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2-Hydroxy-4-methoxybenzaldehyde thiosemicarbazone

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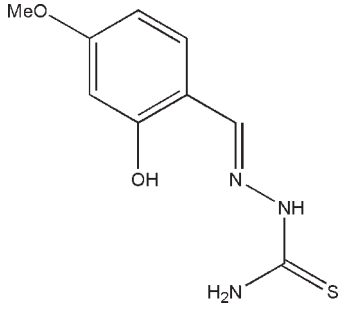
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.101; data-to-parameter ratio = 15.3.

The title Schiff base compound, $C_9H_{11}N_3O_2S$, 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 O—H···N hydrogen bond generates an S(6) ring. In the crystal structure, molecules are linked through intermolecular N—H···O and N—H···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

$C_9H_{11}N_3O_2S$ M_r = 225.27
Monoclinic, $P2_1/n$
a = 4.929 (1) Å
b = 10.519 (2) Å
c = 20.357 (3) Å
β = 92.838 (2)°
V = 1054.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 298 K
0.17 × 0.13 × 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_{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
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3B···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) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (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.
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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.

supporting information

Acta Cryst. (2010). E**66**, o2211 [https://doi.org/10.1107/S1600536810029594]

2-Hydroxy-4-methoxybenzaldehyde thiosemicarbazone

Yu-Mei Hao

S1. 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···N hydrogen bond (Table 1) in the molecule. In the crystal structure, molecules are linked through intermolecular N—H···O and N—H···S hydrogen bonds (Table 1), forming a 3D network (Fig. 2).

S2. 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.

S3. Refinement

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

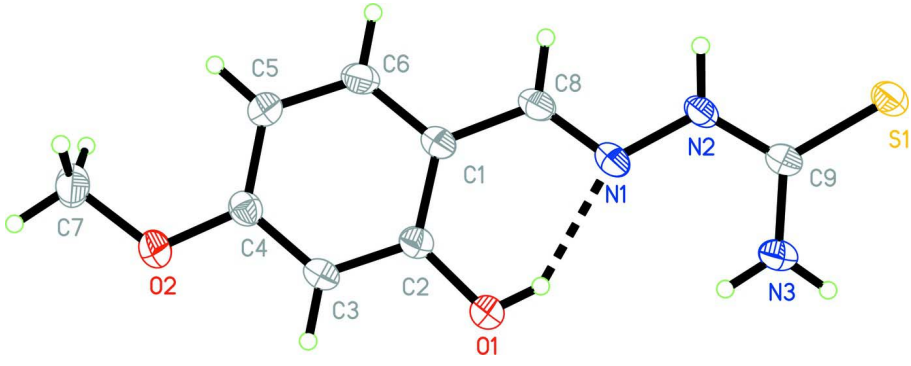

Figure 1

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

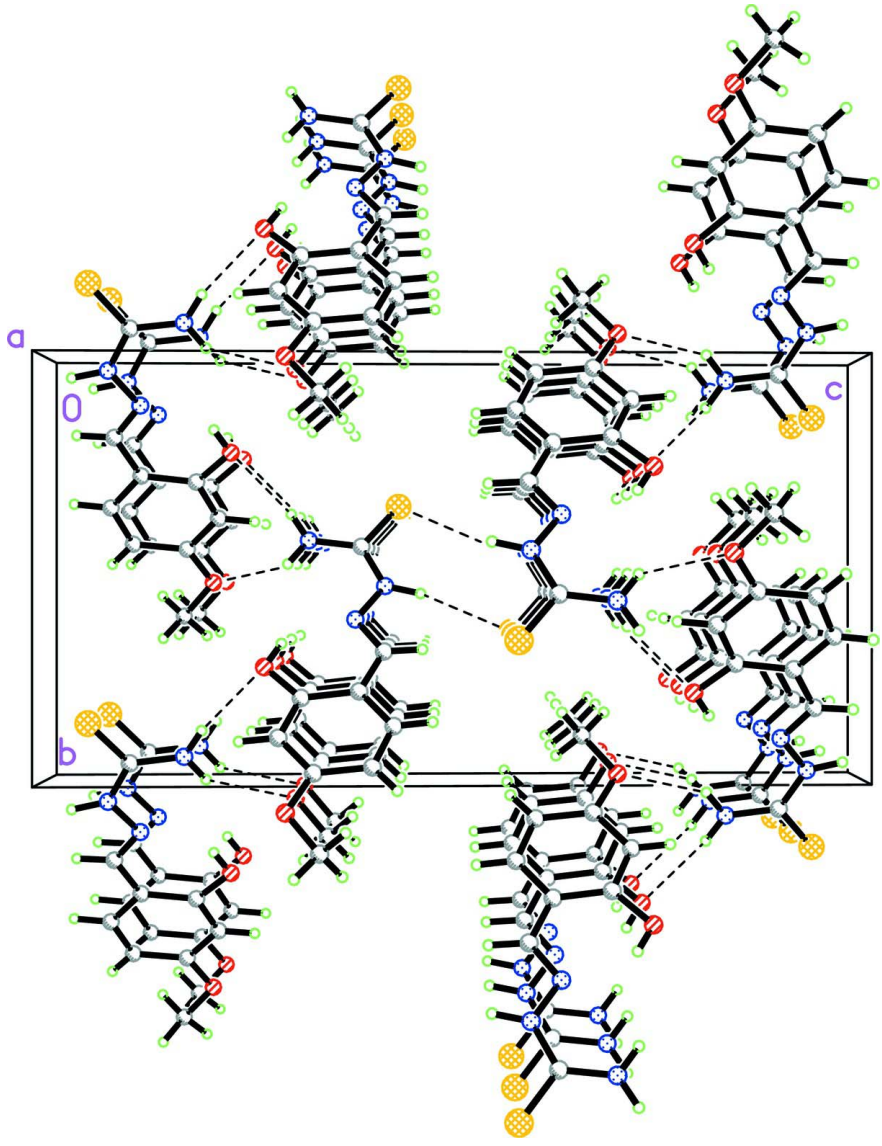

Figure 2

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

2-Hydroxy-4-methoxybenzaldehyde thiosemicarbazone

Crystal data

$C_9H_{11}N_3O_2S$
$M_r = 225.27$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 4.929\ (1)$ Å
$b = 10.519\ (2)$ Å
$c = 20.357\ (3)$ Å
$\beta = 92.838\ (2)°$
$V = 1054.2\ (3)$ Å^3
$Z = 4$

$F(000) = 472$
$D_x = 1.420$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1566 reflections
$\theta = 2.8–26.2°$
$\mu = 0.29$ mm^{-1}
$T = 298$ K
Block, colorless
0.17 × 0.13 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
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_{int} = 0.030
θ_{max} = 26.9°, θ_{min} = 2.0°
h = −6→6
k = −13→11
l = −20→25

Refinement

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

	x	*y*	*z*	U_{iso}*/U_{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 (Å²)

	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)
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 (Å, º)

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 (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*B*···O2^{i}	0.89 (1)	2.26 (2)	2.998 (3)	141 (2)
N3—H3*A*···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*.