organic compounds

4-Hydroxy-N'-(3-nitrobenzylidene)-benzohydrazide

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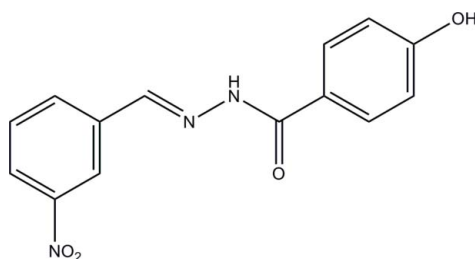Correspondence e-mail: houjinlong09@163.com

The title compound, $C_{14}H_{11}N_3O_4$, was obtained by a condensation reaction between 3-nitrobenzaldehyde and 4-hydroxybenzohydrazide. The whole molecule is approximately planar, with a dihedral angle of 9.2 (3)° between the benzene rings. The molecule displays an E conformation about the C=N bond. In the crystal, molecules are linked via N—H⋯O, O—H⋯O and O—H⋯N hydrogen bonds, generating sheets parallel to the bc plane.

Related literature

For the biological properties of hydrazone compounds, see: Cukurovali et al. (2006); Karthikeyan et al. (2006); Kucukguzel et al. (2006). For related hydrazone compounds, see: Hou (2009); Mohd Lair et al. (2009); Fun et al. (2008); Zhang et al. (2009); Khaledi et al. (2008). For standard bond lengths, see: Allen et al. (1987).

Experimental

Crystal data

$C_{14}H_{11}N_3O_4$	V = 1286.4 (5) Å^3
M_r = 285.26	Z = 4
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 8.018 (2) Å	μ = 0.11 mm^{-1}
b = 11.156 (2) Å	T = 298 K
c = 14.389 (2) Å	0.21 × 0.20 × 0.17 mm
β = 91.773 (2)°	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	9218 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	2386 independent reflections
T_{min} = 0.977, T_{max} = 0.981	2020 reflections with $I > 2\sigma(I)$
	R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.108	
S = 1.10	$\Delta\rho_{max}$ = 0.13 e Å^{-3}
2386 reflections	$\Delta\rho_{min}$ = −0.23 e Å^{-3}
194 parameters	
1 restraint	

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N2—H2A⋯O1^i	0.90 (1)	2.49 (2)	3.0406 (18)	120 (1)
N2—H2A⋯O4^{ii}	0.90 (1)	2.32 (1)	3.0360 (17)	137 (2)
O4—H4⋯N1^{iii}	0.82	2.63	3.0495 (17)	114
O4—H4⋯O3^{iii}	0.82	2.08	2.8929 (16)	173

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

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.

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supplementary materials

4-Hydroxy-*N*′-(3-nitrobenzylidene)benzohydrazide

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Comment

Hydrazones derived from the condensation reactions of hydrazides with aldehydes show excellent biological properties (Cukurovali *et al.*, 2006; Karthikeyan *et al.*, 2006; Kucukguzel *et al.*, 2006). In the last few years, a great deal of hydrazone compounds have been reported for their crystal structures see (Hou, 2009; Mohd Lair *et al.*, 2009; Fun *et al.*, 2008; Zhang *et al.*, 2009; Khaledi *et al.*, 2008). In this paper, the title new compound, derived from the condensation reaction of 3-nitrobenzaldehyde and 4-hydroxybenzohydrazide was synthesized and structurally characterized.

The molecular structure of the compound is shown in Fig. 1. The whole molecule of the compound is approximately coplanar, with the dihedral angle between the mean planes through the two benzene rings of 9.2 (3)°. The molecule displays an *E* configuration about the C=N bond. All the bond lengths are within normal ranges (Allen *et al.*, 1987). In the crystal, molecules are linked *via* N–H⋯O hydrogen bonds (Table 1), generating two-dimensional sheets (Fig. 2).

Experimental

3-Nitrobenzaldehyde (1.0 mmol, 151 mg) and 4-hydroxybenzohydrazide (1.0 mmol, 152 mg) were mixed and refluxed with stirring for two hours. Yellow single crystals were formed after slow evaporation of the solution in air for a week.

Refinement

H2A was located in a difference Fourier map and refined isotropically, with the N–H distance restrained to 0.90 (1) Å. The other H atoms were placed in idealized positions and constrained to ride on their parent atoms with C–H distances of 0.93 Å, O–H distance of 0.82 Å, and with $U_{iso}(H)$ set at $1.2U_{eq}(C)$ and $1.5U_{eq}(O)$.

