

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2-Hydroxy-4-methoxybenzaldehyde thiosemicarbazone

Yu-Mei Hao

Department of Chemistry, Baicheng Normal University, Baicheng 137000, People's Republic of China

Correspondence e-mail: jyxygzb@163.com

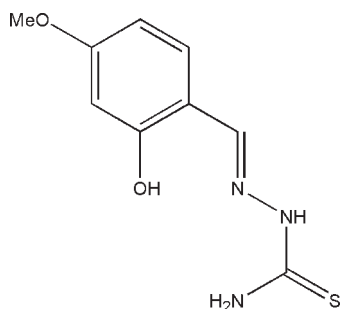
Received 24 July 2010; accepted 25 July 2010

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.040; wR factor = 0.101; data-to-parameter ratio = 15.3.

The title Schiff base compound, $\text{C}_9\text{H}_{11}\text{N}_3\text{O}_2\text{S}$, was prepared by the reaction of equimolar quantities of 2-hydroxy-4-methoxybenzaldehyde with thiosemicarbazide in methanol. The molecule adopts a *trans* configuration with respect to the azomethine group and an intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond generates an $S(6)$ ring. In the crystal structure, molecules are linked through intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds, forming a three-dimensional network.

Related literature

For a related structure and background references, see: Hao (2010). For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

 $\text{C}_9\text{H}_{11}\text{N}_3\text{O}_2\text{S}$ $M_r = 225.27$

Monoclinic, $P2_1/n$
 $a = 4.929$ (1) Å
 $b = 10.519$ (2) Å
 $c = 20.357$ (3) Å
 $\beta = 92.838$ (2)°
 $V = 1054.2$ (3) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.29$ mm⁻¹
 $T = 298$ K
 $0.17 \times 0.13 \times 0.12$ mm

Data collection

Bruker SMART CCD
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.952$, $T_{\max} = 0.966$

5879 measured reflections
 2247 independent reflections
 1650 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.101$
 $S = 1.04$
 2247 reflections
 147 parameters
 4 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.18$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.23$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N3}-\text{H3B}\cdots\text{O2}^{\text{i}}$	0.89 (1)	2.26 (2)	2.998 (3)	141 (2)
$\text{N3}-\text{H3A}\cdots\text{O1}^{\text{ii}}$	0.88 (1)	2.23 (1)	3.076 (3)	162 (2)
$\text{N2}-\text{H2}\cdots\text{S1}^{\text{iii}}$	0.90 (1)	2.48 (1)	3.366 (3)	168 (2)
$\text{O1}-\text{H1}\cdots\text{N1}$	0.82	1.99	2.700 (2)	145

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

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); 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: SHELXL97.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5575).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
 Bruker (2002). SAINT and SMART. Bruker AXS Inc., Madison, Wisconsin, USA.
 Hao, Y.-M. (2010). *Acta Cryst. E* **66**, o1177.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.

supplementary materials

Acta Cryst. (2010). E66, o2211 [doi:10.1107/S1600536810029594]

2-Hydroxy-4-methoxybenzaldehyde thiosemicarbazone

Y.-M. Hao

Comment

As a continuation of our structural studies of Schiff bases (Hao, 2010), in this paper, the title new Schiff base compound, (I), Fig. 1, is reported.

The molecule of the title compound adopts a *trans* configuration with respect to the azomethine group. All the bond lengths are within normal values (Allen *et al.*, 1987). There is an intramolecular O—H \cdots N hydrogen bond (Table 1) in the molecule. In the crystal structure, molecules are linked through intermolecular N—H \cdots O and N—H \cdots S hydrogen bonds (Table 1), forming a 3D network (Fig. 2).

Experimental

2-Hydroxy-4-methoxybenzaldehyde (0.1 mmol, 15.2 mg) and thiosemicarbazide (0.1 mmol, 9.1 mg) were refluxed in a 30 ml methanol solution for 30 min to give a clear colorless solution. Colorless blocks of (I) were formed by slow evaporation of the solvent over several days at room temperature.

Refinement

H2, H3A and H3B were located from a difference Fourier map and refined isotropically, with the N—H and H \cdots H distances restrained to 0.90 (1) Å and 1.53 (2) Å, respectively, and with U_{iso} restrained to 0.08 Å². Other H atoms were constrained to ideal geometries, with $d(\text{C—H}) = 0.93\text{--}0.96\text{Å}$, $d(\text{O—H}) = 0.82\text{Å}$, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{O1 and C7})$.

Figures

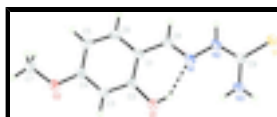


Fig. 1. The molecular structure of the title compound with 30% probability ellipsoids. Intra-molecular hydrogen bond is drawn as a dashed line.

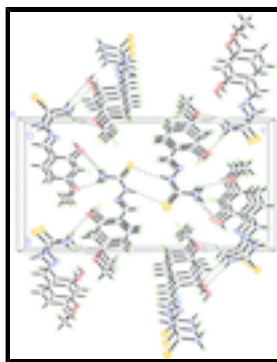


Fig. 2. Molecular packing of the title compound with hydrogen bonds drawn as dashed lines.

