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N'-(2-Hydroxy-5-nitrobenzylidene)-benzenesulfonohydrazide

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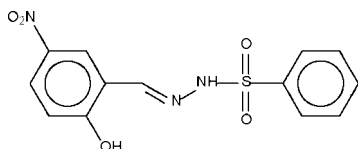
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 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.033; wR factor = 0.089; data-to-parameter ratio = 12.7.

The molecule of the title compound, $\text{C}_{13}\text{H}_{11}\text{N}_3\text{O}_5\text{S}$, shows a phenyl group and an almost planar intramolecularly hydrogen-bonded *N'*-(2-hydroxy-5-nitrobenzylidene)-hydrazino group disposed about the S atom. Adjacent molecules are linked by $\text{N}-\text{H}\cdots\text{O}_{\text{nitro}}$ hydrogen bonds, producing a linear chain that runs along the b axis of the unit cell.

Related literature

 For 2'-[1-(2-hydroxyphenyl)ethylidene]benzenesulfonohydrazide, see: Tai *et al.* (2008).


Experimental

Crystal data

 $\text{C}_{13}\text{H}_{11}\text{N}_3\text{O}_5\text{S}$
 $M_r = 321.31$
 Monoclinic, $P2_1/c$
 $a = 7.8188$ (1) Å
 $b = 14.5904$ (2) Å

 $c = 11.9083$ (1) Å
 $\beta = 98.159$ (1)°
 $V = 1344.74$ (3) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

 $\mu = 0.27$ mm⁻¹
 $T = 100$ (2) K

 $0.47 \times 0.40 \times 0.33$ mm

Data collection

 Bruker SMART APEX
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.883$, $T_{\text{max}} = 0.916$

 16761 measured reflections
 3089 independent reflections
 2972 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.088$
 $S = 1.03$
 3089 reflections
 243 parameters
 11 restraints

 H atoms treated by a mixture of
 independent and constrained
 refinement
 $\Delta\rho_{\text{max}} = 0.42$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.51$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}o\cdots\text{N2}$	0.84 (1)	1.86 (1)	2.628 (1)	153 (2)
$\text{N3}-\text{H3}n\cdots\text{O3}^i$	0.88 (1)	2.13 (1)	2.978 (1)	163 (2)

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

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; 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).

We thank the Science Fund (12-02-03-2031, 12-02-03-2051) and the University of Malaya (PJP) for supporting this study. We are grateful to the University of Malaya for the purchase of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2077).

References

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 Tai, X.-S., Xu, J., Feng, Y.-M. & Liang, Z.-P. (2008). *Acta Cryst.* **E64**, o904.
 Westrip, S. P. (2008). publCIF. In preparation.

supplementary materials

Acta Cryst. (2008). E64, o1583 [doi:10.1107/S1600536808022691]

N'-(2-Hydroxy-5-nitrobenzylidene)benzenesulfonohydrazide

J. Yusnita, H. M. Ali, S. Puvaneswary, W. T. Robinson and S. W. Ng

Comment

The molecules of benzenesulfonic acid-(2-hydroxy-5-nitro-benzylidene)-hydrazide adopt a linear chain-like structure in which the individual molecules are linked by N–H···O hydrogen bonds [3.093 (3) Å] (Tai *et al.*, 2008). The title compound exhibits a phenyl group and an almost planar, intramolecularly hydrogen-bonded (2-hydroxy-5-phenyl)ethylidenehydrazidyl group disposed about the sulfur atom. The nitro group is situated *para* with respect to the OH donor site (Scheme I, Fig. 1). The nitro group also acts as an acceptor towards the amino group of an adjacent molecule to furnish a chain-like structure that runs along the *b*-axis of the unit cell (Fig. 2).

Experimental

2-Hydroxy-5-nitrobenzaldehyde (0.50 g, 3 mmol) and benzene sulfonylhydrazide (0.52 g, 0.3 mmol) were condensed in refluxing ethanol (100 ml) for two hours. The solvent was removed to give the Schiff base, which was collected in nearly quantitative yield. and dried. Crystals were obtained by recrystallization from ethanol.

