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

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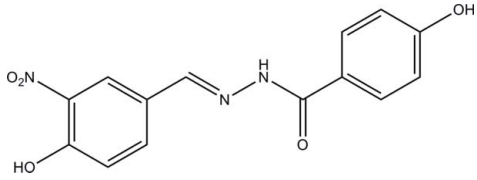
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.054; wR factor = 0.137; data-to-parameter ratio = 14.5.

The molecule of the title compound, $C_{14}H_{11}N_3O_5$, assumes an E configuration with respect to the methylidene unit. An intramolecular O—H···O hydrogen bond is present in the molecule. The dihedral angle between the mean planes of the two benzene rings is 5.46 (15)°. The crystal structure is stabilized by intermolecular O—H···O, O—H···N, and N—H···O hydrogen bonds.

Related literature

For the biological applications of hydrazone compounds, see: Ajani *et al.* (2010); Avaji *et al.* (2009); Fan *et al.* (2010); Rasras *et al.* (2010). For similar hydrazone compounds, see: Ahmad *et al.* (2010); Ban (2010); Ji & Lu (2010); Shalash *et al.* (2010).



Experimental

Crystal data

$C_{14}H_{11}N_3O_5$
M_r = 301.26
Monoclinic, $P2_1/n$
a = 8.786 (3) Å
b = 14.882 (2) Å
c = 10.3064 (17) Å
β = 91.100 (2)°
V = 1347.3 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 K
0.32 × 0.30 × 0.29 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.964, T_{max} = 0.967
7079 measured reflections
2952 independent reflections
1353 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.137
S = 1.06
2952 reflections
204 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O2^{i}	0.82	1.93	2.736 (3)	169
O1—H1A···N2^{i}	0.82	2.61	3.121 (3)	122
O3—H3···O4	0.82	1.90	2.592 (4)	142
N1—H1···O5^{ii}	0.90 (1)	2.32 (1)	3.203 (4)	167 (3)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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 author thanks the Experimental Center of Linyi University for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2243).

References

Ahmad, T., Zia-ur-Rehman, M., Siddiqui, H. L., Mahmud, S. & Parvez, M. (2010). *Acta Cryst.* E**66**, o976.
Ajani, O. O., Obafemi, C. A., Nwinyi, O. C. & Akinpelu, D. A. (2010). *Bioorg. Med. Chem.* **18**, 214–221.
Avaji, P. G., Kumar, C. H. V., Patil, S. A., Shivananda, K. N. & Nagaraju, C. (2009). *Eur. J. Med. Chem.* **44**, 3552–3559.
Ban, H.-Y. (2010). *Acta Cryst.* E**66**, o3240.
Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fan, C. D., Su, H., Zhao, J., Zhao, B. X., Zhang, S. L. & Miao, J. Y. (2010). *Eur. J. Med. Chem.* **45**, 1438–1446.
Ji, X.-H. & Lu, J.-F. (2010). *Acta Cryst.* E**66**, o1514.
Rasras, A. J. M., Al-Tel, T. H., Amal, A. F. & Al-Qawasmeh, R. A. (2010). *Eur. J. Med. Chem.* **45**, 2307–2313.
Shalash, M., Salhin, A., Adnan, R., Yeap, C. S. & Fun, H.-K. (2010). *Acta Cryst.* E**66**, o3126–o3127.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2011). E**67**, o300 [doi:10.1107/S1600536811000195]

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

Zhen Zhang

S1. Comment

Hydrazone compounds have received much attention due to their potential applications in biological chemistry (Ajani *et al.*, 2010; Avaji *et al.*, 2009; Fan *et al.*, 2010; Rasras *et al.*, 2010). As a continuatio of our work on the hydrazone compounds, the new title hydrazone compound was prepared and structurally characterized.

The molecule of the title compound assumes an *E* configuration with respect to the methylidene unit (Fig. 1). The dihedral angle between the best mean planes of the two benzene rings is 5.46 (15)°. An intramolecular O—H···O hydrogen bond is present in the molecule (Table 1). The bond lengths are comparable to those observed in similar hydrazone compounds (Ahmad *et al.*, 2010; Ban, 2010; Ji & Lu, 2010; Shalash *et al.*, 2010).

