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N′-[1-(2-Hydroxyphenyl)ethylidene]-2-nitrobenzohydrazide methanol solvate

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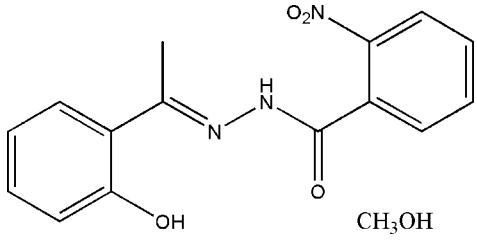
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.124; data-to-parameter ratio = 14.9.

In the title compound, $C_{15}H_{13}N_3O_4\cdot CH_3OH$, the dihedral angle between the two substituted benzene rings is 66.7 (2)°. An intramolecular O—H···N hydrogen bond is observed in the Schiff base molecule. In the crystal structure, the Schiff base and solvent molecules are linked into chains running along the *a* axis by intermolecular O—H···O and N—H···O hydrogen bonds.

Related literature

For the biological properties of hydrazone compounds, see: Bedia *et al.* (2006). For complexes of hydrazone compounds, see: Iskander *et al.* (2001); Aggarwal *et al.* (1981); Aruffo *et al.* (1982). For related structures, see: Fun *et al.* (2008*a*,*b*); Butcher *et al.* (2007); Zhi & Yang (2007); Mohd Lair *et al.* (2009*a*,*b*); Yehye *et al.* (2008). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{15}H_{13}N_3O_4\cdot CH_4O$
M_r = 331.33
Triclinic, $P\overline{1}$
a = 7.124 (2) Å
b = 8.066 (2) Å
c = 15.764 (3) Å
α = 101.950 (2)°
β = 92.972 (2)°
γ = 114.889 (2)°
V = 794.0 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 K
0.23 × 0.23 × 0.22 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.976, T_{max} = 0.977
4659 measured reflections
3371 independent reflections
2660 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.124
S = 1.05
3371 reflections
226 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 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
N1—H1···O5^{i}	0.893 (9)	2.08 (1)	2.9563 (17)	165 (2)
O5—H5···O1	0.82	1.94	2.7451 (16)	168
O4—H4···N2	0.82	1.85	2.5612 (17)	144

Symmetry code: (i) $x-1, y, z$.

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: CI2770).

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supplementary materials

Acta Cryst. (2009). E**65**, o585 [**doi:10.1107/S160053680900508X**]

N'-[1-(2-Hydroxyphenyl)ethylidene]-2-nitrobenzohydrazide methanol solvate

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Comment

Hydrazone compounds have been demonstrated to possess biological properties, such as antimicrobial, antitubercular, anticancer and antitumor (Bedia *et al.*, 2006). Moreover, these compounds are good ligands in the coordination chemistry (Iskander *et al.*, 2001; Aggarwal *et al.*, 1981; Aruffo *et al.*, 1982). Recently, a large number of hydrazone compounds have been reported (Fun *et al.*, 2008*b*; Butcher *et al.*, 2007; Zhi & Yang, 2007). In this paper, a new hydrazone compound (Fig. 1), derived from 1-(2-hydroxyphenyl)ethanone and 2-nitrobenzohydrazide is reported.

The asymmetric unit of the title compound contains a Schiff base molecule and a methanol molecule of crystallization. The dihedral angle between the two substituted benzene rings is 66.7 (2)°, indicating that the Schiff base molecule is twisted. The dihedral angle between the C1-C6 and O2/O3/N3/C2 planes is 26.0 (1)°. All bond lengths in the compound are typical (Allen *et al.*, 1987) and comparable to those observed in similar hydrazone compounds (Fun *et al.*, 2008*a*; Mohd Lair *et al.*, 2009*a*,b; Yehye *et al.*, 2008). An intramolecular O—H···N hydrogen bond is observed in the Schiff base molecule.

In the crystal structure, the Schiff base and methanol molecules are linked through O–H···O and N–H···O hydrogen bonds (Table 1), forming chains running along the *a* axis (Fig. 2).

Experimental

1-(2-Hydroxyphenyl)ethanone (1.0 mmol, 136.2 mg) and 2-nitrobenzohydrazide (1.0 mmol, 197.2 mg) were stirred at room temperature for 3 h. The filtrate was kept in air for a few days to obtain colourless block-shaped crystals of the title compound.

Refinement

Atom H1 attached to N1 was located in a difference Fourier map and refined isotropically, with the N–H distance restrained to 0.90 (1) Å. C- and O-bound H atoms were positioned geometrically and refined using a riding model with d(C–H) = 0.93–0.96 Å, d(O–H) = 0.82 Å and U_{iso} = 1.2U_{eq}(C) and 1.5U_{eq}(O and C_{methyl}).