2-Hydroxy-4-methoxybenzaldehyde thiosemicarbazone

Crystal data

$C_9H_{11}N_3O_2S$	$F(000) = 472$
$M_r = 225.27$	$D_x = 1.420 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2yn	Cell parameters from 1566 reflections
$a = 4.929 (1) \text{ \AA}$	$\theta = 2.8\text{--}26.2^\circ$
$b = 10.519 (2) \text{ \AA}$	$\mu = 0.29 \text{ mm}^{-1}$
$c = 20.357 (3) \text{ \AA}$	$T = 298 \text{ K}$
$\beta = 92.838 (2)^\circ$	Block, colorless
$V = 1054.2 (3) \text{ \AA}^3$	$0.17 \times 0.13 \times 0.12 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART CCD diffractometer	2247 independent reflections
Radiation source: fine-focus sealed tube graphite	1650 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.030$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$\theta_{\text{max}} = 26.9^\circ$, $\theta_{\text{min}} = 2.0^\circ$
$T_{\text{min}} = 0.952$, $T_{\text{max}} = 0.966$	$h = -6 \rightarrow 6$
5879 measured reflections	$k = -13 \rightarrow 11$
	$l = -20 \rightarrow 25$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.040$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.101$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.1092P]$
2247 reflections	where $P = (F_o^2 + 2F_c^2)/3$
147 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
4 restraints	$\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds

in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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	0.6185 (3)	0.11719 (14)	0.11874 (8)	0.0391 (4)
N2	0.7882 (3)	0.04027 (15)	0.08411 (7)	0.0420 (4)
N3	0.9140 (4)	-0.06702 (18)	0.17762 (8)	0.0528 (5)
O1	0.4001 (3)	0.22381 (13)	0.22458 (6)	0.0520 (4)
H1	0.4909	0.1714	0.2055	0.078*
O2	-0.2624 (3)	0.53431 (13)	0.19151 (6)	0.0483 (4)
S1	1.12828 (11)	-0.14847 (5)	0.06920 (2)	0.0530 (2)
C1	0.2729 (4)	0.27756 (17)	0.11180 (9)	0.0370 (4)
C2	0.2464 (4)	0.29172 (18)	0.17967 (9)	0.0371 (4)
C3	0.0662 (4)	0.37755 (18)	0.20376 (9)	0.0419 (5)
H3	0.0521	0.3855	0.2490	0.050*
C4	-0.0943 (4)	0.45216 (17)	0.16165 (9)	0.0381 (4)
C5	-0.0757 (4)	0.43980 (18)	0.09409 (9)	0.0425 (5)
H5	-0.1835	0.4891	0.0652	0.051*
C6	0.1059 (4)	0.35287 (19)	0.07061 (9)	0.0445 (5)
H6	0.1171	0.3443	0.0254	0.053*
C7	-0.4289 (4)	0.61772 (19)	0.15119 (11)	0.0536 (6)
H7A	-0.3154	0.6726	0.1267	0.080*
H7B	-0.5402	0.6681	0.1785	0.080*
H7C	-0.5429	0.5684	0.1213	0.080*
C8	0.4607 (4)	0.18930 (18)	0.08365 (9)	0.0420 (5)
H8	0.4667	0.1849	0.0381	0.050*
C9	0.9333 (4)	-0.05273 (18)	0.11353 (9)	0.0382 (4)
H2	0.799 (5)	0.058 (2)	0.0411 (5)	0.080*
H3A	0.998 (4)	-0.1287 (16)	0.1999 (10)	0.080*
H3B	0.830 (4)	-0.0115 (18)	0.2022 (10)	0.080*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0443 (9)	0.0365 (9)	0.0377 (9)	-0.0007 (7)	0.0136 (7)	-0.0049 (7)
N2	0.0523 (10)	0.0419 (9)	0.0331 (9)	0.0090 (7)	0.0157 (8)	-0.0006 (7)
N3	0.0661 (12)	0.0616 (12)	0.0315 (9)	0.0130 (9)	0.0107 (8)	0.0032 (8)
O1	0.0617 (9)	0.0581 (9)	0.0365 (8)	0.0216 (7)	0.0051 (6)	-0.0001 (6)
O2	0.0500 (8)	0.0493 (8)	0.0463 (8)	0.0142 (6)	0.0110 (7)	-0.0011 (6)
S1	0.0681 (4)	0.0523 (3)	0.0400 (3)	0.0193 (3)	0.0167 (3)	0.0030 (2)
C1	0.0411 (10)	0.0370 (10)	0.0336 (10)	-0.0025 (8)	0.0084 (8)	-0.0039 (8)

