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## Structure Reports

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## 2-Hydroxy-5-nitrobenzaldehyde thiosemicarbazone

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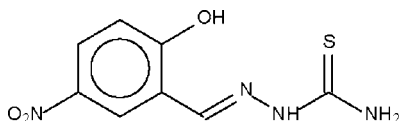
Received 10 July 2008; accepted 22 July 2008

Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  
R factor = 0.039;  $wR$  factor = 0.111; data-to-parameter ratio = 14.0.

The molecule of the title compound,  $\text{C}_8\text{H}_8\text{N}_4\text{O}_3\text{S}$ , is planar. Adjacent molecules are linked through  $\text{O}-\text{H}\cdots\text{S}$ ,  $\text{N}-\text{H}\cdots\text{S}$  and  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds into a three-dimensional network.

## Related literature

For the structure of 2-hydroxybenzaldehyde thiosemicarbazone, see: Chattopadhyay *et al.* (1988).



## Experimental

## Crystal data

$\text{C}_8\text{H}_8\text{N}_4\text{O}_3\text{S}$   
 $M_r = 240.24$   
Monoclinic,  $P2_1/n$   
 $a = 12.6157$  (3) Å  
 $b = 5.4815$  (2) Å  
 $c = 14.2397$  (2) Å  
 $\beta = 94.039$  (2)°

$V = 982.27$  (5) Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.33$  mm<sup>-1</sup>  
 $T = 100$  (2) K  
 $0.49 \times 0.01 \times 0.01$  mm

## Data collection

Bruker SMART APEX  
diffractometer  
Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.856$ ,  $T_{\max} = 0.997$

9462 measured reflections  
2247 independent reflections  
1725 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.037$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.111$   
 $S = 1.04$   
2247 reflections  
161 parameters  
8 restraints

H atoms treated by a mixture of  
independent and constrained  
refinement  
 $\Delta\rho_{\text{max}} = 0.47$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.23$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{S1}^{\text{i}}$	0.84 (1)	2.34 (1)	3.175 (2)	170 (3)
$\text{N3}-\text{H31}\cdots\text{S1}^{\text{ii}}$	0.85 (1)	2.50 (1)	3.337 (2)	167 (2)
$\text{N4}-\text{H41}\cdots\text{O2}^{\text{iii}}$	0.85 (1)	2.14 (1)	2.987 (2)	172 (3)
$\text{N4}-\text{H42}\cdots\text{O3}^{\text{iv}}$	0.85 (1)	2.31 (2)	3.044 (2)	144 (2)

Symmetry codes: (i)  $x + \frac{1}{2}, -y + \frac{5}{2}, z + \frac{1}{2}$ ; (ii)  $-x + 1, -y + 3, -z + 1$ ; (iii)  $-x + \frac{3}{2}, y + \frac{3}{2}, -z + \frac{1}{2}$ ; (iv)  $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$ .

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2102).

## References

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## supporting information

*Acta Cryst.* (2008). E64, o1606 [doi:10.1107/S1600536808022976]

**2-Hydroxy-5-nitrobenzaldehyde thiosemicarbazone**

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**S1. Comment**

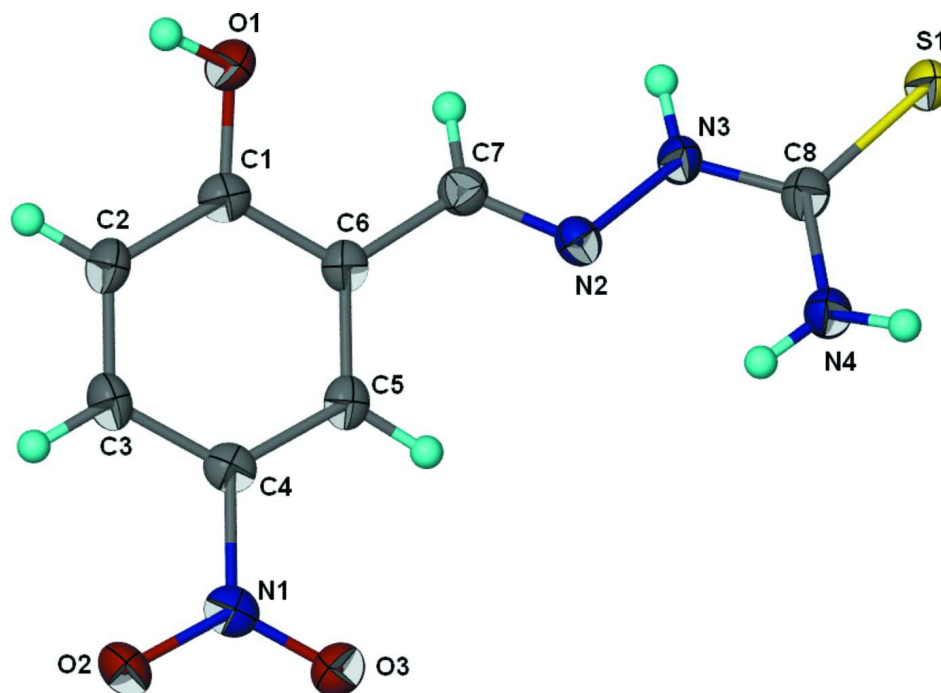
Salicylaldehyde thiosemicarbazone uses its 2-hydroxy group to form an intramolecular hydrogen bond (Chattopadhyay *et al.*, 1988). In the present compound, the electron-withdrawing nitro group that is *para* to the hydroxy group renders the hydroxy group much more acidic, so that the compound (Scheme) is able to use the hydroxy group to form a hydrogen bond to an adjacent molecule instead (Fig. 1).

