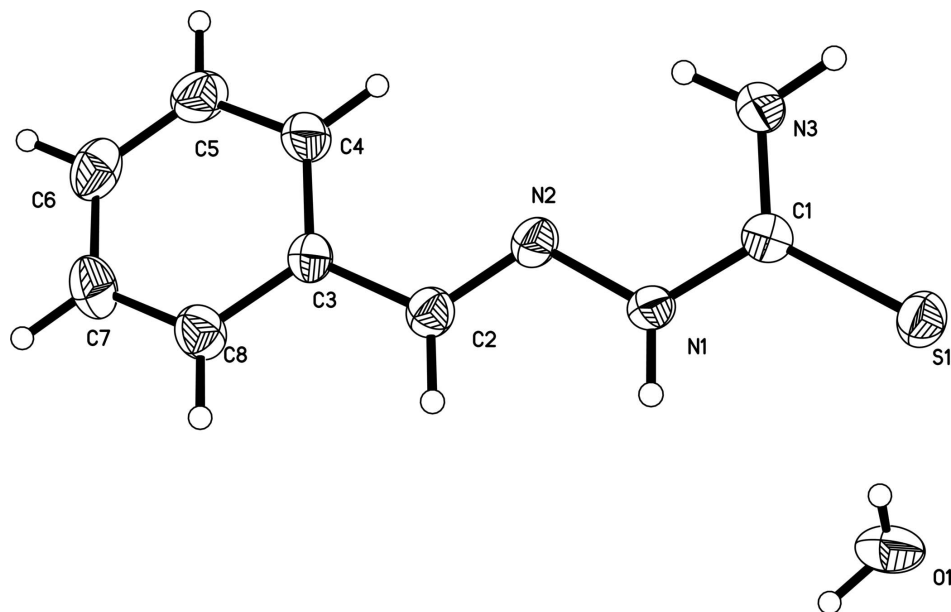
In (I) (Fig. 1), the bond lengths and angles are normal and comparable to those observed in the reported compounds (Jing *et al.*, 2006). Intramolecular N—H $\cdots$ O hydrogen bond (Table 1) contributes to the molecular conformation. Crystalline water molecules are involved in the intermolecular N—H $\cdots$ O and O—H $\cdots$ S hydrogen bonds (Table 1), which link the molecules into ribbons extended along *a* axis. Weak intermolecular N—H $\cdots$ S hydrogen bonds (Table 1) link further these ribbons into layers parallel to *ab* plane with the up and down protruding phenyl rings.

**S2. Experimental**

Benzaldehyde (0.3 mmol) and thiosemicarbazide (0.3 mmol) were mixed in 50 ml flask in the presence of aqueous medium. After stirring 30 min at 373 K, the mixture then cooling slowly to room temperature and affording the title compound, then recrystallized from ethanol, affording the title compound as a colorless crystalline solid. Elemental analysis: calculated for C<sub>8</sub>H<sub>11</sub>N<sub>3</sub>OS: C 48.71, H 5.62, N 21.30%; found: C 48.58, H 5.65, N 21.24%.

**S3. Refinement**

All H atoms were placed in geometrically idealized positions (N—H 0.86, O—H 0.85 and C—H 0.93 Å) and treated as riding on their parent atoms, with  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$  (C, O, N).

**Figure 1**

The content of asymmetric unit of the title compound showing the atomic numbering scheme and 30% probability displacement ellipsoids.

### Benzaldehyde thiosemicarbazone monohydrate

#### Crystal data

$C_8H_9N_3S \cdot H_2O$

$M_r = 197.26$

Orthorhombic,  $P2_12_12_1$

$a = 6.1685$  (10) Å

$b = 7.6733$  (12) Å

$c = 21.131$  (2) Å

$V = 1000.2$  (2) Å<sup>3</sup>

$Z = 4$

$F(000) = 416$

$D_x = 1.310$  Mg m<sup>-3</sup>

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

Cell parameters from 1572 reflections

$\theta = 2.8$ – $22.5^\circ$

$\mu = 0.29$  mm<sup>-1</sup>

$T = 298$  K

Block, orange

$0.49 \times 0.30 \times 0.28$  mm

#### Data collection

Bruker SMART CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.871$ ,  $T_{\max} = 0.924$

4749 measured reflections

1764 independent reflections

1438 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.065$

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

$h = -7 \rightarrow 7$

$k = -9 \rightarrow 6$

$l = -25 \rightarrow 24$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.105$

$S = 1.08$

1764 reflections

118 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0438P)^2 + 0.0825P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$   
 Absolute structure: Flack (1983), 689 Friedel pairs  
 Absolute structure parameter:  $-0.05$  (13)

