Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-4-Hydroxy-*N*′-(4-nitrobenzylidene)-benzohydrazide

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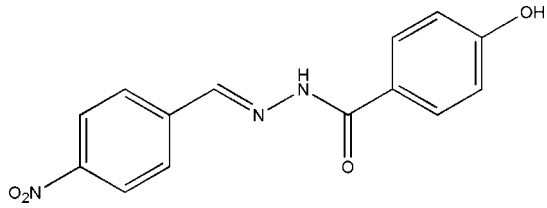
Received 22 May 2009; accepted 26 May 2009

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.111; data-to-parameter ratio = 14.5.

The molecule of the title compound, $C_{14}H_{11}N_3O_4$, is approximately planar, the dihedral angle between the planes of the two substituted benzene rings being 2.54 (7)°. The molecule exists in a *trans* configuration with respect to the central methylidene unit. In the crystal structure, molecules are linked through intermolecular O—H···O, N—H···O and C—H···O hydrogen bonds, forming layers parallel to (101). The O/N—H···O and C—H···O interactions form a pair of bifurcated acceptor bonds involving the cabonyl/nitro O atom, generating an $R_2^1(6)$ motif.

Related literature

For the biological activity of hydrazones, see: Zhong *et al.* (2007); Raj *et al.* (2007); Jimenez-Pulido *et al.* (2008). For related structures, see: Ban & Li (2008*a*,*b*); Li & Ban (2009*a*,*b*); Yehye *et al.* (2008); Fun *et al.* (2008*a*,*b*); Yang *et al.* (2008); Ejsmont *et al.* (2008).



Experimental

Crystal data

$C_{14}H_{11}N_3O_4$
M_r = 285.26
Monoclinic, $P2_1/n$
a = 7.659 (1) Å
b = 13.587 (2) Å
c = 12.561 (2) Å
β = 92.784 (5)°
V = 1305.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 K
0.20 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.979, T_{max} = 0.981
7862 measured reflections
2835 independent reflections
2109 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.111
S = 1.04
2835 reflections
195 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.82	2.02	2.8131 (15)	164
N2—H2B···O4^{ii}	0.90 (1)	2.22 (1)	3.0513 (17)	155 (2)
C7—H7···O4^{ii}	0.93	2.41	3.235 (2)	148
C13—H13···O1^{i}	0.93	2.35	3.0713 (19)	134

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; 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*.

The authors acknowledge financial support by the Research Foundation of Liaoning Province (grant No. 2008470).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2812).

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supporting information

Acta Cryst. (2009). E**65**, o1466 [doi:10.1107/S1600536809020066]

(*E*)-4-Hydroxy-*N*′-(4-nitrobenzylidene)benzohydrazide

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S1. Comment

Schiff bases derived from the condensation of aldehydes with hydrazides have been demonstrated to possess excellent biological activities (Zhong *et al.*, 2007; Raj *et al.*, 2007; Jimenez-Pulido *et al.*, 2008). Due to the easy synthesis of such compounds, a great deal of Schiff bases have been synthesized and structurally characterized (Yehye *et al.*, 2008; Fun *et al.*, 2008a,b; Yang *et al.*, 2008; Ejsmont *et al.*, 2008). Recently, we have reported a few such compounds (Ban & Li, 2008a,b; Li & Ban, 2009a,b). We report here the crystal structure of the title new compound.

The title Schiff base molecule (Fig. 1) is nearly planar, with the dihedral angle between the two benzene rings being 2.54 (7)°. The molecule exists in a *trans* configuration with respect to the central methylidene (C7═N1) unit. The N2—N1—C7—C1 torsion angle is 179.12 (14)°.

In the crystal structure, the molecules are linked through intermolecular O—H···O and N—H···O hydrogen bonds (Table 1), forming layers parallel to the (101) (Fig. 2). Within the layer, C—H···O hydrogen bonds are also observed.

S2. Experimental

The title compound was prepared by refluxing 4-nitrobenzaldehyde (1.0 mol) with 4-hydroxybenzohydrazide (1.0 mol) in methanol (100 ml). Excess methanol was removed from the mixture by distillation. A colourless solid product was filtered, and washed three times with methanol. Colourless block-shaped crystals of the title compound were obtained from a methanol solution by slow evaporation in air.

S3. Refinement

Atom H2B was located in a difference Fourier map and refined isotropically, with the N–H distance restrained to 0.90 (1)Å and U_{iso} fixed at 0.08 Å^2. The remaining H atoms were placed in calculated positions (C–H = 0.93 Å and O–H = 0.82 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$ and $1.5U_{eq}(O)$.