The crystal structure is stabilized by intermolecular O—H···O, O—H···N, and N—H···O hydrogen bonds (Table 1 and Fig. 2).

S2. Experimental

An ethanol solution (50 ml) of 4-hydroxybenzohydrazide (0.01 mol) and 4-hydroxy-3-nitrobenzaldehyde (0.01 mol) was stirred at room temperature for 30 min to give a yellow solution. Yellow block-shaped single crystals, suitable for X-ray diffraction, were formed by slow evaporation of the solution in air.

S3. Refinement

The amino H-atom, H1, was located from a difference Fourier map and refined with a N—H distance restraint to 0.90 (1) Å. The remaining H atoms were positioned geometrically and refined using the riding-model approximation: C—H = 0.93 Å, and O—H = 0.82 Å, with U_{iso}(H) = 1.2U_{eq}(parent C-atom) and 1.5U_{eq}(parent O-atom).

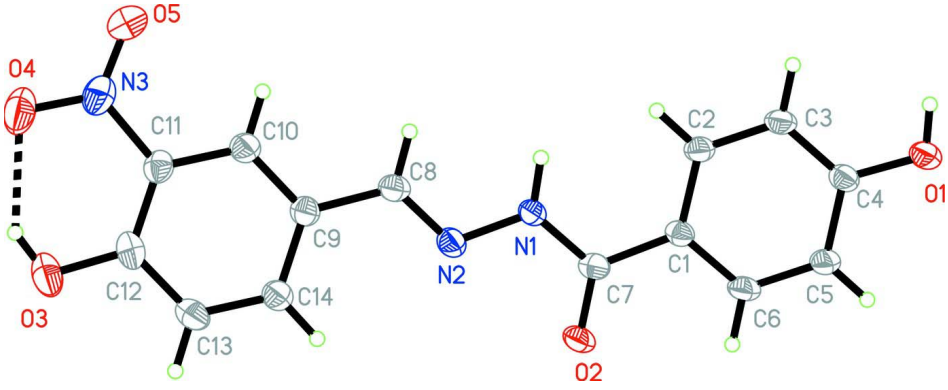


Figure 1

A view of the molecular structure of the title compound, showing the displacement ellipsoids at the 30% probability level. The intramolecular O—H···O hydrogen bond is shown as a dashed line.

