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N′-(2,5-Dihydroxybenzylidene)benzenesulfonohydrazide

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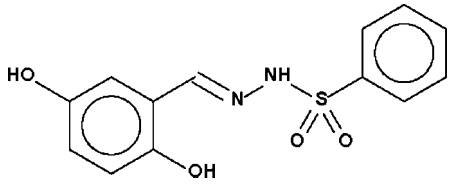
Key indicators: single-crystal X-ray study; T = 128 K; mean σ(C–C) = 0.002 Å; R factor = 0.032; wR factor = 0.104; data-to-parameter ratio = 12.8.

In the title compound, $C_{13}H_{12}N_2O_4S$, the dihedral angle between the two aromatic rings is 89.5 (1)°. In the crystal structure, molecules are linked by O—H···$O_{hydroxy}$ and N—H···$O_{sulfonyl}$ hydrogen bonds, forming a ribbon that propagates along the *b* axis; there is also an intramolecular O—H···N hydrogen bond.

Related literature

For the structure of 2′-(5-bromo-2-hydroxybenzylidene)-benzenesulfonohydrazide, see: Ali *et al.* (2007).



Experimental

Crystal data

$C_{13}H_{12}N_2O_4S$
M_r = 292.31
Monoclinic, $P2_1/n$
a = 12.5814 (2) Å
b = 7.1601 (1) Å
c = 14.6727 (2) Å
β = 105.540 (1)°
V = 1273.46 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 128 (2) K
0.65 × 0.33 × 0.29 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: none
16406 measured reflections
2924 independent reflections
2684 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.104
S = 1.07
2924 reflections
229 parameters
11 restraints
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3O···N2	0.85 (1)	1.82 (2)	2.562 (2)	145 (2)
O4—H4O···O3^{i}	0.85 (1)	1.85 (1)	2.698 (2)	175 (2)
N1—H1N···O1^{ii}	0.85 (1)	2.05 (1)	2.897 (2)	171 (2)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); 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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2236).

References

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
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supporting information

Acta Cryst. (2008). E**64**, o522 [doi:10.1107/S1600536808002225]

N′-(2,5-Dihydroxybenzylidene)benzenesulfonohydrazide

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S1. Comment

A recent study reported the crystal structure of 2′-(5-bromo-2-hydroxybenzylidene)benzenesulfonohydrazide (Ali *et al.*, 2007). A second hydroxy group is introduced in the title compound.

S2. Experimental

Benzenesulfohydrazine (0.29 g, 1.7 mmol) and 2,5-dihydroxybenzaldehyde (0.24 g, 1.7 mmol) were refluxed in ethanol (50 ml) for 2 h. The solvent was removed to give the product Schiff base, and crystals were obtained upon recrystallization from ethanol.

S3. Refinement

All hydrogen atoms were located in difference Fourier maps. Those bonded to C were restrained to 0.95±0.01 Å, and those bonded to N or O to 0.85±0.01 Å. All displacement parameters were freely refined.