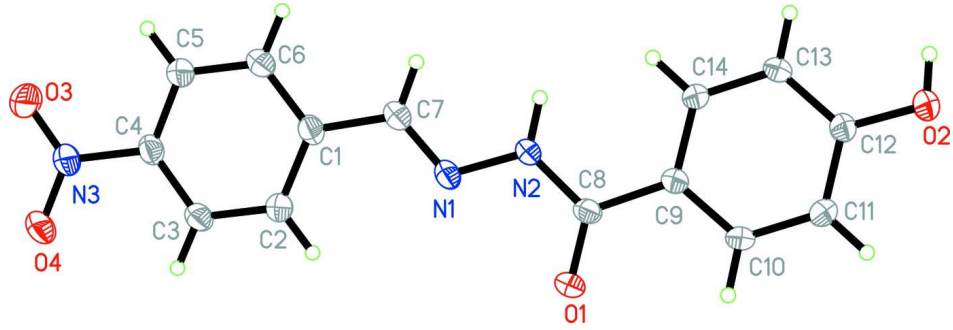

Figure 1

The molecular structure of the title compound, showing 30% probability displacement ellipsoids for the non-hydrogen atoms.

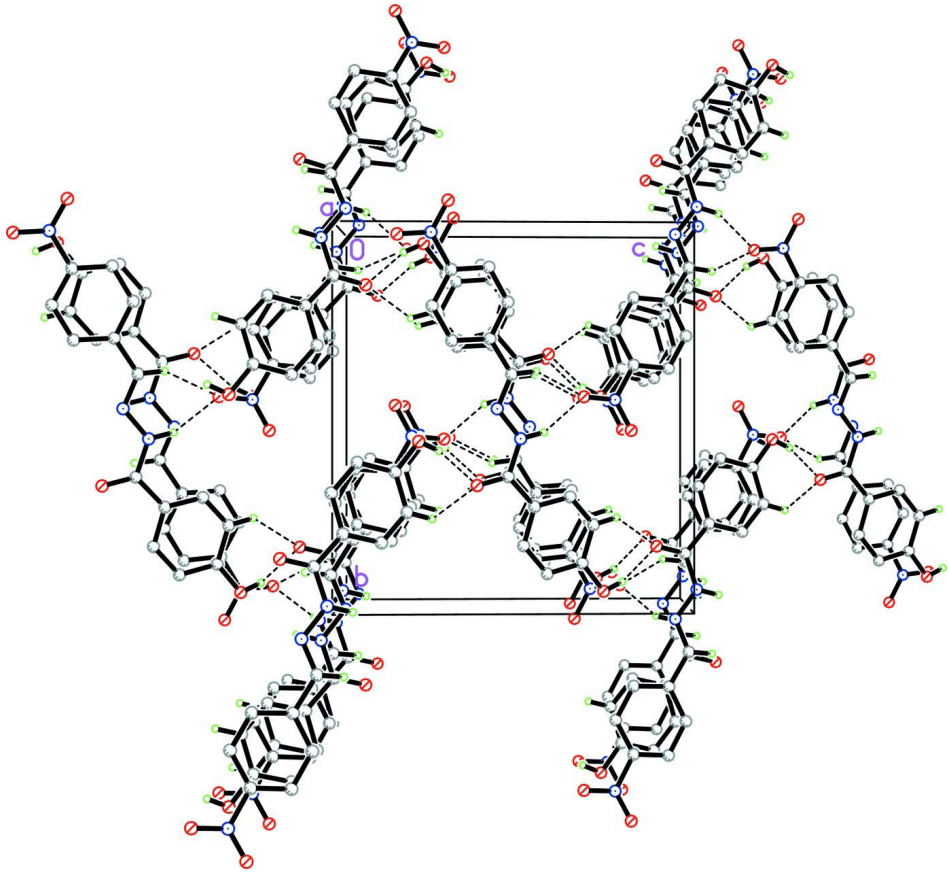

Figure 2

The packing diagram of the title compound, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

