

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Benzaldehyde thiosemicarbazone monohydrate

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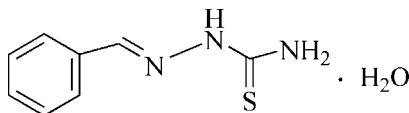
Received 2 July 2008; accepted 20 July 2008

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) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.29$ mm⁻¹ $T = 298$ (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

 $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.17$ e Å⁻³

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: SAINT (Siemens, 1996); 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.

The authors thank the Nature Science Foundation of Guangxi (No. 0640190 and No. 0728229), the Tackle Key Problem Foundation of Guangxi (No. 0815005-1-17), the Nature Science Foundation of Guilin (No. 20070305 and No. 20080103-5) and the Education Foundation of Guangxi (No. 200710MS144) for financial support.

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

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supplementary materials

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

Benzaldehyde thiosemicarbazone monohydrate

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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.

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 C₈H₁₁N₃OS: C 48.71, H 5.62, N 21.30%; found: C 48.58, H 5.65, N 21.24%.

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).

Figures

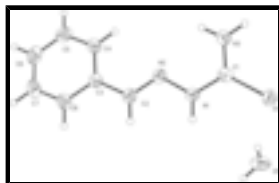


Fig. 1. The content of asymmetric unit of the title compound showing the atomic numbering scheme and 30% probability displacement ellipsoids.

Benzaldehyde thiosemicarbazone monohydrate

Crystal data

C₈H₉N₃S·H₂O

$M_r = 197.26$

$D_x = 1.310 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

supplementary materials

Orthorhombic, $P2_12_12_1$	$\lambda = 0.71073 \text{ \AA}$
$a = 6.1685 (10) \text{ \AA}$	Cell parameters from 1572 reflections
$b = 7.6733 (12) \text{ \AA}$	$\theta = 2.8\text{--}22.5^\circ$
$c = 21.131 (2) \text{ \AA}$	$\mu = 0.29 \text{ mm}^{-1}$
$V = 1000.2 (2) \text{ \AA}^3$	$T = 298 (2) \text{ K}$
$Z = 4$	Block, orange
$F_{000} = 416$	$0.49 \times 0.30 \times 0.28 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer	1764 independent reflections
Radiation source: fine-focus sealed tube	1438 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.065$
$T = 298(2) \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.9^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -7 \rightarrow 7$
$T_{\text{min}} = 0.871, T_{\text{max}} = 0.924$	$k = -9 \rightarrow 6$
4749 measured reflections	$l = -25 \rightarrow 24$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.045$	$w = 1/[\sigma^2(F_o^2) + (0.0438P)^2 + 0.0825P]$
$wR(F^2) = 0.105$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1764 reflections	$\Delta\rho_{\text{max}} = 0.25 \text{ e \AA}^{-3}$
118 parameters	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none
Secondary atom site location: difference Fourier map	Absolute structure: Flack (1983), 689 Friedel pairs
	Flack 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (\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 (\AA , $^\circ$)

N1—C2	1.270 (3)	C3—C8	1.373 (4)
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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 (\AA , $^\circ$)

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

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

Fig. 1