Refinement

All hydrogen atoms were located in a difference Fourier map, and were refined with distance restraints of C–H 0.95±0.01, N–H 0.88±0.01 and O–H 0.84±0.01 Å. Their temperature factors were refined freely.

Figures

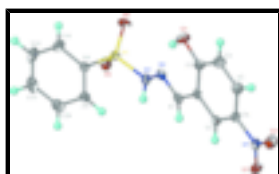


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) plot of C₁₃H₁₁N₃O₅S at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

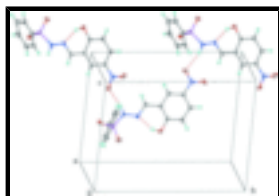


Fig. 2. Hydrogen-bonded chain structure. Hydrogen bonds are denoted by dashed lines.

N'-(2-Hydroxy-5-nitrobenzylidene)benzenesulfonohydrazide

Crystal data

C₁₃H₁₁N₃O₅S

$F_{000} = 664$

supplementary materials

$M_r = 321.31$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 7.8188$ (1) Å

$b = 14.5904$ (2) Å

$c = 11.9083$ (1) Å

$\beta = 98.159$ (1)°

$V = 1344.74$ (3) Å³

$Z = 4$

$D_x = 1.587$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 9991 reflections

$\theta = 2.4$ – 30.4 °

$\mu = 0.27$ mm⁻¹

$T = 100$ (2) K

Triangular plate, yellow

$0.47 \times 0.40 \times 0.33$ mm

Data collection

Bruker SMART APEX
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 100$ (2) K

ω scans

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

$T_{\min} = 0.883$, $T_{\max} = 0.916$

16761 measured reflections

3089 independent reflections

2972 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\text{max}} = 27.5$ °

$\theta_{\text{min}} = 2.2$ °

$h = -10 \rightarrow 9$

$k = -18 \rightarrow 18$

$l = -14 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.088$

$S = 1.04$

3089 reflections

243 parameters

11 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

$$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 0.7306P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.42$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.51$ e Å⁻³

Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.78969 (4)	0.172790 (19)	0.37657 (2)	0.01358 (10)
N1	0.89489 (14)	0.67461 (7)	0.75588 (9)	0.0169 (2)
N2	0.82479 (13)	0.33431 (7)	0.46383 (9)	0.0149 (2)
N3	0.89849 (13)	0.24787 (7)	0.46141 (9)	0.0150 (2)
O1	0.65151 (13)	0.47538 (6)	0.36923 (8)	0.0233 (2)