Computing details

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT* (Bruker, 1998); 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* (Sheldrick, 2008).
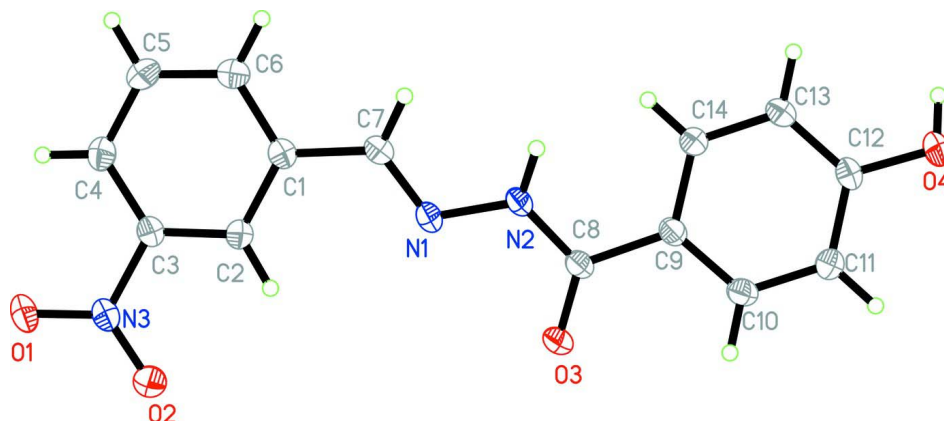
Figure 1

Molecular structure with displacement ellipsoids drawn at 30% probability for non-H atoms.
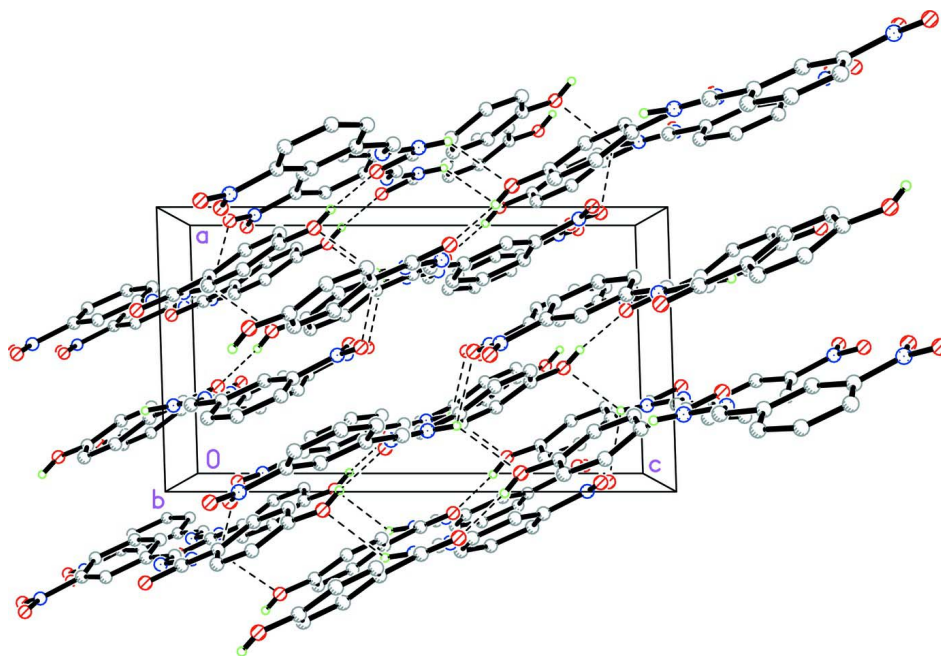
Figure 2

Molecular packing diagram, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

4-Hydroxy-*N*′-(3-nitrobenzylidene)benzohydrazide

Crystal data

$C_{14}H_{11}N_3O_4$	$F(000) = 592$
$M_r = 285.26$	$D_x = 1.473$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.018$ (2) Å	Cell parameters from 4759 reflections
$b = 11.156$ (2) Å	$\theta = 2.8–27.0°$
$c = 14.389$ (2) Å	$\mu = 0.11$ mm^{-1}
$\beta = 91.773$ (2)°	$T = 298$ K
$V = 1286.4$ (5) Å^3	Block, yellow
$Z = 4$	$0.21 \times 0.20 \times 0.17$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	9218 measured reflections
Radiation source: fine-focus sealed tube	2386 independent reflections
Graphite monochromator	2020 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.018$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 25.5°$, $\theta_{min} = 2.9°$
$T_{min} = 0.977$, $T_{max} = 0.981$	$h = -9 \rightarrow 9$
	$k = -13 \rightarrow 13$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.035$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.108$	
$S = 1.10$	H atoms treated by a mixture of independent and constrained refinement
2386 reflections	
194 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 0.347P]$ where $P = (F_o^2 + 2F_c^2)/3$
1 restraint	
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} < 0.001$
	$\Delta\rho_{max} = 0.13$ e Å^{-3}
	$\Delta\rho_{min} = -0.23$ e Å^{-3}