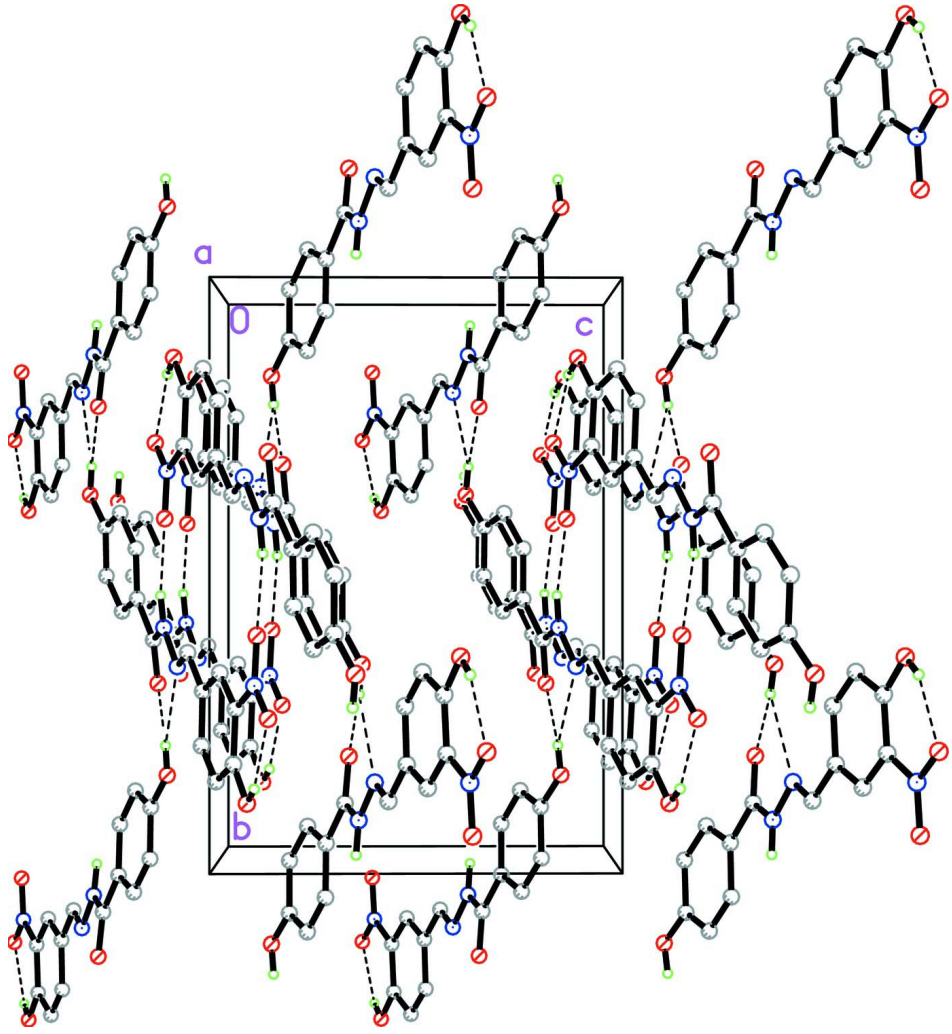


Figure 2

A perspective view along the a-axis of the crystal packing of the title compound. The hydrogen bonds are shown as dashed lines. H-atoms not involved in hydrogen bonding have been omitted for clarity.

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

Crystal data

$C_{14}H_{11}N_3O_5$
$M_r = 301.26$
Monoclinic, $P2_1/n$
$a = 8.786$ (3) Å
$b = 14.882$ (2) Å
$c = 10.3064$ (17) Å
$\beta = 91.100$ (2)°
$V = 1347.3$ (5) Å^3
$Z = 4$

$F(000) = 624$
$D_x = 1.485$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1674 reflections
$\theta = 2.4$–26.9°
$\mu = 0.12$ mm^{-1}
$T = 298$ K
Block, yellow
0.32 × 0.30 × 0.29 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.964, T_{max} = 0.967
7079 measured reflections
2952 independent reflections
1353 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 27.5°, θ_{min} = 2.4°
h = −9→11
k = −19→19
l = −13→7

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.137
S = 1.06
2952 reflections
204 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
$w = 1/[\sigma^2(F_o^2) + (0.0262P)^2 + 0.9404P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
N1	0.4602 (3)	0.40859 (16)	0.1386 (3)	0.0442 (7)
N2	0.3606 (3)	0.34194 (16)	0.0989 (3)	0.0419 (7)
O1	1.0054 (2)	0.64891 (13)	0.3680 (3)	0.0577 (7)
H1A	0.9704	0.6998	0.3614	0.087*
O2	0.6482 (2)	0.30744 (13)	0.1654 (2)	0.0521 (7)
O3	−0.2256 (3)	0.12672 (15)	−0.0998 (3)	0.0675 (8)
H3	−0.3047	0.1529	−0.1200	0.101*
O4	−0.3830 (3)	0.26935 (19)	−0.1553 (3)	0.0766 (9)
O5	−0.2875 (3)	0.40196 (19)	−0.1256 (3)	0.0801 (9)
C1	0.7049 (3)	0.45812 (18)	0.2226 (3)	0.0344 (7)
C2	0.6644 (3)	0.54841 (18)	0.2232 (3)	0.0411 (8)
H2	0.5691	0.5655	0.1909	0.049*
C3	0.7623 (3)	0.61274 (19)	0.2707 (3)	0.0432 (8)
H3A	0.7332	0.6728	0.2699	0.052*
C4	0.9033 (3)	0.58892 (19)	0.3194 (3)	0.0398 (8)