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.5077 (4)	0.4006 (3)	0.35842 (11)	0.0430 (6)
N2	0.4610 (4)	0.4442 (3)	0.42017 (10)	0.0402 (6)
H2	0.5473	0.5107	0.4412	0.048*
N3	0.1572 (5)	0.2820 (4)	0.41293 (12)	0.0616 (9)
H3A	0.1923	0.2576	0.3746	0.074*
H3B	0.0400	0.2401	0.4289	0.074*
O1	0.2807 (3)	0.8582 (3)	0.51847 (12)	0.0681 (7)
H1A	0.2874	0.7483	0.5138	0.082*
H1B	0.4026	0.9030	0.5091	0.082*
S1	0.22368 (12)	0.43397 (10)	0.52322 (3)	0.0473 (3)
C1	0.2821 (5)	0.3831 (3)	0.44708 (14)	0.0398 (7)
C2	0.6891 (5)	0.4535 (4)	0.33743 (13)	0.0436 (7)
H2A	0.7840	0.5112	0.3645	0.052*
C3	0.7511 (4)	0.4251 (4)	0.27170 (12)	0.0412 (7)
C4	0.6111 (6)	0.3517 (4)	0.22810 (15)	0.0534 (9)
H4	0.4745	0.3146	0.2409	0.064*
C5	0.6728 (7)	0.3333 (5)	0.16566 (16)	0.0640 (11)
H5	0.5766	0.2870	0.1362	0.077*
C6	0.8751 (7)	0.3834 (5)	0.14733 (17)	0.0636 (11)
H6	0.9180	0.3671	0.1056	0.076*
C7	1.0158 (6)	0.4572 (5)	0.18932 (16)	0.0630 (10)
H7	1.1523	0.4940	0.1762	0.076*
C8	0.9527 (5)	0.4761 (4)	0.25132 (15)	0.0536 (9)
H8	1.0490	0.5248	0.2802	0.064*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0480 (15)	0.0477 (15)	0.0332 (13)	0.0000 (13)	0.0012 (12)	-0.0020 (11)
N2	0.0443 (14)	0.0422 (14)	0.0342 (12)	-0.0074 (14)	-0.0004 (11)	-0.0034 (12)

N3	0.061 (2)	0.079 (2)	0.0456 (17)	-0.0290 (17)	0.0118 (14)	-0.0121 (15)
O1	0.0524 (15)	0.0609 (13)	0.0910 (19)	0.0017 (12)	0.0204 (15)	-0.0130 (13)
S1	0.0467 (5)	0.0576 (5)	0.0376 (4)	0.0009 (4)	0.0015 (4)	-0.0018 (4)
C1	0.0402 (17)	0.0381 (16)	0.0411 (16)	0.0008 (15)	-0.0035 (15)	0.0036 (12)
C2	0.0415 (17)	0.0469 (17)	0.0422 (16)	0.0035 (17)	-0.0009 (14)	-0.0018 (14)
C3	0.0421 (17)	0.0433 (14)	0.0383 (15)	0.0013 (18)	0.0036 (14)	0.0015 (14)
C4	0.056 (2)	0.061 (2)	0.0429 (19)	-0.0134 (17)	0.0061 (17)	-0.0012 (17)
C5	0.083 (3)	0.065 (2)	0.044 (2)	-0.013 (2)	0.0033 (19)	-0.0067 (18)
C6	0.086 (3)	0.061 (2)	0.043 (2)	0.005 (2)	0.017 (2)	0.0038 (18)
C7	0.053 (2)	0.080 (3)	0.057 (2)	-0.002 (2)	0.0154 (18)	0.010 (2)
C8	0.050 (2)	0.066 (2)	0.0446 (17)	-0.0061 (17)	0.0023 (16)	0.0054 (16)

*Geometric parameters (Å, °)*

N1—C2	1.270 (3)	C3—C8	1.373 (4)
N1—N2	1.378 (3)	C3—C4	1.382 (4)
N2—C1	1.327 (3)	C4—C5	1.380 (4)
N2—H2	0.8600	C4—H4	0.9300
N3—C1	1.310 (4)	C5—C6	1.362 (5)
N3—H3A	0.8600	C5—H5	0.9300
N3—H3B	0.8600	C6—C7	1.364 (5)
O1—H1A	0.8499	C6—H6	0.9300
O1—H1B	0.8499	C7—C8	1.375 (5)
S1—C1	1.695 (3)	C7—H7	0.9300
C2—C3	1.457 (4)	C8—H8	0.9300
C2—H2A	0.9300		
C2—N1—N2	115.9 (3)	C4—C3—C2	122.2 (3)
C1—N2—N1	119.6 (2)	C5—C4—C3	120.4 (3)
C1—N2—H2	120.2	C5—C4—H4	119.8
N1—N2—H2	120.2	C3—C4—H4	119.8
C1—N3—H3A	120.0	C6—C5—C4	119.7 (4)
C1—N3—H3B	120.0	C6—C5—H5	120.2
H3A—N3—H3B	120.0	C4—C5—H5	120.2
H1A—O1—H1B	109.4	C5—C6—C7	121.0 (3)
N3—C1—N2	117.6 (3)	C5—C6—H6	119.5
N3—C1—S1	122.3 (2)	C7—C6—H6	119.5
N2—C1—S1	120.1 (2)	C6—C7—C8	118.9 (3)
N1—C2—C3	121.1 (3)	C6—C7—H7	120.5
N1—C2—H2A	119.5	C8—C7—H7	120.5
C3—C2—H2A	119.5	C3—C8—C7	121.7 (3)
C8—C3—C4	118.2 (3)	C3—C8—H8	119.2
C8—C3—C2	119.6 (3)	C7—C8—H8	119.2

*Hydrogen-bond geometry (Å, °)*

<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>
N3—H3A $\cdots$ N1	0.86	2.26	2.613 (4)	105

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N2—H2···O1 <sup>i</sup>	0.86	1.95	2.805 (3)	171
N3—H3B···S1 <sup>ii</sup>	0.86	2.57	3.423 (3)	170
O1—H1A···S1	0.85	2.45	3.276 (2)	164
O1—H1B···S1 <sup>i</sup>	0.85	2.44	3.284 (2)	172

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Symmetry codes: (i)  $x+1/2, -y+3/2, -z+1$ ; (ii)  $x-1/2, -y+1/2, -z+1$ .