O2	0.84138 (13)	0.75301 (6)	0.76435 (8)	0.0239 (2)
O3	1.00400 (12)	0.63904 (7)	0.82771 (8)	0.0224 (2)
O4	0.74085 (13)	0.21585 (7)	0.26881 (7)	0.0230 (2)
O5	0.89964 (11)	0.09391 (6)	0.38421 (8)	0.01914 (19)
C1	0.70881 (16)	0.52031 (8)	0.46569 (10)	0.0166 (2)
C2	0.66093 (17)	0.61234 (9)	0.47357 (11)	0.0202 (3)
C3	0.71928 (16)	0.66280 (8)	0.56918 (11)	0.0181 (2)
C4	0.82690 (15)	0.62056 (8)	0.65697 (10)	0.0150 (2)
C5	0.87528 (15)	0.52972 (8)	0.65268 (10)	0.0146 (2)
C6	0.81634 (15)	0.47794 (8)	0.55664 (10)	0.0143 (2)
C7	0.87482 (15)	0.38342 (8)	0.55157 (10)	0.0149 (2)
C8	0.60116 (14)	0.14633 (8)	0.43497 (10)	0.0129 (2)
C9	0.58974 (16)	0.16378 (8)	0.54832 (10)	0.0157 (2)
C10	0.44273 (17)	0.13509 (9)	0.59181 (11)	0.0208 (3)
C11	0.31115 (16)	0.08982 (9)	0.52296 (13)	0.0231 (3)
C12	0.32481 (16)	0.07308 (9)	0.41038 (13)	0.0231 (3)
C13	0.47021 (16)	0.10120 (9)	0.36497 (11)	0.0189 (2)
H10	0.692 (3)	0.4223 (8)	0.3795 (18)	0.043 (6)*
H3N	0.944 (2)	0.2249 (12)	0.5268 (10)	0.027 (4)*
H2	0.587 (2)	0.6401 (12)	0.4127 (12)	0.028 (4)*
H3	0.691 (2)	0.7249 (7)	0.5788 (15)	0.025 (4)*
H5	0.9475 (18)	0.5022 (10)	0.7134 (11)	0.019 (4)*
H7	0.9524 (18)	0.3611 (11)	0.6142 (10)	0.019 (4)*
H9	0.6803 (17)	0.1930 (11)	0.5961 (12)	0.023 (4)*
H10	0.431 (2)	0.1463 (13)	0.6687 (9)	0.033 (5)*
H11	0.2133 (16)	0.0685 (11)	0.5530 (14)	0.026 (4)*
H12	0.2367 (19)	0.0410 (11)	0.3635 (13)	0.030 (4)*
H13	0.482 (2)	0.0875 (11)	0.2882 (9)	0.025 (4)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01629 (16)	0.01318 (16)	0.01162 (15)	-0.00058 (9)	0.00324 (11)	-0.00033 (9)
N1	0.0174 (5)	0.0180 (5)	0.0160 (5)	-0.0050 (4)	0.0042 (4)	-0.0018 (4)
N2	0.0170 (5)	0.0106 (4)	0.0175 (5)	-0.0002 (3)	0.0038 (4)	0.0021 (3)
N3	0.0161 (5)	0.0110 (4)	0.0174 (5)	-0.0007 (4)	0.0006 (4)	0.0002 (4)
O1	0.0356 (5)	0.0154 (4)	0.0158 (4)	0.0037 (4)	-0.0069 (4)	-0.0016 (3)
O2	0.0272 (5)	0.0183 (4)	0.0260 (5)	-0.0003 (4)	0.0035 (4)	-0.0078 (4)
O3	0.0245 (5)	0.0236 (5)	0.0174 (4)	-0.0045 (4)	-0.0028 (4)	-0.0012 (3)
O4	0.0299 (5)	0.0266 (5)	0.0125 (4)	-0.0012 (4)	0.0028 (4)	0.0040 (3)
O5	0.0187 (4)	0.0158 (4)	0.0240 (5)	0.0009 (3)	0.0067 (3)	-0.0037 (3)
C1	0.0203 (6)	0.0150 (6)	0.0139 (5)	-0.0006 (4)	0.0003 (4)	0.0002 (4)
C2	0.0250 (6)	0.0160 (6)	0.0180 (6)	0.0030 (5)	-0.0024 (5)	0.0018 (4)
C3	0.0205 (6)	0.0133 (5)	0.0204 (6)	0.0012 (4)	0.0026 (5)	0.0000 (4)
C4	0.0162 (5)	0.0153 (5)	0.0137 (5)	-0.0037 (4)	0.0031 (4)	-0.0018 (4)
C5	0.0149 (5)	0.0158 (5)	0.0132 (5)	-0.0022 (4)	0.0020 (4)	0.0023 (4)
C6	0.0156 (5)	0.0132 (5)	0.0142 (5)	-0.0016 (4)	0.0028 (4)	0.0018 (4)
C7	0.0156 (5)	0.0136 (5)	0.0158 (5)	-0.0012 (4)	0.0027 (4)	0.0034 (4)

supplementary materials

C8	0.0132 (5)	0.0109 (5)	0.0148 (5)	0.0008 (4)	0.0023 (4)	0.0007 (4)
C9	0.0162 (5)	0.0163 (5)	0.0147 (5)	0.0007 (4)	0.0020 (4)	0.0001 (4)
C10	0.0210 (6)	0.0215 (6)	0.0214 (6)	0.0038 (5)	0.0084 (5)	0.0036 (5)
C11	0.0156 (6)	0.0169 (6)	0.0383 (8)	0.0020 (4)	0.0090 (5)	0.0046 (5)
C12	0.0154 (6)	0.0164 (6)	0.0359 (7)	-0.0010 (4)	-0.0020 (5)	-0.0038 (5)
C13	0.0183 (6)	0.0172 (6)	0.0199 (6)	0.0012 (4)	-0.0020 (4)	-0.0036 (4)