(*E*)-4-Hydroxy-*N*′-(4-nitrobenzylidene)benzohydrazide

Crystal data

$C_{14}H_{11}N_3O_4$
$M_r = 285.26$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.659$ (1) Å
$b = 13.587$ (2) Å
$c = 12.561$ (2) Å
$\beta = 92.784$ (5)°
$V = 1305.6$ (3) Å^3
$Z = 4$
$F(000) = 592$
$D_x = 1.451$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2390 reflections
$\theta = 3.0$–30.5°
$\mu = 0.11$ mm^{-1}
$T = 298$ K
Block, colourless
0.20 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.979$, $T_{max} = 0.981$
7862 measured reflections
2835 independent reflections
2109 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.2°$
$h = -9 \rightarrow 9$
$k = -17 \rightarrow 15$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.111$
$S = 1.04$
2835 reflections
195 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 0.3196P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0104 (15)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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_{iso}*/U_{eq}
N1	0.20673 (17)	0.97706 (9)	1.04460 (10)	0.0410 (3)
N2	0.26363 (19)	1.05171 (9)	0.98149 (10)	0.0431 (3)
N3	0.0157 (2)	0.54652 (10)	1.21921 (11)	0.0504 (4)
O1	0.18428 (15)	1.16731 (8)	1.09824 (8)	0.0472 (3)
O2	0.51173 (18)	1.44263 (8)	0.75115 (9)	0.0541 (3)
H2	0.5548	1.4194	0.6982	0.081*
O3	0.0321 (2)	0.46801 (10)	1.17422 (12)	0.0883 (6)
O4	−0.04623 (18)	0.55386 (9)	1.30667 (10)	0.0611 (4)
C1	0.1672 (2)	0.80400 (11)	1.06387 (12)	0.0391 (4)
C2	0.1011 (2)	0.80979 (11)	1.16524 (12)	0.0423 (4)
H2A	0.0898	0.8707	1.1980	0.051*
C3	0.0527 (2)	0.72557 (12)	1.21665 (12)	0.0418 (4)
H3	0.0085	0.7288	1.2842	0.050*
C4	0.0707 (2)	0.63608 (11)	1.16619 (12)	0.0402 (4)
C5	0.1366 (2)	0.62796 (12)	1.06648 (13)	0.0455 (4)
H5	0.1483	0.5668	1.0344	0.055*
C6	0.1845 (2)	0.71259 (12)	1.01580 (12)	0.0450 (4)
H6	0.2290	0.7087	0.9484	0.054*
C7	0.2198 (2)	0.89103 (11)	1.00586 (13)	0.0439 (4)
H7	0.2642	0.8835	0.9387	0.053*
C8	0.25098 (19)	1.14649 (11)	1.01458 (11)	0.0354 (3)
C9	0.32220 (19)	1.22217 (11)	0.94326 (11)	0.0348 (3)
C10	0.3187 (2)	1.32057 (11)	0.97329 (12)	0.0404 (4)

H10	0.2734	1.3378	1.0381	0.048*
C11	0.3812 (2)	1.39297 (11)	0.90850 (13)	0.0447 (4)
H11	0.3772	1.4585	0.9298	0.054*
C12	0.4503 (2)	1.36889 (11)	0.81144 (12)	0.0390 (4)
C13	0.4543 (2)	1.27096 (11)	0.78040 (12)	0.0410 (4)
H13	0.4995	1.2538	0.7156	0.049*
C14	0.3915 (2)	1.19946 (11)	0.84559 (12)	0.0403 (4)
H14	0.3952	1.1340	0.8240	0.048*
H2B	0.303 (3)	1.0358 (15)	0.9176 (10)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0486 (8)	0.0377 (7)	0.0377 (7)	−0.0014 (6)	0.0127 (6)	0.0081 (6)
N2	0.0616 (9)	0.0335 (7)	0.0359 (7)	−0.0021 (6)	0.0200 (6)	0.0028 (5)
N3	0.0650 (10)	0.0397 (8)	0.0477 (8)	−0.0055 (7)	0.0146 (7)	0.0081 (6)
O1	0.0630 (7)	0.0441 (6)	0.0364 (6)	0.0005 (5)	0.0217 (5)	−0.0009 (5)
O2	0.0760 (9)	0.0361 (6)	0.0525 (7)	−0.0056 (6)	0.0280 (6)	0.0047 (5)
O3	0.1579 (16)	0.0359 (7)	0.0749 (10)	−0.0156 (9)	0.0465 (10)	−0.0013 (7)
O4	0.0814 (9)	0.0505 (7)	0.0537 (7)	−0.0059 (6)	0.0289 (7)	0.0118 (6)
C1	0.0401 (8)	0.0381 (8)	0.0395 (8)	−0.0015 (6)	0.0068 (6)	0.0070 (6)
C2	0.0511 (10)	0.0350 (8)	0.0413 (9)	−0.0008 (7)	0.0087 (7)	0.0012 (6)
C3	0.0477 (9)	0.0425 (9)	0.0358 (8)	0.0001 (7)	0.0103 (7)	0.0051 (7)
C4	0.0440 (9)	0.0364 (8)	0.0408 (8)	−0.0029 (7)	0.0073 (7)	0.0076 (6)
C5	0.0563 (10)	0.0368 (8)	0.0446 (9)	−0.0019 (7)	0.0129 (8)	0.0002 (7)
C6	0.0546 (10)	0.0436 (9)	0.0379 (8)	−0.0008 (7)	0.0141 (7)	0.0029 (7)
C7	0.0526 (10)	0.0418 (9)	0.0384 (8)	−0.0023 (7)	0.0135 (7)	0.0047 (7)
C8	0.0375 (8)	0.0388 (8)	0.0307 (7)	0.0026 (6)	0.0091 (6)	0.0001 (6)
C9	0.0378 (8)	0.0342 (7)	0.0331 (7)	0.0017 (6)	0.0095 (6)	−0.0003 (6)
C10	0.0495 (9)	0.0379 (8)	0.0352 (8)	0.0012 (7)	0.0158 (7)	−0.0050 (6)
C11	0.0568 (10)	0.0313 (8)	0.0473 (9)	−0.0003 (7)	0.0161 (7)	−0.0051 (7)
C12	0.0432 (9)	0.0333 (7)	0.0414 (8)	−0.0014 (6)	0.0111 (7)	0.0027 (6)
C13	0.0516 (9)	0.0385 (8)	0.0345 (8)	−0.0003 (7)	0.0178 (7)	−0.0022 (6)
C14	0.0529 (9)	0.0302 (7)	0.0394 (8)	−0.0006 (6)	0.0165 (7)	−0.0034 (6)

