Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Juahir Yusnita, Hapipah M. Ali, Subramaniam Puvaneswary, Ward T. Robinson and Seik Weng Ng*

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

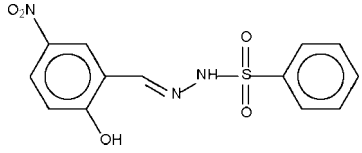
Received 10 July 2008; accepted 19 July 2008

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.089; data-to-parameter ratio = 12.7.

The molecule of the title compound, $C_{13}H_{11}N_3O_5S$, shows a phenyl group and an almost planar intramolecularly hydrogen-bonded *N*′-(2-hydroxy-5-phenylebenzylidene)-hydrazino group disposed about the S atom. Adjacent molecules are linked by N—H···O_{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

$C_{13}H_{11}N_3O_5S$
M_r = 321.31
Monoclinic, $P2_1/c$
a = 7.8188 (1) Å
b = 14.5904 (2) Å
c = 11.9083 (1) Å
β = 98.159 (1)°
V = 1344.74 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 100 (2) K
0.47 × 0.40 × 0.33 mm

Data collection

Bruker SMART APEX diffractometer
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_{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
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*o*···N2	0.84 (1)	1.86 (1)	2.628 (1)	153 (2)
N3—H3*n*···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

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tai, X.-S., Xu, J., Feng, Y.-M. & Liang, Z.-P. (2008). *Acta Cryst.* E**64**, o904.
Westrip, S. P. (2008). *publCIF*. In preparation.

supporting information

Acta Cryst. (2008). E**64**, o1583 [doi:10.1107/S1600536808022691]

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

Juahir Yusnita, Hapipah M. Ali, Subramaniam Puvaneswary, Ward T. Robinson and Seik Weng Ng

S1. 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)ethyl-idenehydrazidyl 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).

S2. 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 quantative yield. and dried. Crystals were obtained by recrystallization from ethanol.

S3. Refinement

All hydrogen atoms were located in a difference Fouier 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.

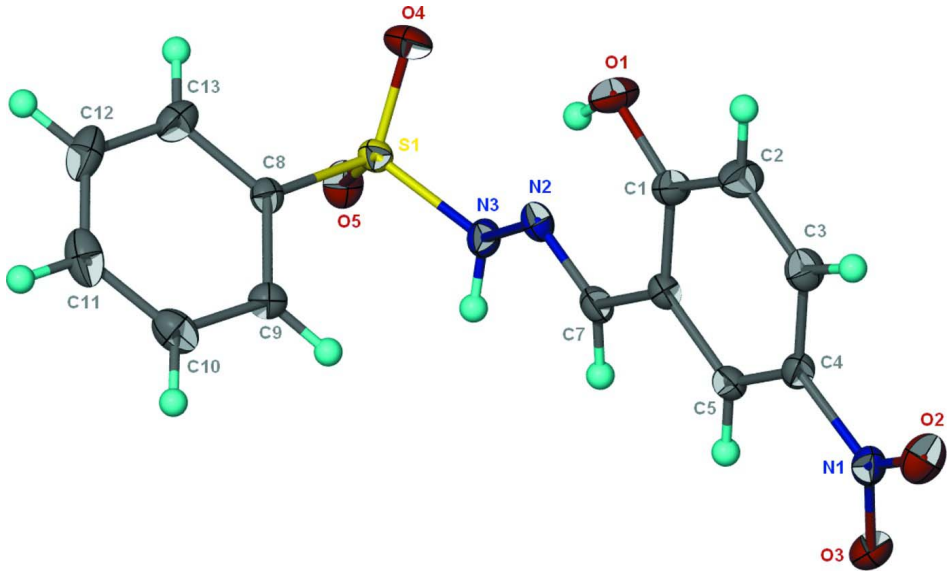


Figure 1

Thermal ellipsoid plot (Barbour, 2001) plot of $C_{13}H_{11}N_3O_5S$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

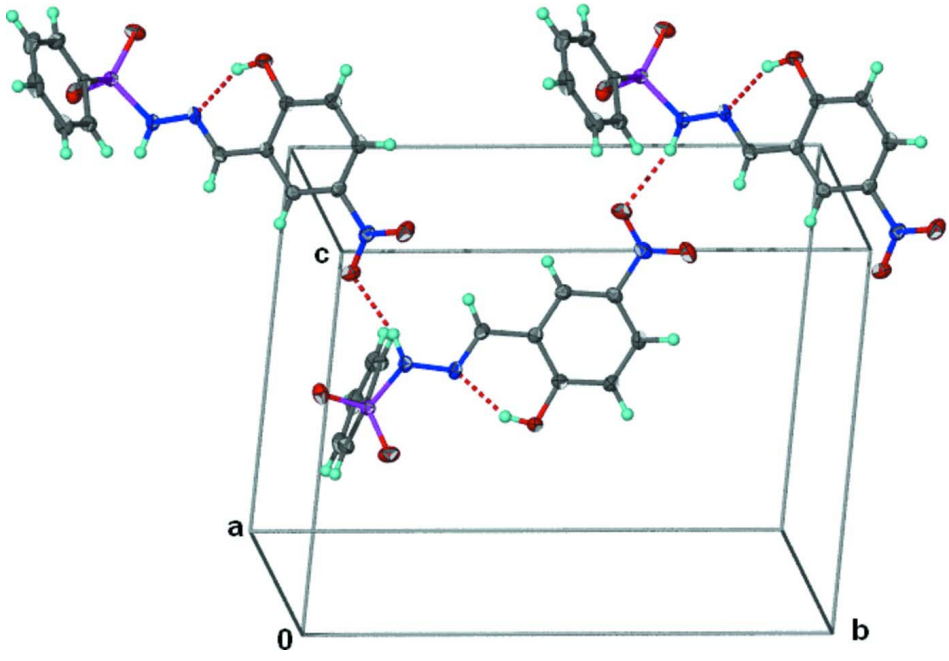


Figure 2

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

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

Crystal data

$C_{13}H_{11}N_3O_5S$
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
β = 98.159 (1)°
V = 1344.74 (3) Å^3
Z = 4
F(000) = 664
D_x = 1.587 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 9991 reflections
θ = 2.4–30.4°
μ = 0.27 mm^{-1}
T = 100 K
Triangular plate, yellow
0.47 × 0.40 × 0.33 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
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_{int} = 0.024
θ_{max} = 27.5°, θ_{min} = 2.2°
h = −10→9
k = −18→18
l = −14→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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{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)
H1O	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 (Å²)

	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)
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, º)

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, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1o···N2	0.84 (1)	1.86 (1)	2.628 (1)	153 (2)
N3—H3n···O3^{i}	0.88 (1)	2.13 (1)	2.978 (1)	163 (2)

Symmetry code: (i) −*x*+2, *y*−1/2, −*z*+3/2.