Geometric parameters (Å, °)

S1—O4	1.4312 (9)	C3—H3	0.943 (9)
S1—O5	1.4316 (9)	C4—C5	1.3812 (17)
S1—N3	1.6434 (10)	C5—C6	1.3934 (16)
S1—C8	1.7597 (12)	C5—H5	0.942 (9)
N1—O2	1.2271 (14)	C6—C7	1.4569 (16)
N1—O3	1.2336 (14)	C7—H7	0.950 (9)
N1—C4	1.4540 (15)	C8—C9	1.3889 (16)
N2—C7	1.2815 (16)	C8—C13	1.3908 (16)
N2—N3	1.3886 (13)	C9—C10	1.3905 (17)
N3—H3N	0.875 (9)	C9—H9	0.945 (9)
O1—C1	1.3431 (14)	C10—C11	1.3884 (19)
O1—H1O	0.838 (9)	C10—H10	0.947 (9)
C1—C2	1.4006 (17)	C11—C12	1.382 (2)
C1—C6	1.4155 (16)	C11—H11	0.942 (9)
C2—C3	1.3780 (17)	C12—C13	1.3884 (19)
C2—H2	0.952 (9)	C12—H12	0.947 (9)
C3—C4	1.3897 (17)	C13—H13	0.953 (9)
O4—S1—O5	119.43 (6)	C4—C5—H5	121.2 (10)
O4—S1—N3	107.91 (6)	C6—C5—H5	119.4 (10)
O5—S1—N3	104.06 (5)	C5—C6—C1	118.86 (11)
O4—S1—C8	108.59 (6)	C5—C6—C7	118.56 (10)
O5—S1—C8	109.12 (5)	C1—C6—C7	122.52 (11)
N3—S1—C8	107.03 (5)	N2—C7—C6	120.12 (11)
O2—N1—O3	123.06 (11)	N2—C7—H7	122.4 (10)
O2—N1—C4	118.84 (10)	C6—C7—H7	117.4 (10)
O3—N1—C4	118.09 (10)	C9—C8—C13	121.73 (11)
C7—N2—N3	116.43 (10)	C9—C8—S1	121.12 (9)
N2—N3—S1	115.98 (8)	C13—C8—S1	116.98 (9)
N2—N3—H3N	116.5 (12)	C8—C9—C10	118.51 (11)
S1—N3—H3N	113.7 (12)	C8—C9—H9	121.5 (10)
C1—O1—H1O	104.7 (15)	C10—C9—H9	120.0 (10)
O1—C1—C2	117.73 (10)	C11—C10—C9	120.38 (12)
O1—C1—C6	122.07 (11)	C11—C10—H10	119.2 (12)
C2—C1—C6	120.18 (11)	C9—C10—H10	120.4 (12)
C3—C2—C1	120.48 (11)	C12—C11—C10	120.28 (12)
C3—C2—H2	119.8 (11)	C12—C11—H11	119.3 (11)
C1—C2—H2	119.7 (11)	C10—C11—H11	120.4 (11)
C2—C3—C4	118.60 (11)	C11—C12—C13	120.40 (12)
C2—C3—H3	123.8 (11)	C11—C12—H12	120.7 (11)
C4—C3—H3	117.6 (11)	C13—C12—H12	118.9 (11)