C5	0.9456 (4)	0.49917 (19)	0.3202 (3)	0.0503 (10)
H5	1.0406	0.4824	0.3534	0.060*
C6	0.8471 (3)	0.4349 (2)	0.2720 (3)	0.0503 (10)
H6	0.8766	0.3749	0.2726	0.060*
C7	0.6036 (3)	0.38575 (19)	0.1743 (3)	0.0382 (8)
C8	0.2280 (4)	0.3678 (2)	0.0654 (3)	0.0444 (9)
H8	0.2047	0.4287	0.0685	0.053*
C9	0.1110 (3)	0.3044 (2)	0.0222 (3)	0.0398 (8)
C10	−0.0244 (3)	0.3372 (2)	−0.0261 (3)	0.0428 (8)
H10	−0.0398	0.3989	−0.0301	0.051*
C11	−0.1385 (3)	0.2798 (2)	−0.0692 (3)	0.0410 (8)
C12	−0.1193 (4)	0.1874 (2)	−0.0631 (3)	0.0455 (9)
C13	0.0193 (4)	0.1544 (2)	−0.0147 (3)	0.0486 (9)
H13	0.0347	0.0926	−0.0102	0.058*
C14	0.1324 (4)	0.2110 (2)	0.0262 (3)	0.0446 (9)
H14	0.2243	0.1875	0.0569	0.053*
N3	−0.2779 (3)	0.3202 (2)	−0.1195 (3)	0.0561 (8)
H1	0.424 (3)	0.4649 (10)	0.144 (3)	0.067*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0310 (16)	0.0268 (13)	0.075 (2)	0.0000 (12)	−0.0032 (14)	−0.0041 (14)
N2	0.0364 (17)	0.0336 (14)	0.0558 (19)	−0.0036 (12)	0.0034 (14)	−0.0015 (13)
O1	0.0463 (15)	0.0301 (12)	0.0962 (19)	−0.0006 (11)	−0.0149 (14)	−0.0028 (13)
O2	0.0450 (14)	0.0267 (11)	0.0845 (18)	0.0024 (10)	−0.0015 (12)	−0.0041 (12)
O3	0.0593 (17)	0.0626 (16)	0.081 (2)	−0.0226 (13)	−0.0018 (15)	−0.0094 (15)
O4	0.0474 (17)	0.094 (2)	0.088 (2)	−0.0095 (16)	−0.0138 (15)	−0.0119 (17)
O5	0.0598 (18)	0.0628 (18)	0.117 (2)	0.0151 (15)	−0.0165 (16)	0.0017 (17)
C1	0.0307 (18)	0.0281 (15)	0.045 (2)	−0.0001 (13)	0.0075 (15)	0.0008 (14)
C2	0.033 (2)	0.0310 (17)	0.060 (2)	0.0037 (14)	0.0008 (17)	0.0031 (15)
C3	0.040 (2)	0.0259 (16)	0.064 (2)	0.0021 (14)	0.0000 (17)	0.0058 (16)
C4	0.0373 (19)	0.0296 (16)	0.052 (2)	−0.0024 (14)	0.0029 (17)	−0.0002 (15)
C5	0.036 (2)	0.0318 (17)	0.082 (3)	0.0063 (16)	−0.0109 (19)	−0.0018 (18)
C6	0.043 (2)	0.0273 (17)	0.080 (3)	0.0083 (15)	−0.004 (2)	−0.0029 (17)
C7	0.037 (2)	0.0315 (17)	0.046 (2)	0.0021 (14)	0.0081 (16)	0.0033 (15)
C8	0.041 (2)	0.0315 (17)	0.061 (2)	0.0032 (15)	0.0027 (18)	0.0009 (16)
C9	0.0335 (19)	0.0374 (17)	0.049 (2)	−0.0010 (14)	0.0075 (16)	0.0014 (16)
C10	0.037 (2)	0.0374 (18)	0.054 (2)	0.0014 (15)	0.0057 (17)	−0.0008 (16)
C11	0.034 (2)	0.048 (2)	0.042 (2)	0.0007 (15)	0.0065 (16)	−0.0042 (16)
C12	0.044 (2)	0.050 (2)	0.042 (2)	−0.0134 (17)	0.0061 (17)	−0.0068 (17)
C13	0.055 (2)	0.0335 (17)	0.057 (2)	−0.0027 (17)	0.0083 (19)	−0.0020 (17)
C14	0.041 (2)	0.0375 (19)	0.056 (2)	−0.0010 (15)	0.0033 (17)	0.0012 (16)
N3	0.0403 (19)	0.071 (2)	0.057 (2)	0.0008 (17)	0.0035 (16)	−0.0043 (17)