Figures

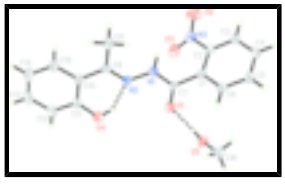

Fig. 1. The molecular structure of the title compound, with 30% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines.

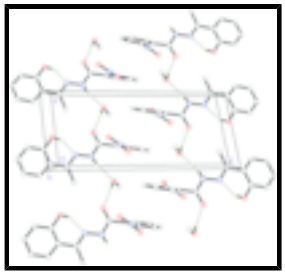

Fig. 2. Hydrogen-bonded (dashed lines) chains in the title compound, viewed along the *b* axis. H atoms not involved in the interactions have been omitted for clarity.

N'-[1-(2-Hydroxyphenyl)ethylidene]-2-nitrobenzohydrazide methanol solvate

Crystal data

$C_{15}H_{13}N_3O_4 \cdot CH_4O$	$Z = 2$
$M_r = 331.33$	$F_{000} = 348$
Triclinic, $P\overline{1}$	$D_x = 1.386$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.124$ (2) Å	Cell parameters from 1968 reflections
$b = 8.066$ (2) Å	$\theta = 2.6$–28.5º
$c = 15.764$ (3) Å	$\mu = 0.11$ mm^{-1}
$\alpha = 101.950$ (2)º	$T = 298$ K
$\beta = 92.972$ (2)º	Block, colourless
$\gamma = 114.889$ (2)º	0.23 × 0.23 × 0.22 mm
$V = 794.0$ (3) Å^3	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	3371 independent reflections
Radiation source: fine-focus sealed tube	2660 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.014$
$T = 298$ K	$\theta_{max} = 27.0$º
ω scans	$\theta_{min} = 2.7$º
Absorption correction: multi-scan (SADABS; Bruker, 2001)	$h = -8 \rightarrow 9$
$T_{min} = 0.976$, $T_{max} = 0.977$	$k = -10 \rightarrow 10$
4659 measured reflections	$l = -19 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.043$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.124$	$w = 1/[\sigma^2(F_o^2) + (0.0585P)^2 + 0.1607P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.05	$(\Delta/\sigma)_{max}$ = 0.001
3371 reflections	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
226 parameters	$\Delta\rho_{min}$ = −0.23 e Å^{-3}
1 restraint	Extinction correction: SHELXTL (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.066 (6)

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}
O1	0.46363 (18)	0.2874 (2)	0.22886 (7)	0.0558 (3)
O2	0.2215 (3)	0.7197 (2)	0.41556 (13)	0.0902 (5)
O3	0.4022 (2)	0.65556 (18)	0.32038 (9)	0.0639 (4)
O4	0.3900 (2)	0.2749 (2)	−0.00437 (8)	0.0597 (4)
H4	0.3625	0.2787	0.0457	0.089*
O5	0.81043 (17)	0.27761 (18)	0.30757 (8)	0.0522 (3)
H5	0.7158	0.2968	0.2867	0.078*
N1	0.13992 (19)	0.27191 (19)	0.20210 (8)	0.0396 (3)
N2	0.1602 (2)	0.27247 (19)	0.11548 (8)	0.0403 (3)
N3	0.2967 (2)	0.61957 (19)	0.37848 (10)	0.0495 (4)
C1	0.2777 (2)	0.2991 (2)	0.34961 (9)	0.0336 (3)
C2	0.2633 (2)	0.4482 (2)	0.40702 (9)	0.0363 (3)
C3	0.2296 (2)	0.4472 (2)	0.49259 (10)	0.0441 (4)
H3	0.2163	0.5472	0.5288	0.053*
C4	0.2163 (2)	0.2946 (2)	0.52303 (10)	0.0472 (4)
H4A	0.1947	0.2917	0.5805	0.057*
C5	0.2348 (2)	0.1460 (2)	0.46837 (11)	0.0450 (4)
H5A	0.2286	0.0448	0.4896	0.054*
C6	0.2625 (2)	0.1475 (2)	0.38232 (10)	0.0386 (3)
H6	0.2712	0.0453	0.3458	0.046*
C7	0.3056 (2)	0.2892 (2)	0.25478 (9)	0.0375 (3)
C8	0.0013 (2)	0.2459 (2)	0.06163 (9)	0.0383 (3)
C9	−0.2106 (3)	0.2077 (3)	0.08602 (11)	0.0564 (5)
H9A	−0.2363	0.1373	0.1298	0.085*
H9B	−0.3155	0.1361	0.0350	0.085*