supplementary materials

C2	0.0380 (10)	0.0402 (10)	0.0333 (10)	-0.0004 (8)	0.0049 (8)	-0.0005 (8)
C3	0.0472 (11)	0.0478 (12)	0.0314 (10)	0.0040 (9)	0.0085 (9)	-0.0041 (8)
C4	0.0367 (10)	0.0380 (10)	0.0404 (11)	-0.0010 (8)	0.0085 (8)	-0.0007 (8)
C5	0.0460 (11)	0.0430 (11)	0.0386 (11)	0.0049 (9)	0.0025 (9)	0.0045 (9)
C6	0.0545 (12)	0.0485 (12)	0.0311 (10)	0.0008 (10)	0.0072 (9)	-0.0017 (8)
C7	0.0537 (13)	0.0443 (12)	0.0631 (14)	0.0107 (10)	0.0073 (11)	0.0033 (10)
C8	0.0510 (12)	0.0427 (11)	0.0332 (10)	-0.0005 (9)	0.0114 (9)	-0.0039 (8)
C9	0.0416 (11)	0.0401 (11)	0.0333 (10)	-0.0046 (8)	0.0077 (8)	-0.0021 (8)

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

N1—C8	1.279 (2)	C1—C2	1.402 (3)
N1—N2	1.382 (2)	C1—C8	1.449 (3)
N2—C9	1.336 (2)	C2—C3	1.374 (3)
N2—H2	0.899 (10)	C3—C4	1.382 (3)
N3—C9	1.321 (2)	C3—H3	0.9300
N3—H3A	0.882 (9)	C4—C5	1.389 (3)
N3—H3B	0.886 (9)	C5—C6	1.381 (3)
O1—C2	1.361 (2)	C5—H5	0.9300
O1—H1	0.8200	C6—H6	0.9300
O2—C4	1.361 (2)	C7—H7A	0.9600
O2—C7	1.432 (2)	C7—H7B	0.9600
S1—C9	1.685 (2)	C7—H7C	0.9600
C1—C6	1.393 (3)	C8—H8	0.9300
C8—N1—N2	115.42 (16)	C3—C4—C5	119.83 (17)
C9—N2—N1	121.62 (16)	C6—C5—C4	118.70 (17)
C9—N2—H2	122.0 (15)	C6—C5—H5	120.7
N1—N2—H2	116.4 (15)	C4—C5—H5	120.7
C9—N3—H3A	122.4 (15)	C5—C6—C1	122.82 (18)
C9—N3—H3B	122.7 (15)	C5—C6—H6	118.6
H3A—N3—H3B	114.6 (18)	C1—C6—H6	118.6
C2—O1—H1	109.5	O2—C7—H7A	109.5
C4—O2—C7	118.50 (16)	O2—C7—H7B	109.5
C6—C1—C2	116.84 (17)	H7A—C7—H7B	109.5
C6—C1—C8	119.77 (17)	O2—C7—H7C	109.5
C2—C1—C8	123.39 (17)	H7A—C7—H7C	109.5
O1—C2—C3	116.95 (16)	H7B—C7—H7C	109.5
O1—C2—C1	122.03 (16)	N1—C8—C1	122.81 (18)
C3—C2—C1	121.00 (17)	N1—C8—H8	118.6
C2—C3—C4	120.80 (17)	C1—C8—H8	118.6
C2—C3—H3	119.6	N3—C9—N2	117.57 (17)
C4—C3—H3	119.6	N3—C9—S1	122.11 (15)
O2—C4—C3	115.21 (17)	N2—C9—S1	120.31 (14)
O2—C4—C5	124.97 (17)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H3B \cdots O2 ⁱ	0.89 (1)	2.26 (2)	2.998 (3)	141.(2)

N3—H3A···O1 ⁱⁱ	0.88 (1)	2.23 (1)	3.076 (3)	162 (2)
N2—H2···S1 ⁱⁱⁱ	0.90 (1)	2.48 (1)	3.366 (3)	168 (2)
O1—H1···N1	0.82	1.99	2.700 (2)	145

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

Fig. 1

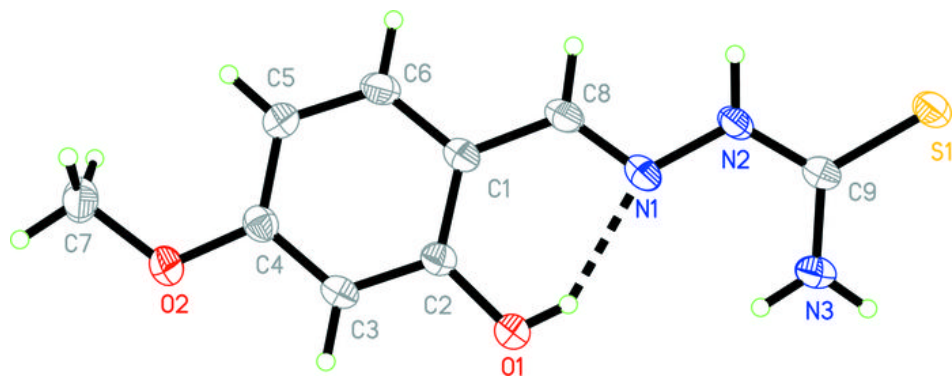


Fig. 2

