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N'-(4-Hydroxybenzylidene)-4-nitrobenzohydrazide

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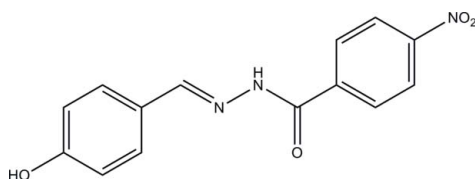
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.045; wR factor = 0.119; data-to-parameter ratio = 14.5.

The title compound, $\text{C}_{14}\text{H}_{11}\text{N}_3\text{O}_4$, was prepared by the reaction of 4-nitrobenzohydrazide with 4-hydroxybenzaldehyde. The whole molecule of the compound is approximately planar, with a mean deviation from the least-squares plane through all the non-H atoms of 0.050 (2) Å; the dihedral angle between the two benzene rings is 2.0 (2)°. In the crystal, the benzohydrazide molecules are linked through intermolecular $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming layers in the bc plane.

Related literature

For medical applications of hydrazones, see: Ajani *et al.* (2010); Zhang *et al.* (2010); Angelusiu *et al.* (2010). For related structures, see: Huang & Wu (2010); Khaledi *et al.* (2010); Zhou & Yang (2010); Ji & Lu (2010); Singh & Singh (2010); Ahmad *et al.* (2010).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{11}\text{N}_3\text{O}_4$
 $M_r = 285.26$
 Monoclinic, $P2_1/c$
 $a = 7.856$ (3) Å
 $b = 13.368$ (5) Å

$c = 12.527$ (5) Å
 $\beta = 95.748$ (4)°
 $V = 1309.0$ (9) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.11$ mm⁻¹
 $T = 298$ K

0.18 × 0.17 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2001)
 $T_{\min} = 0.981$, $T_{\max} = 0.982$

7505 measured reflections
 2822 independent reflections
 1736 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.119$
 $S = 1.04$
 2822 reflections
 194 parameters
 1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.16$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{O2}^{\text{i}}$	0.82	1.98	2.7841 (18)	166
$\text{N2}-\text{H2}\cdots\text{O4}^{\text{ii}}$	0.90 (1)	2.24 (1)	3.094 (2)	159 (2)

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

Data collection: SMART (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; 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: SJ5045).

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supplementary materials

Acta Cryst. (2010). E66, o2942 [doi:10.1107/S1600536810042364]

N'-(4-Hydroxybenzylidene)-4-nitrobenzohydrazide

C.-H. Dai and F.-L. Mao

Comment

Recently, medical applications of a number of hydrazone compounds have been reported (Ajani *et al.*, 2010; Zhang *et al.*, 2010; Angelusiu *et al.*, 2010). Structural investigations of several hydrazone derivatives have also been determined (Huang & Wu, 2010; Khaledi *et al.*, 2010; Zhou & Yang, 2010; Ji & Lu, 2010; Singh & Singh, 2010; Ahmad *et al.*, 2010). In this paper, we report the structure of the new derivative *N'*-(4-Hydroxybenzylidene)-4-nitrobenzohydrazide (I).

The whole molecule of (I) is approximately planar, with a mean deviation from the least squares plane through all 21 non-hydrogen atoms of 0.050 (2) Å; the dihedral angle between the C1...C6 and C9...C14 benzene rings is 2.0 (2)°. The bond lengths and angles are comparable to those found in the hydrazone compounds cited above. In the crystal structure, the hydrazone molecules are linked through intermolecular O1–H1...O2 and N2–H2...O4 hydrogen bonds (Table 1), to form two-dimensional layers in the *bc* plane, as shown in Fig. 2.

Experimental

The reaction of 4-nitrobenzohydrazide (0.181 g, 1 mmol) with 4-hydroxybenzaldehyde (0.122 g, 1 mmol) in 50 ml methanol at room temperature afforded the title compound. Single crystals were formed by slow evaporation of the clear solution in air.

Refinement

The amino H atom was located in a difference Fourier map and refined with N–H = 0.90 (1) Å, and $U_{\text{iso}} = 0.08 \text{ \AA}^2$. Other H atoms were positioned geometrically (C–H = 0.93 Å, O–H = 0.82 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5 U_{\text{eq}}(\text{O})$.