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_{iso}*/U_{eq}
N1	0.18378 (15)	0.04393 (11)	0.44521 (8)	0.0361 (3)
N2	0.21274 (16)	−0.03537 (12)	0.51683 (8)	0.0372 (3)
N3	0.02101 (16)	0.30324 (12)	0.15979 (9)	0.0405 (3)
O1	−0.00751 (15)	0.39126 (11)	0.11049 (8)	0.0533 (3)
O2	−0.01799 (18)	0.20157 (11)	0.13697 (9)	0.0610 (4)
O3	0.14307 (14)	−0.19871 (10)	0.43108 (7)	0.0420 (3)
O4	0.43051 (15)	−0.43925 (11)	0.80509 (8)	0.0477 (3)
H4	0.4981	−0.4032	0.8386	0.072*
C1	0.18748 (18)	0.24891 (14)	0.39868 (10)	0.0357 (3)
C2	0.11420 (18)	0.22848 (13)	0.31117 (10)	0.0348 (3)
H2	0.0733	0.1532	0.2946	0.042*
C3	0.10429 (18)	0.32288 (13)	0.25017 (10)	0.0349 (3)
C4	0.1649 (2)	0.43600 (15)	0.27064 (12)	0.0435 (4)
H4A	0.1576	0.4974	0.2270	0.052*
C5	0.2364 (2)	0.45524 (15)	0.35766 (13)	0.0499 (4)
H5	0.2780	0.5306	0.3735	0.060*

C6	0.2463 (2)	0.36266 (15)	0.42129 (12)	0.0453 (4)
H6	0.2930	0.3767	0.4802	0.054*
C7	0.20456 (19)	0.15322 (14)	0.46738 (10)	0.0379 (4)
H7	0.2313	0.1727	0.5289	0.045*
C8	0.20226 (17)	−0.15485 (14)	0.50337 (9)	0.0329 (3)
C9	0.26711 (17)	−0.22804 (13)	0.58334 (9)	0.0331 (3)
C10	0.22900 (19)	−0.34905 (14)	0.58765 (10)	0.0389 (4)
H10	0.1649	−0.3839	0.5399	0.047*
C11	0.2843 (2)	−0.41868 (14)	0.66141 (11)	0.0415 (4)
H11	0.2574	−0.4997	0.6633	0.050*
C12	0.38050 (18)	−0.36750 (14)	0.73294 (10)	0.0358 (3)
C13	0.4214 (2)	−0.24795 (15)	0.72935 (11)	0.0447 (4)
H13	0.4862	−0.2135	0.7770	0.054*
C14	0.3661 (2)	−0.17939 (15)	0.65496 (11)	0.0446 (4)
H14	0.3956	−0.0989	0.6526	0.054*
H2A	0.231 (2)	−0.0045 (16)	0.5739 (8)	0.054*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0422 (7)	0.0365 (7)	0.0290 (6)	0.0028 (5)	−0.0068 (5)	0.0051 (5)
N2	0.0512 (7)	0.0345 (7)	0.0253 (6)	0.0020 (6)	−0.0102 (5)	0.0017 (5)
N3	0.0406 (7)	0.0410 (8)	0.0394 (7)	0.0042 (6)	−0.0070 (5)	0.0063 (6)
O1	0.0567 (7)	0.0501 (7)	0.0519 (7)	0.0071 (6)	−0.0149 (6)	0.0157 (6)
O2	0.0864 (10)	0.0441 (8)	0.0509 (7)	−0.0059 (7)	−0.0252 (7)	−0.0004 (6)
O3	0.0542 (7)	0.0405 (6)	0.0304 (5)	−0.0024 (5)	−0.0128 (5)	−0.0017 (5)
O4	0.0570 (7)	0.0467 (7)	0.0386 (6)	−0.0020 (5)	−0.0126 (5)	0.0146 (5)
C1	0.0363 (7)	0.0347 (8)	0.0357 (8)	0.0015 (6)	−0.0037 (6)	0.0021 (6)
C2	0.0366 (7)	0.0302 (8)	0.0372 (8)	0.0014 (6)	−0.0036 (6)	0.0014 (6)
C3	0.0345 (7)	0.0347 (8)	0.0353 (8)	0.0033 (6)	−0.0031 (6)	0.0026 (6)
C4	0.0497 (9)	0.0354 (9)	0.0453 (9)	−0.0014 (7)	−0.0030 (7)	0.0089 (7)
C5	0.0612 (10)	0.0340 (9)	0.0541 (10)	−0.0117 (8)	−0.0063 (8)	0.0002 (8)
C6	0.0522 (9)	0.0416 (9)	0.0415 (9)	−0.0062 (7)	−0.0093 (7)	−0.0016 (7)
C7	0.0423 (8)	0.0382 (9)	0.0324 (8)	−0.0004 (6)	−0.0091 (6)	0.0006 (6)
C8	0.0339 (7)	0.0373 (8)	0.0273 (7)	0.0000 (6)	−0.0033 (5)	0.0007 (6)
C9	0.0352 (7)	0.0359 (8)	0.0278 (7)	0.0013 (6)	−0.0038 (6)	0.0013 (6)
C10	0.0455 (8)	0.0364 (9)	0.0340 (8)	−0.0002 (6)	−0.0085 (6)	−0.0039 (6)
C11	0.0511 (9)	0.0313 (8)	0.0415 (9)	−0.0017 (7)	−0.0064 (7)	0.0029 (7)
C12	0.0374 (7)	0.0392 (9)	0.0305 (7)	0.0042 (6)	−0.0019 (6)	0.0068 (6)
C13	0.0524 (9)	0.0422 (9)	0.0382 (8)	−0.0051 (7)	−0.0178 (7)	0.0033 (7)
C14	0.0545 (9)	0.0357 (9)	0.0424 (9)	−0.0079 (7)	−0.0183 (7)	0.0062 (7)