**S2. Experimental**

The Schiff base was prepared by refluxing thiosemicarbazide (0.30 g, 3.3 mmol) and 5-nitro-2-hydroxybenzaldehyde (0.55 g, 3.3 mmol) in ethanol for 2 h. The product was recrystallized from ethanol.

**S3. Refinement**

Nitrogen- and oxygen-bound hydrogen atoms were refined with a distance restraint of N—H and O—H 0.85 (1) Å. Their temperature factors were freely refined. The carbon-bound ones were placed in geometric positions with C—H = 0.95 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

**Figure 1**

Molecular structure of title compound with the atom numbering scheme (Barbour, 2001). Displacement ellipsoids are drawn at 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

## 2-Hydroxy-5-nitrobenzaldehyde thiosemicarbazone

### Crystal data

$C_8H_8N_4O_3S$

$M_r = 240.24$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P 2_1n$

$a = 12.6157(3) \text{ \AA}$

$b = 5.4815(2) \text{ \AA}$

$c = 14.2397(2) \text{ \AA}$

$\beta = 94.039(2)^\circ$

$V = 982.27(5) \text{ \AA}^3$

$Z = 4$

$F(000) = 496$

$D_x = 1.625 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2540 reflections

$\theta = 2.2\text{--}29.5^\circ$

$\mu = 0.33 \text{ mm}^{-1}$

$T = 100 \text{ K}$

Block, yellow

$0.49 \times 0.01 \times 0.01 \text{ mm}$

### Data collection

Bruker SMART APEX

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.856$ ,  $T_{\max} = 0.997$

9462 measured reflections

2247 independent reflections

1725 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 2.1^\circ$

$h = -16 \rightarrow 15$

$k = -5 \rightarrow 7$

$l = -18 \rightarrow 18$

Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.111$   
 $S = 1.04$   
 2247 reflections  
 161 parameters  
 8 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0616P)^2 + 0.4126P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.47 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$

Special details

**Geometry.** All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.45571 (4)	1.58779 (11)	0.35227 (3)	0.02368 (17)
O1	0.78686 (12)	0.8925 (3)	0.67628 (10)	0.0271 (4)
O2	0.92212 (11)	0.0797 (3)	0.40282 (10)	0.0258 (3)
O3	0.82499 (11)	0.3034 (3)	0.30592 (9)	0.0243 (3)
N1	0.86480 (13)	0.2593 (3)	0.38518 (11)	0.0200 (4)
N2	0.64392 (12)	1.0445 (3)	0.42660 (11)	0.0179 (4)
N3	0.56870 (13)	1.2266 (3)	0.42711 (11)	0.0199 (4)
N4	0.57458 (14)	1.2637 (4)	0.26788 (12)	0.0222 (4)
C1	0.80683 (15)	0.7307 (4)	0.60870 (13)	0.0201 (4)
C2	0.87612 (16)	0.5353 (4)	0.62369 (13)	0.0215 (4)
H2	0.9106	0.5093	0.6843	0.026*
C3	0.89488 (15)	0.3793 (4)	0.55093 (13)	0.0211 (4)
H3	0.9414	0.2440	0.5609	0.025*
C4	0.84439 (15)	0.4236 (4)	0.46231 (13)	0.0185 (4)
C5	0.77372 (15)	0.6132 (4)	0.44565 (13)	0.0180 (4)
H5	0.7398	0.6378	0.3847	0.022*
C6	0.75301 (14)	0.7676 (4)	0.51947 (13)	0.0179 (4)
C7	0.67610 (15)	0.9650 (4)	0.50831 (13)	0.0197 (4)
H7	0.6492	1.0363	0.5626	0.024*
C8	0.53819 (15)	1.3452 (4)	0.34732 (13)	0.0195 (4)
H1	0.8337 (17)	0.880 (5)	0.7212 (14)	0.044 (8)*
H31	0.5515 (19)	1.281 (5)	0.4800 (11)	0.036 (7)*
H41	0.570 (2)	1.360 (4)	0.2208 (13)	0.038 (7)*
H42	0.6185 (16)	1.145 (3)	0.2711 (17)	0.030 (7)*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0247 (3)	0.0265 (3)	0.0193 (2)	0.0085 (2)	-0.00213 (18)	-0.0004 (2)
O1	0.0303 (8)	0.0290 (9)	0.0209 (7)	0.0072 (7)	-0.0059 (6)	-0.0041 (6)
O2	0.0268 (8)	0.0220 (8)	0.0287 (7)	0.0074 (7)	0.0022 (6)	-0.0008 (6)
O3	0.0268 (8)	0.0259 (9)	0.0200 (7)	0.0004 (7)	0.0016 (6)	0.0007 (6)
N1	0.0181 (8)	0.0185 (9)	0.0239 (8)	-0.0036 (7)	0.0047 (6)	0.0002 (7)
N2	0.0152 (8)	0.0175 (9)	0.0210 (7)	0.0005 (7)	0.0014 (6)	0.0008 (6)
N3	0.0201 (8)	0.0220 (10)	0.0174 (8)	0.0051 (7)	0.0008 (6)	0.0007 (7)
N4	0.0249 (9)	0.0228 (10)	0.0191 (8)	0.0049 (8)	0.0019 (7)	0.0015 (7)
C1	0.0201 (10)	0.0204 (11)	0.0200 (9)	-0.0020 (9)	0.0024 (7)	-0.0005 (8)
C2	0.0197 (10)	0.0242 (12)	0.0200 (9)	0.0011 (9)	-0.0029 (7)	0.0025 (8)
C3	0.0178 (10)	0.0198 (11)	0.0254 (10)	0.0022 (8)	-0.0001 (7)	0.0037 (8)
C4	0.0158 (9)	0.0175 (10)	0.0224 (9)	-0.0035 (8)	0.0029 (7)	-0.0005 (8)
C5	0.0155 (9)	0.0187 (11)	0.0198 (9)	-0.0028 (8)	0.0012 (7)	0.0043 (7)
C6	0.0149 (9)	0.0189 (11)	0.0199 (9)	-0.0013 (8)	0.0012 (7)	0.0023 (8)
C7	0.0188 (10)	0.0205 (11)	0.0198 (9)	0.0007 (8)	0.0015 (7)	-0.0010 (8)
C8	0.0166 (9)	0.0208 (11)	0.0207 (9)	-0.0024 (8)	-0.0016 (7)	0.0009 (8)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