Figures

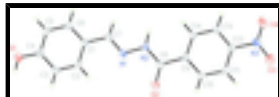


Fig. 1. The molecular structure of the title compound showing 30% probability displacement ellipsoids and the atomic numbering.

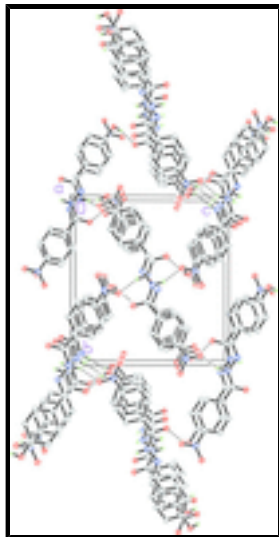


Fig. 2. Crystal packing of the title compound, viewed down the *a* axis. Intermolecular interactions are drawn as dashed lines.

***N'*-(4-Hydroxybenzylidene)-4-nitrobenzohydrazide**

Crystal data

$C_{14}H_{11}N_3O_4$

$M_r = 285.26$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 7.856$ (3) Å

$b = 13.368$ (5) Å

$c = 12.527$ (5) Å

$\beta = 95.748$ (4)°

$V = 1309.0$ (9) Å³

$Z = 4$

$F(000) = 592$

$D_x = 1.447$ Mg m⁻³

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

Cell parameters from 1632 reflections

$\theta = 2.2$ – 26.0 °

$\mu = 0.11$ mm⁻¹

$T = 298$ K

Block, yellow

$0.18 \times 0.17 \times 0.17$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

ω scans

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

$T_{\min} = 0.981$, $T_{\max} = 0.982$

7505 measured reflections

2822 independent reflections

1736 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 27.0$ °, $\theta_{\min} = 2.2$ °

$h = -9 \rightarrow 10$

$k = -17 \rightarrow 14$

$l = -16 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

$$R[F^2 > 2\sigma(F^2)] = 0.045$$

$$wR(F^2) = 0.119$$

$$S = 1.04$$

2822 reflections

194 parameters

1 restraint

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 0.1504P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.20526 (19)	-0.05033 (11)	0.04617 (11)	0.0433 (4)
N2	0.2594 (2)	0.02540 (11)	-0.01727 (11)	0.0420 (4)
N3	0.4845 (2)	0.44506 (12)	-0.22229 (13)	0.0510 (4)
O1	0.0039 (2)	-0.46112 (9)	0.25453 (11)	0.0598 (4)
H1	-0.0521	-0.4411	0.3021	0.090*
O2	0.17995 (17)	0.13969 (9)	0.09997 (10)	0.0512 (4)
O3	0.4755 (2)	0.52833 (11)	-0.18407 (13)	0.0872 (6)
O4	0.54671 (18)	0.42836 (10)	-0.30610 (10)	0.0603 (4)
C1	0.1654 (2)	-0.22332 (13)	0.07612 (14)	0.0395 (4)
C2	0.1046 (2)	-0.20626 (13)	0.17594 (14)	0.0433 (5)
H2A	0.1006	-0.1412	0.2020	0.052*
C3	0.0511 (2)	-0.28366 (13)	0.23572 (14)	0.0444 (5)
H3	0.0107	-0.2708	0.3017	0.053*
C4	0.0567 (2)	-0.38146 (13)	0.19834 (14)	0.0424 (5)
C5	0.1185 (2)	-0.39987 (13)	0.10044 (15)	0.0463 (5)
H5	0.1240	-0.4651	0.0753	0.056*
C6	0.1721 (2)	-0.32147 (13)	0.04010 (14)	0.0446 (5)
H6	0.2132	-0.3346	-0.0256	0.053*
C7	0.2197 (2)	-0.13939 (13)	0.01282 (14)	0.0422 (4)
H7	0.2649	-0.1512	-0.0519	0.051*
C8	0.2426 (2)	0.12013 (13)	0.01678 (13)	0.0372 (4)
C9	0.3070 (2)	0.20201 (12)	-0.05002 (13)	0.0352 (4)
C10	0.2986 (2)	0.29875 (13)	-0.01120 (14)	0.0454 (5)