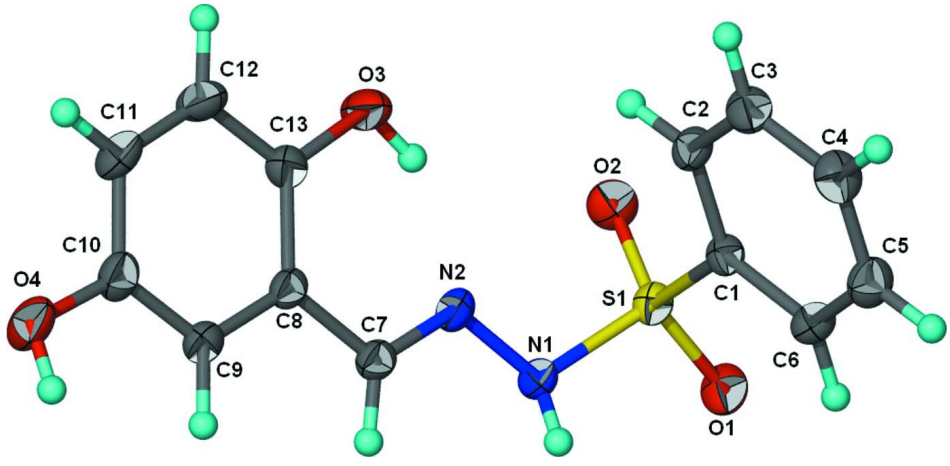

Figure 1

Molecular structure of the title compound. Displacement ellipsoids are drawn at the 70% probability level, and H atoms are shown as spheres of arbitrary radius.

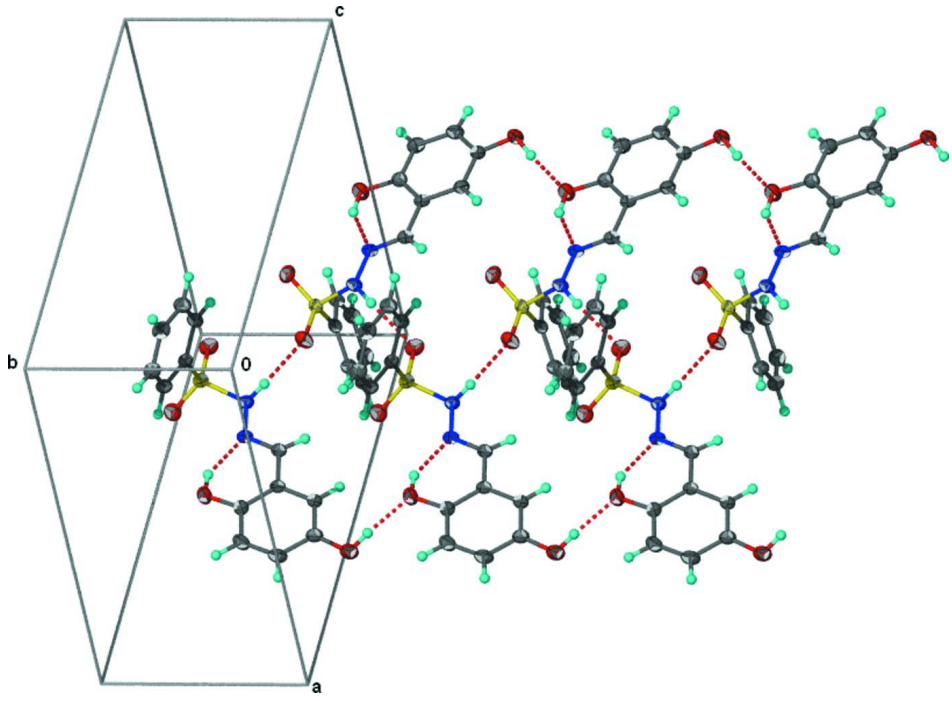

Figure 2

A view of the ribbon structure of the title compound. Dashed lines indicate hydrogen bonds.

N′-(2,5-Dihydroxybenzylidene)benzenesulfonohydrazide

Crystal data

$C_{13}H_{12}N_2O_4S$
$M_r = 292.31$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 12.5814\ (2)$ Å
$b = 7.1601\ (1)$ Å
$c = 14.6727\ (2)$ Å
$\beta = 105.540\ (1)°$
$V = 1273.46\ (3)$ Å^3
$Z = 4$
$F(000) = 608$
$D_x = 1.525$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9918 reflections
$\theta = 5.0–63.2°$
$\mu = 0.27$ mm^{-1}
$T = 128$ K
Block, yellow
0.65 × 0.33 × 0.29 mm

Data collection

Bruker APEXII diffractometer
Radiation source: medium-focus sealed tube
Graphite monochromator
φ and ω scans
16406 measured reflections
2924 independent reflections
2684 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.9°$
$h = -16\rightarrow16$
$k = -9\rightarrow9$
$l = -18\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.104$
$S = 1.07$
2924 reflections
229 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