C5—C4—C3	122.51 (11)	C12—C13—C8	118.71 (12)
C5—C4—N1	118.68 (10)	C12—C13—H13	120.5 (11)
C3—C4—N1	118.79 (11)	C8—C13—H13	120.8 (11)
C4—C5—C6	119.35 (11)		
C7—N2—N3—S1	158.80 (9)	O1—C1—C6—C7	1.05 (19)
O4—S1—N3—N2	47.89 (10)	C2—C1—C6—C7	-177.91 (11)
O5—S1—N3—N2	175.72 (8)	N3—N2—C7—C6	174.40 (10)
C8—S1—N3—N2	-68.82 (9)	C5—C6—C7—N2	-179.10 (11)
O1—C1—C2—C3	-178.31 (12)	C1—C6—C7—N2	-2.18 (18)
C6—C1—C2—C3	0.69 (19)	O4—S1—C8—C9	-135.67 (10)
C1—C2—C3—C4	0.3 (2)	O5—S1—C8—C9	92.63 (10)
C2—C3—C4—C5	-1.02 (19)	N3—S1—C8—C9	-19.41 (11)
C2—C3—C4—N1	177.34 (11)	O4—S1—C8—C13	48.99 (11)
O2—N1—C4—C5	-174.10 (11)	O5—S1—C8—C13	-82.72 (10)
O3—N1—C4—C5	5.76 (16)	N3—S1—C8—C13	165.25 (9)
O2—N1—C4—C3	7.48 (17)	C13—C8—C9—C10	-0.06 (18)
O3—N1—C4—C3	-172.65 (11)	S1—C8—C9—C10	-175.19 (9)
C3—C4—C5—C6	0.71 (18)	C8—C9—C10—C11	0.18 (18)
N1—C4—C5—C6	-177.64 (10)	C9—C10—C11—C12	-0.20 (19)
C4—C5—C6—C1	0.30 (17)	C10—C11—C12—C13	0.1 (2)
C4—C5—C6—C7	177.34 (10)	C11—C12—C13—C8	0.03 (19)
O1—C1—C6—C5	177.97 (11)	C9—C8—C13—C12	-0.04 (18)
C2—C1—C6—C5	-0.99 (18)	S1—C8—C13—C12	175.28 (9)

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 _o \cdots N2	0.84 (1)	1.86 (1)	2.628 (1)	153 (2)
N3—H3 _n \cdots O3 ⁱ	0.88 (1)	2.13 (1)	2.978 (1)	163 (2)

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

Fig. 1

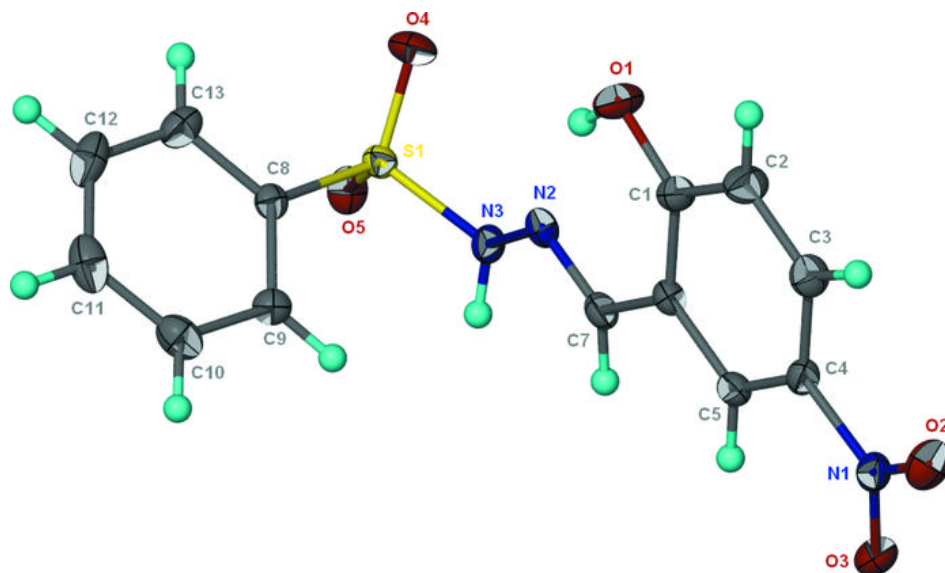


Fig. 2