supplementary materials

H10	0.2541	0.3099	0.0538	0.055*
C11	0.3552 (2)	0.37861 (13)	-0.06733 (15)	0.0482 (5)
H11	0.3492	0.4435	-0.0411	0.058*
C12	0.4209 (2)	0.36016 (13)	-0.16304 (14)	0.0395 (4)
C13	0.4312 (2)	0.26543 (14)	-0.20412 (14)	0.0449 (5)
H13	0.4760	0.2548	-0.2691	0.054*
C14	0.3738 (2)	0.18647 (13)	-0.14713 (14)	0.0446 (5)
H14	0.3799	0.1219	-0.1740	0.054*
H2	0.299 (3)	0.0083 (16)	-0.0797 (11)	0.080*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0554 (10)	0.0363 (9)	0.0402 (9)	-0.0019 (7)	0.0149 (7)	0.0061 (7)
N2	0.0593 (10)	0.0337 (8)	0.0360 (8)	-0.0025 (7)	0.0187 (7)	0.0034 (7)
N3	0.0596 (11)	0.0466 (10)	0.0482 (10)	-0.0082 (8)	0.0127 (8)	0.0080 (8)
O1	0.0868 (11)	0.0365 (7)	0.0615 (9)	-0.0022 (7)	0.0338 (8)	0.0061 (7)
O2	0.0688 (9)	0.0451 (8)	0.0442 (8)	0.0005 (7)	0.0273 (6)	-0.0002 (6)
O3	0.1470 (17)	0.0418 (9)	0.0802 (12)	-0.0201 (10)	0.0472 (11)	0.0006 (8)
O4	0.0700 (10)	0.0636 (9)	0.0511 (9)	-0.0088 (7)	0.0247 (7)	0.0110 (7)
C1	0.0415 (10)	0.0392 (10)	0.0385 (10)	0.0004 (8)	0.0075 (8)	0.0030 (8)
C2	0.0554 (11)	0.0332 (10)	0.0429 (10)	-0.0023 (8)	0.0127 (9)	-0.0005 (8)
C3	0.0553 (11)	0.0411 (10)	0.0390 (10)	0.0011 (9)	0.0154 (9)	-0.0003 (9)
C4	0.0467 (11)	0.0362 (10)	0.0453 (11)	0.0007 (8)	0.0100 (9)	0.0077 (9)
C5	0.0585 (12)	0.0326 (9)	0.0495 (11)	0.0012 (9)	0.0135 (9)	-0.0022 (9)
C6	0.0524 (11)	0.0432 (11)	0.0400 (10)	0.0015 (9)	0.0142 (9)	-0.0008 (9)
C7	0.0472 (11)	0.0441 (11)	0.0367 (10)	-0.0006 (9)	0.0123 (8)	0.0042 (9)
C8	0.0394 (10)	0.0405 (10)	0.0327 (9)	0.0025 (8)	0.0084 (8)	0.0015 (8)
C9	0.0367 (9)	0.0369 (9)	0.0330 (9)	0.0032 (7)	0.0083 (7)	0.0026 (8)
C10	0.0589 (12)	0.0430 (11)	0.0373 (10)	-0.0008 (9)	0.0197 (9)	-0.0045 (8)
C11	0.0632 (12)	0.0353 (10)	0.0481 (11)	-0.0029 (9)	0.0159 (10)	-0.0030 (9)
C12	0.0424 (10)	0.0371 (10)	0.0400 (10)	-0.0021 (8)	0.0088 (8)	0.0068 (8)
C13	0.0554 (11)	0.0457 (11)	0.0363 (10)	0.0012 (9)	0.0180 (9)	0.0024 (9)
C14	0.0605 (12)	0.0343 (9)	0.0417 (10)	0.0032 (9)	0.0186 (9)	-0.0013 (8)

Geometric parameters (\AA , $^\circ$)