All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0666P)^2 + 0.482P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}

Special details

Experimental. A medium-focus collimator of 0.8 mm diameter was used on the diffractometer to measure the somewhat large crystal.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.77742 (3)	0.75958 (4)	0.66733 (2)	0.01720 (12)
N1	0.83576 (9)	0.55528 (16)	0.65868 (8)	0.0190 (2)
N2	0.84430 (9)	0.52079 (16)	0.56754 (8)	0.0186 (2)
O1	0.77923 (9)	0.76844 (15)	0.76588 (7)	0.0245 (2)
O2	0.83351 (8)	0.89724 (14)	0.62689 (7)	0.0249 (2)
O3	0.85080 (9)	0.64124 (14)	0.40471 (7)	0.0276 (2)
O4	0.91487 (10)	−0.08605 (15)	0.30407 (8)	0.0307 (3)
C1	0.63909 (11)	0.74943 (17)	0.59938 (9)	0.0164 (3)
C2	0.61433 (11)	0.7976 (2)	0.50377 (9)	0.0198 (3)
C3	0.50524 (12)	0.7887 (2)	0.44977 (10)	0.0228 (3)
C4	0.42314 (12)	0.7347 (2)	0.49182 (11)	0.0241 (3)
C5	0.44840 (12)	0.6872 (2)	0.58702 (11)	0.0242 (3)
C6	0.55757 (11)	0.69292 (19)	0.64210 (9)	0.0204 (3)
C7	0.86202 (10)	0.35212 (19)	0.54498 (9)	0.0182 (3)
C8	0.87129 (10)	0.31449 (19)	0.44957 (9)	0.0175 (3)
C9	0.88699 (11)	0.12938 (19)	0.42327 (10)	0.0199 (3)
C10	0.89662 (11)	0.08955 (19)	0.33332 (10)	0.0220 (3)
C11	0.88899 (13)	0.2343 (2)	0.26805 (10)	0.0248 (3)
C12	0.87375 (12)	0.4168 (2)	0.29252 (10)	0.0248 (3)
C13	0.86505 (11)	0.45797 (19)	0.38301 (9)	0.0203 (3)
H3O	0.8478 (19)	0.650 (3)	0.4615 (8)	0.052 (6)*
H4O	0.8920 (18)	−0.168 (3)	0.3363 (15)	0.050 (6)*
H1N	0.8083 (15)	0.470 (2)	0.6862 (13)	0.034 (5)*
H2	0.6718 (11)	0.834 (2)	0.4758 (12)	0.026 (4)*
H3	0.4885 (15)	0.824 (3)	0.3850 (7)	0.031 (5)*
H4	0.3471 (18)	0.729 (3)	0.4543 (14)	0.033 (5)*
H5	0.3922 (12)	0.651 (3)	0.6166 (12)	0.032 (5)*
H6	0.5728 (13)	0.657 (2)	0.7068 (7)	0.024 (4)*
H7	0.8721 (15)	0.2494 (17)	0.5872 (10)	0.019 (4)*
H9	0.8924 (15)	0.031 (2)	0.4681 (10)	0.028 (4)*
H11	0.8879 (18)	0.201 (3)	0.2057 (8)	0.045 (6)*
H12	0.8656 (15)	0.515 (2)	0.2476 (11)	0.032 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01696 (19)	0.01935 (18)	0.01461 (19)	−0.00098 (10)	0.00306 (13)	−0.00294 (10)
N1	0.0212 (5)	0.0213 (5)	0.0157 (5)	0.0023 (4)	0.0070 (4)	0.0008 (4)
N2	0.0174 (5)	0.0235 (6)	0.0161 (5)	0.0000 (4)	0.0066 (4)	−0.0005 (4)
O1	0.0261 (5)	0.0312 (6)	0.0144 (5)	0.0025 (4)	0.0025 (4)	−0.0053 (4)
O2	0.0229 (5)	0.0226 (5)	0.0288 (5)	−0.0048 (4)	0.0065 (4)	−0.0010 (4)
O3	0.0434 (6)	0.0195 (5)	0.0232 (5)	0.0037 (4)	0.0147 (5)	0.0033 (4)
O4	0.0461 (7)	0.0225 (5)	0.0318 (6)	−0.0037 (4)	0.0249 (5)	−0.0047 (4)
C1	0.0166 (6)	0.0173 (6)	0.0150 (6)	0.0003 (4)	0.0037 (5)	−0.0022 (4)
C2	0.0210 (6)	0.0231 (6)	0.0164 (6)	0.0009 (5)	0.0069 (5)	−0.0010 (5)
C3	0.0235 (7)	0.0274 (7)	0.0163 (6)	0.0028 (5)	0.0033 (5)	−0.0013 (5)
C4	0.0187 (7)	0.0266 (7)	0.0247 (7)	−0.0007 (5)	0.0020 (6)	−0.0034 (5)
C5	0.0208 (6)	0.0255 (7)	0.0281 (7)	−0.0030 (5)	0.0097 (5)	−0.0006 (6)
C6	0.0225 (6)	0.0219 (6)	0.0176 (6)	−0.0010 (5)	0.0069 (5)	0.0010 (5)
C7	0.0162 (6)	0.0214 (6)	0.0175 (6)	0.0003 (5)	0.0054 (5)	0.0018 (5)
C8	0.0148 (6)	0.0219 (6)	0.0167 (6)	0.0003 (5)	0.0058 (4)	0.0011 (5)
C9	0.0200 (6)	0.0207 (6)	0.0211 (6)	0.0002 (5)	0.0091 (5)	0.0018 (5)
C10	0.0220 (6)	0.0220 (6)	0.0253 (7)	−0.0025 (5)	0.0118 (5)	−0.0027 (5)
C11	0.0286 (7)	0.0300 (7)	0.0187 (7)	−0.0033 (5)	0.0116 (6)	−0.0017 (5)
C12	0.0298 (7)	0.0260 (7)	0.0201 (7)	−0.0009 (6)	0.0095 (5)	0.0051 (5)
C13	0.0202 (6)	0.0200 (6)	0.0214 (7)	0.0011 (5)	0.0071 (5)	0.0010 (5)

