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1,2-Bis(3-hydroxybenzylidene)diazane

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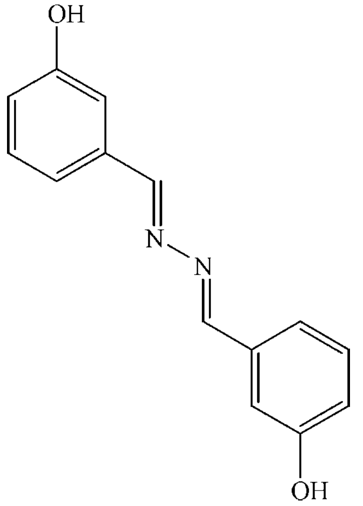
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; R factor = 0.064; wR factor = 0.185; data-to-parameter ratio = 13.0.

The asymmetric unit of the title compound, $C_{14}H_{12}N_2O_2$, which was synthesized unexpectedly by refluxing an ethanolic solution of isonicotinic hydrazide and 3-hydroxybenzaldehyde, contains one half-molecule with the center of the N—N bond lying on a crystallographic center of inversion. In the crystal structure, molecules are linked by intermolecular O—H···N hydrogen bonds into an infinite layer structure parallel to (110).

Related literature

For general background to salicyclic aldehyde complexes, see: Zelewsky & von Knof (1999); Alam *et al.* (2003).



Experimental

Crystal data

$C_{14}H_{12}N_2O_2$
M_r = 240.26
Monoclinic, $P2_1/n$
a = 4.883 (2) Å
b = 8.212 (3) Å
c = 14.575 (6) Å
β = 95.267 (6)°
V = 582.0 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 K
0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001) $T_{\min}$ = 0.989, $T_{\max}$ = 0.993
4234 measured reflections
1079 independent reflections
814 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.185
S = 1.00
1079 reflections
83 parameters
H-atom parameters not refined
$\Delta\rho_{\max}$ = 0.62 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
O1—H2···N1$^{\rm i}$	0.82	2.03	2.811 (3)	159

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2128).

References

Alam, M. A., Nethaji, M. & Ray, M. (2003). *Angew. Chem. Int. Ed.* **42**, 1940–1942.
Bruker (2001). *SAINT-Plus* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2004). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Zelewsky, A. & von Knof, U. (1999). *Angew. Chem. Int. Ed.* **38**, 302–322.

supplementary materials

Acta Cryst. (2009). E**65**, o1929 [**doi:10.1107/S1600536809027652**]

1,2-Bis(3-hydroxybenzylidene)diazane

L. Zhu and **X.-H. Zhao**

Comment

The synthesis of complexes consisting of ligands derived from salicylic aldehyde has attracted continuous research interest not only with regard to their appealing structural and topological novelty, but also due to their unusual optical, electronic, magnetic and catalytic properties as well as their potential medical application (Zelewsky *et al.* 1999; Alam *et al.* 2003). In the present paper, we describe the synthesis and structural characterization of *N*,*N*'-di(3-hydroxybenzylidene)-hydrazine.

As shown in Fig. 1, the asymmetrical unit contains one half of the molecule. The center of the N-N bond represents a crystallographic center of inversion. One intermolecular hydrogen bond O(1)—H(2)···N(1) (2.811 (3) Å) is observed in the crystal structure leading to infinite layers of molecules (Fig. 2).

Experimental

An ethanolic solution of isonicotinic hydrazide (10 mmol) and 3-hydroxybenzaldehyde (10 mmol) refluxed for five hours. After filtration a yellow powder was obtained. Suitable crystals for X-ray diffraction were obtained by recrystallization from dichloromethane. Anal. Calc. for $C_{14}H_{12}N_2O_2$: C 69.92, H 4.99, N 9.99%; Found: C 69.89, H 4.79, N 9.78.

Refinement

All H atoms were placed in calculated positions with C—H = 0.93Å and refined as riding with $U_{\mathrm{iso}}(\mathrm{H}) = 1.2U_{\mathrm{eq}}(\mathrm{carrier})$. The H atom of the hydroxy group was located from difference density maps and was refined with a distance restraint of d(O–H) = 0.82 (1) Å.