N1—C7	1.271 (2)	C4—C5	1.386 (2)
N1—N2	1.3803 (19)	C5—C6	1.382 (2)
N2—C8	1.347 (2)	C5—H5	0.9300
N2—H2	0.899 (9)	C6—H6	0.9300
N3—O3	1.217 (2)	C7—H7	0.9300
N3—O4	1.2220 (19)	C8—C9	1.496 (2)
N3—C12	1.471 (2)	C9—C10	1.386 (2)
O1—C4	1.364 (2)	C9—C14	1.388 (2)
O1—H1	0.8200	C10—C11	1.376 (2)
O2—C8	1.2247 (19)	C10—H10	0.9300
C1—C6	1.390 (2)	C11—C12	1.374 (2)
C1—C2	1.401 (2)	C11—H11	0.9300

C1—C7	1.462 (2)	C12—C13	1.372 (2)
C2—C3	1.368 (2)	C13—C14	1.375 (2)
C2—H2A	0.9300	C13—H13	0.9300
C3—C4	1.391 (2)	C14—H14	0.9300
C3—H3	0.9300		
C7—N1—N2	117.08 (14)	C1—C6—H6	119.5
C8—N2—N1	117.50 (13)	N1—C7—C1	120.02 (15)
C8—N2—H2	124.6 (15)	N1—C7—H7	120.0
N1—N2—H2	117.9 (15)	C1—C7—H7	120.0
O3—N3—O4	123.30 (16)	O2—C8—N2	122.05 (15)
O3—N3—C12	118.13 (16)	O2—C8—C9	120.45 (15)
O4—N3—C12	118.55 (16)	N2—C8—C9	117.49 (14)
C4—O1—H1	109.5	C10—C9—C14	118.85 (15)
C6—C1—C2	118.08 (16)	C10—C9—C8	117.14 (14)
C6—C1—C7	121.71 (15)	C14—C9—C8	124.01 (15)
C2—C1—C7	120.20 (16)	C11—C10—C9	121.04 (16)
C3—C2—C1	121.08 (16)	C11—C10—H10	119.5
C3—C2—H2A	119.5	C9—C10—H10	119.5
C1—C2—H2A	119.5	C12—C11—C10	118.37 (17)
C2—C3—C4	120.32 (16)	C12—C11—H11	120.8
C2—C3—H3	119.8	C10—C11—H11	120.8
C4—C3—H3	119.8	C13—C12—C11	122.28 (16)
O1—C4—C5	118.02 (16)	C13—C12—N3	119.15 (15)
O1—C4—C3	122.60 (15)	C11—C12—N3	118.56 (16)
C5—C4—C3	119.38 (16)	C12—C13—C14	118.62 (15)
C6—C5—C4	120.15 (17)	C12—C13—H13	120.7
C6—C5—H5	119.9	C14—C13—H13	120.7
C4—C5—H5	119.9	C13—C14—C9	120.83 (16)
C5—C6—C1	120.97 (16)	C13—C14—H14	119.6
C5—C6—H6	119.5	C9—C14—H14	119.6

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots O2 ⁱ	0.82	1.98	2.7841 (18)	166
N2—H2 \cdots O4 ⁱⁱ	0.90 (1)	2.24 (1)	3.094 (2)	159 (2)

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Fig. 1

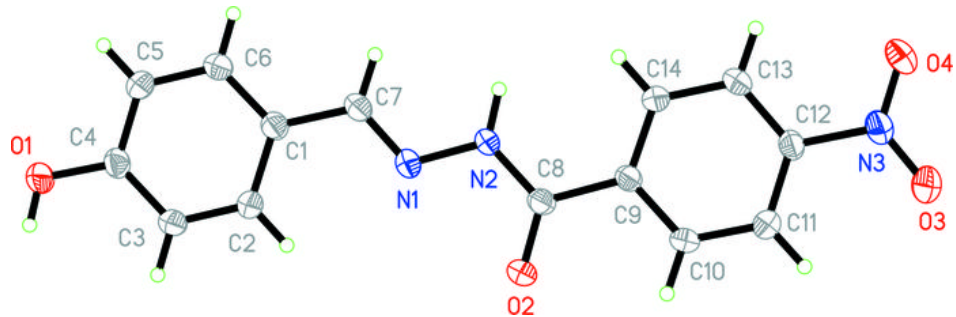


Fig. 2