Geometric parameters (Å, º)

S1—O2	1.4304 (10)	C4—C5	1.389 (2)
S1—O1	1.4415 (10)	C4—H4	0.97 (2)
S1—N1	1.6567 (12)	C5—C6	1.395 (2)
S1—C1	1.7602 (13)	C5—H5	0.959 (9)
N1—N2	1.3920 (15)	C6—H6	0.953 (9)
N1—H1N	0.85 (1)	C7—C8	1.4607 (17)
N2—C7	1.2874 (17)	C7—H7	0.948 (9)
O3—C13	1.3733 (16)	C8—C13	1.4051 (18)
O3—H3O	0.85 (1)	C8—C9	1.4090 (18)
O4—C10	1.3677 (16)	C9—C10	1.3870 (19)
O4—H4O	0.85 (1)	C9—H9	0.953 (9)
C1—C2	1.3963 (18)	C10—C11	1.397 (2)
C1—C6	1.3976 (18)	C11—C12	1.382 (2)
C2—C3	1.3904 (19)	C11—H11	0.943 (9)
C2—H2	0.958 (9)	C12—C13	1.3928 (19)
C3—C4	1.392 (2)	C12—H12	0.950 (9)
C3—H3	0.951 (9)		
O2—S1—O1	120.59 (6)	C6—C5—H5	118.4 (11)
O2—S1—N1	107.38 (6)	C5—C6—C1	118.40 (12)
O1—S1—N1	103.14 (6)	C5—C6—H6	118.2 (10)
O2—S1—C1	108.44 (6)	C1—C6—H6	123.4 (10)