Geometric parameters (Å, º)

N1—C7	1.270 (2)	C4—H4A	0.9300
N1—N2	1.3725 (17)	C5—C6	1.381 (2)
N2—C8	1.349 (2)	C5—H5	0.9300
N2—H2A	0.898 (9)	C6—H6	0.9300
N3—O2	1.2188 (18)	C7—H7	0.9300
N3—O1	1.2286 (17)	C8—C9	1.491 (2)

N3—C3	1.460 (2)	C9—C10	1.386 (2)
O3—C8	1.2312 (17)	C9—C14	1.392 (2)
O4—C12	1.3614 (17)	C10—C11	1.377 (2)
O4—H4	0.8200	C10—H10	0.9300
C1—C6	1.389 (2)	C11—C12	1.390 (2)
C1—C2	1.392 (2)	C11—H11	0.9300
C1—C7	1.458 (2)	C12—C13	1.375 (2)
C2—C3	1.372 (2)	C13—C14	1.378 (2)
C2—H2	0.9300	C13—H13	0.9300
C3—C4	1.381 (2)	C14—H14	0.9300
C4—C5	1.378 (2)		
C7—N1—N2	114.35 (12)	C1—C6—H6	119.5
C8—N2—N1	121.38 (12)	N1—C7—C1	121.57 (14)
C8—N2—H2A	121.2 (12)	N1—C7—H7	119.2
N1—N2—H2A	117.3 (13)	C1—C7—H7	119.2
O2—N3—O1	123.13 (14)	O3—C8—N2	122.31 (13)
O2—N3—C3	119.08 (13)	O3—C8—C9	123.39 (14)
O1—N3—C3	117.79 (13)	N2—C8—C9	114.30 (12)
C12—O4—H4	109.5	C10—C9—C14	117.92 (14)
C6—C1—C2	119.49 (14)	C10—C9—C8	119.72 (13)
C6—C1—C7	119.08 (14)	C14—C9—C8	122.36 (14)
C2—C1—C7	121.44 (14)	C11—C10—C9	121.17 (14)
C3—C2—C1	117.89 (14)	C11—C10—H10	119.4
C3—C2—H2	121.1	C9—C10—H10	119.4
C1—C2—H2	121.1	C10—C11—C12	119.81 (15)
C2—C3—C4	123.52 (14)	C10—C11—H11	120.1
C2—C3—N3	118.08 (14)	C12—C11—H11	120.1
C4—C3—N3	118.38 (13)	O4—C12—C13	122.28 (14)
C5—C4—C3	118.04 (15)	O4—C12—C11	117.83 (14)
C5—C4—H4A	121.0	C13—C12—C11	119.89 (14)
C3—C4—H4A	121.0	C12—C13—C14	119.76 (15)
C4—C5—C6	119.98 (16)	C12—C13—H13	120.1
C4—C5—H5	120.0	C14—C13—H13	120.1
C6—C5—H5	120.0	C13—C14—C9	121.43 (15)
C5—C6—C1	121.07 (15)	C13—C14—H14	119.3
C5—C6—H6	119.5	C9—C14—H14	119.3

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
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O4—H4⋯N1[iii]	0.82	2.63	3.0495 (17)	114
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Symmetry codes: (i) $x+1/2, -y+1/2, z+1/2$; (ii) $-x+1/2, y+1/2, -z+3/2$; (iii) $x+1/2, -y-1/2, z+1/2$.