Geometric parameters (Å, º)

N1—C7	1.272 (2)	C4—C5	1.377 (2)
N1—N2	1.3714 (17)	C5—C6	1.373 (2)
N2—C8	1.3581 (19)	C5—H5	0.93
N2—H2B	0.899 (9)	C6—H6	0.93
N3—O3	1.2164 (18)	C7—H7	0.93
N3—O4	1.2219 (17)	C8—C9	1.4848 (19)
N3—C4	1.4590 (19)	C9—C10	1.390 (2)
O1—C8	1.2237 (16)	C9—C14	1.395 (2)
O2—C12	1.3539 (17)	C10—C11	1.377 (2)
O2—H2	0.82	C10—H10	0.93
C1—C6	1.390 (2)	C11—C12	1.392 (2)

C1—C2	1.395 (2)	C11—H11	0.93
C1—C7	1.456 (2)	C12—C13	1.387 (2)
C2—C3	1.374 (2)	C13—C14	1.373 (2)
C2—H2A	0.93	C13—H13	0.93
C3—C4	1.381 (2)	C14—H14	0.93
C3—H3	0.93		

C7—N1—N2	115.18 (12)	C1—C6—H6	119.5
C8—N2—N1	119.67 (12)	N1—C7—C1	121.73 (14)
C8—N2—H2B	122.3 (14)	N1—C7—H7	119.1
N1—N2—H2B	117.9 (14)	C1—C7—H7	119.1
O3—N3—O4	122.86 (14)	O1—C8—N2	121.40 (13)
O3—N3—C4	118.74 (14)	O1—C8—C9	122.61 (13)
O4—N3—C4	118.40 (14)	N2—C8—C9	115.99 (12)
C12—O2—H2	109.5	C10—C9—C14	117.70 (13)
C6—C1—C2	119.55 (14)	C10—C9—C8	119.34 (13)
C6—C1—C7	118.28 (14)	C14—C9—C8	122.96 (13)
C2—C1—C7	122.18 (14)	C11—C10—C9	120.99 (14)
C3—C2—C1	120.02 (14)	C11—C10—H10	119.5
C3—C2—H2A	120.0	C9—C10—H10	119.5
C1—C2—H2A	120.0	C10—C11—C12	120.51 (14)
C2—C3—C4	118.84 (14)	C10—C11—H11	119.7
C2—C3—H3	120.6	C12—C11—H11	119.7
C4—C3—H3	120.6	O2—C12—C13	122.66 (13)
C5—C4—C3	122.48 (14)	O2—C12—C11	118.25 (13)
C5—C4—N3	118.35 (14)	C13—C12—C11	119.09 (14)
C3—C4—N3	119.16 (13)	C14—C13—C12	119.87 (13)
C6—C5—C4	118.20 (15)	C14—C13—H13	120.1
C6—C5—H5	120.9	C12—C13—H13	120.1
C4—C5—H5	120.9	C13—C14—C9	121.84 (14)
C5—C6—C1	120.91 (14)	C13—C14—H14	119.1
C5—C6—H6	119.5	C9—C14—H14	119.1

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1^{i}	0.82	2.02	2.8131 (15)	164
N2—H2*B*···O4^{ii}	0.90 (1)	2.22 (1)	3.0513 (17)	155 (2)
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Symmetry codes: (i) $x+1/2, -y+5/2, z-1/2$; (ii) $x+1/2, -y+3/2, z-1/2$.