O1—S1—C1	108.51 (6)	N2—C7—C8	118.90 (12)
N1—S1—C1	108.14 (6)	N2—C7—H7	124.2 (10)
N2—N1—S1	112.33 (9)	C8—C7—H7	116.9 (10)
N2—N1—H1N	118.3 (13)	C13—C8—C9	118.90 (12)
S1—N1—H1N	110.4 (13)	C13—C8—C7	121.83 (12)
C7—N2—N1	118.47 (11)	C9—C8—C7	119.28 (12)
C13—O3—H3O	110.2 (17)	C10—C9—C8	120.55 (12)
C10—O4—H4O	110.5 (16)	C10—C9—H9	119.8 (11)
C2—C1—C6	121.94 (12)	C8—C9—H9	119.6 (11)
C2—C1—S1	118.65 (10)	O4—C10—C9	123.53 (13)
C6—C1—S1	119.40 (10)	O4—C10—C11	116.96 (12)
C3—C2—C1	118.80 (12)	C9—C10—C11	119.50 (13)
C3—C2—H2	120.7 (11)	C12—C11—C10	120.81 (13)
C1—C2—H2	120.5 (11)	C12—C11—H11	121.8 (14)
C2—C3—C4	119.77 (13)	C10—C11—H11	117.1 (14)
C2—C3—H3	118.6 (11)	C11—C12—C13	119.98 (13)
C4—C3—H3	121.6 (11)	C11—C12—H12	121.0 (12)
C5—C4—C3	121.10 (13)	C13—C12—H12	118.9 (12)
C5—C4—H4	119.2 (12)	O3—C13—C12	118.16 (12)
C3—C4—H4	119.7 (12)	O3—C13—C8	121.59 (12)
C4—C5—C6	119.98 (13)	C12—C13—C8	120.25 (12)
C4—C5—H5	121.6 (11)		

O2—S1—N1—N2	−50.34 (10)	S1—C1—C6—C5	179.56 (10)
O1—S1—N1—N2	−178.71 (8)	N1—N2—C7—C8	−179.90 (10)
C1—S1—N1—N2	66.48 (10)	N2—C7—C8—C13	2.14 (19)
S1—N1—N2—C7	−163.01 (10)	N2—C7—C8—C9	−177.89 (12)
O2—S1—C1—C2	27.78 (12)	C13—C8—C9—C10	0.28 (19)
O1—S1—C1—C2	160.41 (10)	C7—C8—C9—C10	−179.68 (12)
N1—S1—C1—C2	−88.36 (11)	C8—C9—C10—O4	178.20 (12)
O2—S1—C1—C6	−152.52 (10)	C8—C9—C10—C11	−1.0 (2)
O1—S1—C1—C6	−19.89 (12)	O4—C10—C11—C12	−178.16 (14)
N1—S1—C1—C6	91.34 (11)	C9—C10—C11—C12	1.0 (2)
C6—C1—C2—C3	−0.1 (2)	C10—C11—C12—C13	−0.5 (2)
S1—C1—C2—C3	179.64 (10)	C11—C12—C13—O3	179.51 (13)
C1—C2—C3—C4	0.8 (2)	C11—C12—C13—C8	−0.2 (2)
C2—C3—C4—C5	−0.7 (2)	C9—C8—C13—O3	−179.42 (12)
C3—C4—C5—C6	−0.1 (2)	C7—C8—C13—O3	0.5 (2)
C4—C5—C6—C1	0.8 (2)	C9—C8—C13—C12	0.32 (19)
C2—C1—C6—C5	−0.8 (2)	C7—C8—C13—C12	−179.71 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*O*···N2	0.85 (1)	1.82 (2)	2.562 (2)	145 (2)

O4—H4*O*···O3^{i}	0.85 (1)	1.85 (1)	2.698 (2)	175 (2)
N1—H1*N*···O1^{ii}	0.85 (1)	2.05 (1)	2.897 (2)	171 (2)

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