Figures

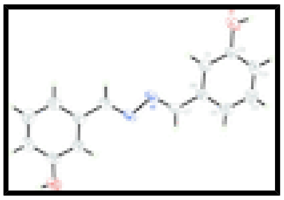

Fig. 1. The molecular structure of (I), drawn with 50% probability displacement ellipsoids.

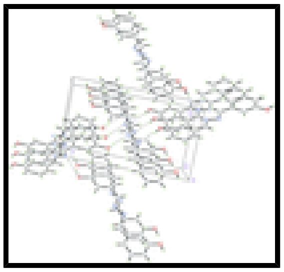

Fig. 2. Three-dimensional network formed by hydrogen bonds (dashed lines).

1,2-Bis(3-hydroxybenzylidene)diazane

Crystal data

$C_{14}H_{12}N_2O_2$

$M_r = 240.26$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 4.883$ (2) Å

$b = 8.212$ (3) Å

$c = 14.575$ (6) Å

$\beta = 95.267$ (6)°

$V = 582.0$ (4) Å^3

$Z = 2$

$F_{000} = 252$

$D_x = 1.371$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1079 reflections

$\theta = 2.8$–25.5°

$\mu = 0.09$ mm^{-1}

$T = 295$ K

Block, yellow

0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 295$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2001)

$T_{min} = 0.989$, $T_{max} = 0.993$

4234 measured reflections

1079 independent reflections

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

$R_{int} = 0.039$

$\theta_{max} = 25.5$°

$\theta_{min} = 2.8$°

$h = -5 \rightarrow 5$

$k = -9 \rightarrow 9$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.064$

$wR(F^2) = 0.185$

$S = 1.00$

1079 reflections

83 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters not refined

$w = 1/[\sigma^2(F_o^2) + (0.103P)^2 + 0.3322P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.62$ e Å^{-3}

$\Delta\rho_{min} = -0.23$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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 (Å^2)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
C1	0.3137 (6)	0.2100 (3)	0.82062 (17)	0.0364 (7)
H3	0.1725	0.1396	0.7996	0.044*
C2	0.4468 (6)	0.2997 (3)	0.75848 (18)	0.0380 (7)
C3	0.6607 (6)	0.4021 (4)	0.7888 (2)	0.0434 (8)
H6	0.7546	0.4600	0.7467	0.052*
C4	0.7340 (6)	0.4175 (4)	0.8822 (2)	0.0469 (8)
H9	0.8749	0.4882	0.9031	0.056*
C5	0.6000 (6)	0.3292 (4)	0.94433 (19)	0.0414 (8)
H7	0.6516	0.3401	1.0070	0.050*
C6	0.3891 (6)	0.2240 (3)	0.91470 (17)	0.0342 (7)
C7	0.2426 (6)	0.1379 (3)	0.98224 (18)	0.0367 (7)
H4	0.2836	0.1634	1.0441	0.044*
N1	0.0615 (5)	0.0295 (3)	0.96142 (14)	0.0358 (6)
O1	0.3545 (6)	0.2849 (3)	0.66792 (14)	0.0637 (8)
H2	0.4174	0.3590	0.6386	0.096*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0469 (16)	0.0311 (14)	0.0322 (15)	0.0014 (12)	0.0096 (12)	0.0011 (11)
C2	0.0528 (17)	0.0348 (15)	0.0280 (14)	0.0046 (13)	0.0126 (12)	0.0034 (11)
C3	0.0420 (16)	0.0442 (17)	0.0463 (18)	0.0016 (13)	0.0157 (13)	0.0103 (14)
C4	0.0448 (17)	0.0431 (17)	0.0520 (18)	−0.0014 (14)	−0.0003 (14)	0.0064 (14)
C5	0.0475 (17)	0.0418 (17)	0.0343 (15)	0.0059 (14)	0.0004 (13)	0.0027 (12)
C6	0.0413 (15)	0.0338 (15)	0.0286 (13)	0.0092 (12)	0.0095 (11)	0.0029 (11)
C7	0.0501 (17)	0.0369 (16)	0.0241 (13)	0.0076 (13)	0.0085 (12)	0.0016 (11)
N1	0.0514 (14)	0.0354 (12)	0.0228 (11)	0.0081 (11)	0.0150 (9)	0.0040 (9)
O1	0.105 (2)	0.0549 (15)	0.0329 (12)	−0.0206 (14)	0.0170 (12)	0.0046 (10)