Geometric parameters (Å, º)

N1—C7	1.349 (4)	C3—H3A	0.9300
N1—N2	1.380 (3)	C4—C5	1.387 (4)
N1—H1	0.899 (10)	C5—C6	1.376 (4)
N2—C8	1.269 (4)	C5—H5	0.9300
O1—C4	1.355 (3)	C6—H6	0.9300
O1—H1A	0.8200	C8—C9	1.458 (4)
O2—C7	1.234 (3)	C8—H8	0.9300
O3—C12	1.347 (4)	C9—C10	1.371 (4)
O3—H3	0.8200	C9—C14	1.403 (4)
O4—N3	1.244 (3)	C10—C11	1.383 (4)
O5—N3	1.222 (4)	C10—H10	0.9300
C1—C6	1.383 (4)	C11—C12	1.387 (4)
C1—C2	1.390 (4)	C11—N3	1.451 (4)
C1—C7	1.478 (4)	C12—C13	1.396 (4)
C2—C3	1.371 (4)	C13—C14	1.363 (4)
C2—H2	0.9300	C13—H13	0.9300
C3—C4	1.374 (4)	C14—H14	0.9300
C7—N1—N2	118.9 (2)	N1—C7—C1	117.5 (3)
C7—N1—H1	123 (2)	N2—C8—C9	121.7 (3)
N2—N1—H1	118 (2)	N2—C8—H8	119.2
C8—N2—N1	115.9 (2)	C9—C8—H8	119.2
C4—O1—H1A	109.5	C10—C9—C14	118.5 (3)
C12—O3—H3	109.5	C10—C9—C8	118.9 (3)
C6—C1—C2	118.0 (3)	C14—C9—C8	122.6 (3)
C6—C1—C7	118.4 (3)	C9—C10—C11	121.0 (3)
C2—C1—C7	123.6 (3)	C9—C10—H10	119.5
C3—C2—C1	121.1 (3)	C11—C10—H10	119.5
C3—C2—H2	119.4	C10—C11—C12	120.8 (3)
C1—C2—H2	119.4	C10—C11—N3	117.4 (3)
C2—C3—C4	120.4 (3)	C12—C11—N3	121.9 (3)
C2—C3—H3A	119.8	O3—C12—C11	124.7 (3)
C4—C3—H3A	119.8	O3—C12—C13	117.3 (3)
O1—C4—C3	123.4 (3)	C11—C12—C13	118.0 (3)
O1—C4—C5	117.2 (3)	C14—C13—C12	121.2 (3)
C3—C4—C5	119.4 (3)	C14—C13—H13	119.4
C6—C5—C4	120.0 (3)	C12—C13—H13	119.4
C6—C5—H5	120.0	C13—C14—C9	120.5 (3)
C4—C5—H5	120.0	C13—C14—H14	119.8
C5—C6—C1	121.1 (3)	C9—C14—H14	119.8
C5—C6—H6	119.4	O5—N3—O4	122.7 (3)
C1—C6—H6	119.4	O5—N3—C11	119.2 (3)
O2—C7—N1	120.9 (3)	O4—N3—C11	118.1 (3)
O2—C7—C1	121.6 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O2^{i}	0.82	1.93	2.736 (3)	169
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O3—H3···O4	0.82	1.90	2.592 (4)	142
N1—H1···O5^{ii}	0.90 (1)	2.32 (1)	3.203 (4)	167 (3)

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