S1—C8	1.693 (2)	N4—H42	0.852 (10)
O1—C1	1.345 (2)	C1—C2	1.390 (3)
O1—H1	0.842 (10)	C1—C6	1.412 (2)
O2—N1	1.237 (2)	C2—C3	1.377 (3)
O3—N1	1.227 (2)	C2—H2	0.9500
N1—C4	1.457 (3)	C3—C4	1.394 (3)
N2—C7	1.281 (2)	C3—H3	0.9500
N2—N3	1.378 (2)	C4—C5	1.379 (3)
N3—C8	1.342 (2)	C5—C6	1.389 (3)
N3—H31	0.852 (10)	C5—H5	0.9500
N4—C8	1.328 (3)	C6—C7	1.455 (3)
N4—H41	0.853 (10)	C7—H7	0.9500
C1—O1—H1	109 (2)	C2—C3—H3	120.6
O3—N1—O2	122.64 (17)	C4—C3—H3	120.6
O3—N1—C4	119.27 (17)	C5—C4—C3	122.38 (19)
O2—N1—C4	118.09 (16)	C5—C4—N1	118.87 (17)
C7—N2—N3	114.57 (16)	C3—C4—N1	118.73 (18)
C8—N3—N2	120.22 (16)	C4—C5—C6	118.88 (17)
C8—N3—H31	120.1 (18)	C4—C5—H5	120.6
N2—N3—H31	118.6 (18)	C6—C5—H5	120.6
C8—N4—H41	116.8 (18)	C5—C6—C1	119.31 (18)
C8—N4—H42	118.3 (17)	C5—C6—C7	122.03 (17)
H41—N4—H42	122 (2)	C1—C6—C7	118.65 (18)
O1—C1—C2	123.07 (17)	N2—C7—C6	121.22 (18)
O1—C1—C6	116.53 (18)	N2—C7—H7	119.4

C2—C1—C6	120.40 (18)	C6—C7—H7	119.4
C3—C2—C1	120.18 (18)	N4—C8—N3	117.54 (19)
C3—C2—H2	119.9	N4—C8—S1	123.38 (15)
C1—C2—H2	119.9	N3—C8—S1	119.07 (15)
C2—C3—C4	118.77 (19)		

*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>
O1—H1 $\cdots$ S1 <sup>i</sup>	0.84 (1)	2.34 (1)	3.175 (2)	170 (3)
N3—H31 $\cdots$ S1 <sup>ii</sup>	0.85 (1)	2.50 (1)	3.337 (2)	167 (2)
N4—H41 $\cdots$ O2 <sup>iii</sup>	0.85 (1)	2.14 (1)	2.987 (2)	172 (3)
N4—H42 $\cdots$ O3 <sup>iv</sup>	0.85 (1)	2.31 (2)	3.044 (2)	144 (2)

Symmetry codes: (i)  $x+1/2, -y+5/2, z+1/2$ ; (ii)  $-x+1, -y+3, -z+1$ ; (iii)  $-x+3/2, y+3/2, -z+1/2$ ; (iv)  $-x+3/2, y+1/2, -z+1/2$ .