Geometric parameters (Å, °)

C1—C2	1.376 (4)	C4—H9	0.9300
C1—C6	1.392 (4)	C5—C6	1.382 (4)
C1—H3	0.9300	C5—H7	0.9300
C2—O1	1.360 (3)	C6—C7	1.453 (4)
C2—C3	1.382 (4)	C7—N1	1.272 (4)
C3—C4	1.382 (4)	C7—H4	0.9300

C3—H6	0.9300	N1—N1^{i}	1.409 (4)
C4—C5	1.372 (4)	O1—H2	0.8200
C2—C1—C6	120.4 (3)	C4—C5—C6	120.7 (3)
C2—C1—H3	119.8	C4—C5—H7	119.6
C6—C1—H3	119.8	C6—C5—H7	119.6
O1—C2—C1	117.2 (3)	C5—C6—C1	118.8 (3)
O1—C2—C3	122.5 (3)	C5—C6—C7	119.4 (2)
C1—C2—C3	120.3 (3)	C1—C6—C7	121.7 (3)
C2—C3—C4	119.4 (3)	N1—C7—C6	123.6 (2)
C2—C3—H6	120.3	N1—C7—H4	118.2
C4—C3—H6	120.3	C6—C7—H4	118.2
C5—C4—C3	120.4 (3)	C7—N1—N1^{i}	112.8 (3)
C5—C4—H9	119.8	C2—O1—H2	109.5
C3—C4—H9	119.8		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H2···N1^{ii}	0.82	2.03	2.811 (3)	159

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

Fig. 1

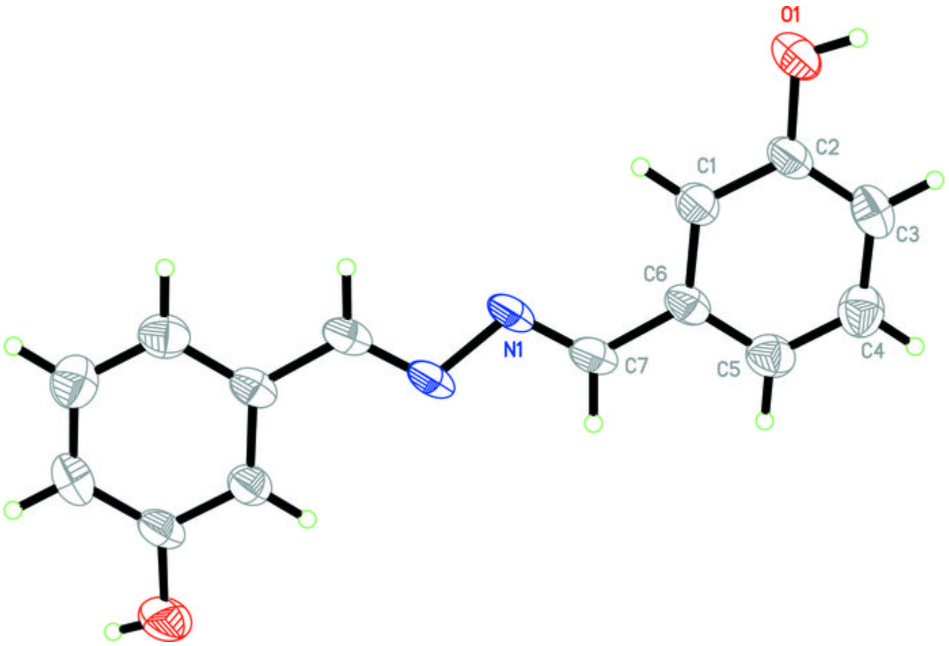

Fig. 2