H9C	−0.2160	0.3253	0.1089	0.085*
C10	0.0411 (3)	0.2564 (2)	−0.02863 (9)	0.0412 (4)
C11	0.2322 (3)	0.2729 (2)	−0.05640 (10)	0.0468 (4)
C12	0.2639 (4)	0.2882 (3)	−0.14152 (12)	0.0639 (5)
H12	0.3905	0.3006	−0.1595	0.077*
C13	0.1107 (4)	0.2853 (3)	−0.19919 (12)	0.0712 (6)
H13	0.1341	0.2953	−0.2558	0.085*
C14	−0.0770 (4)	0.2676 (3)	−0.17371 (12)	0.0678 (6)
H14	−0.1808	0.2648	−0.2131	0.081*
C15	−0.1110 (3)	0.2540 (3)	−0.08957 (11)	0.0543 (4)
H15	−0.2382	0.2429	−0.0728	0.065*
C16	0.7246 (3)	0.1002 (3)	0.32738 (15)	0.0637 (5)
H16A	0.8322	0.0598	0.3340	0.096*
H16B	0.6154	0.0097	0.2806	0.096*
H16C	0.6675	0.1103	0.3810	0.096*
H1	0.030 (2)	0.277 (3)	0.2252 (11)	0.052 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0432 (7)	0.0981 (10)	0.0467 (6)	0.0446 (7)	0.0145 (5)	0.0297 (6)
O2	0.1112 (14)	0.0669 (9)	0.1256 (14)	0.0645 (10)	0.0392 (11)	0.0317 (9)
O3	0.0749 (9)	0.0548 (7)	0.0645 (8)	0.0235 (7)	0.0108 (7)	0.0313 (6)
O4	0.0544 (8)	0.0912 (9)	0.0469 (7)	0.0439 (7)	0.0126 (6)	0.0183 (7)
O5	0.0372 (6)	0.0664 (7)	0.0588 (7)	0.0265 (6)	0.0036 (5)	0.0209 (6)
N1	0.0333 (7)	0.0574 (8)	0.0337 (6)	0.0243 (6)	0.0058 (5)	0.0135 (6)
N2	0.0391 (7)	0.0543 (7)	0.0329 (6)	0.0252 (6)	0.0042 (5)	0.0123 (5)
N3	0.0477 (8)	0.0415 (7)	0.0602 (9)	0.0216 (6)	−0.0014 (7)	0.0133 (6)
C1	0.0243 (6)	0.0407 (7)	0.0365 (7)	0.0144 (6)	0.0020 (5)	0.0115 (6)
C2	0.0294 (7)	0.0382 (7)	0.0420 (8)	0.0157 (6)	0.0004 (6)	0.0111 (6)
C3	0.0368 (8)	0.0516 (9)	0.0412 (8)	0.0209 (7)	0.0040 (6)	0.0038 (7)
C4	0.0399 (8)	0.0664 (10)	0.0351 (8)	0.0206 (8)	0.0087 (6)	0.0184 (7)
C5	0.0402 (8)	0.0495 (9)	0.0501 (9)	0.0180 (7)	0.0088 (7)	0.0258 (7)
C6	0.0345 (8)	0.0390 (7)	0.0443 (8)	0.0174 (6)	0.0056 (6)	0.0120 (6)
C7	0.0315 (7)	0.0471 (8)	0.0391 (8)	0.0204 (6)	0.0062 (6)	0.0146 (6)
C8	0.0388 (8)	0.0385 (7)	0.0379 (8)	0.0202 (6)	−0.0001 (6)	0.0050 (6)
C9	0.0400 (9)	0.0804 (12)	0.0470 (9)	0.0277 (9)	−0.0005 (7)	0.0123 (9)
C10	0.0492 (9)	0.0390 (7)	0.0340 (7)	0.0215 (7)	−0.0019 (6)	0.0039 (6)
C11	0.0557 (10)	0.0477 (9)	0.0377 (8)	0.0264 (8)	0.0039 (7)	0.0053 (7)
C12	0.0784 (14)	0.0734 (13)	0.0419 (9)	0.0364 (11)	0.0161 (9)	0.0110 (9)
C13	0.1033 (18)	0.0730 (13)	0.0326 (9)	0.0357 (12)	0.0059 (10)	0.0119 (9)
C14	0.0875 (15)	0.0701 (12)	0.0408 (10)	0.0355 (11)	−0.0158 (9)	0.0084 (9)
C15	0.0589 (11)	0.0597 (10)	0.0424 (9)	0.0286 (9)	−0.0080 (7)	0.0077 (8)
C16	0.0495 (11)	0.0652 (12)	0.0875 (14)	0.0311 (9)	0.0180 (10)	0.0274 (10)

Geometric parameters (Å, °)

O1—C7	1.2218 (18)	C5—H5A	0.93
O2—N3	1.2172 (19)	C6—H6	0.93

O3—N3	1.2192 (19)	C8—C10	1.478 (2)
O4—C11	1.349 (2)	C8—C9	1.497 (2)
O4—H4	0.82	C9—H9A	0.96
O5—C16	1.410 (2)	C9—H9B	0.96
O5—H5	0.82	C9—H9C	0.96
N1—C7	1.3472 (18)	C10—C15	1.401 (2)
N1—N2	1.3812 (17)	C10—C11	1.412 (2)
N1—H1	0.893 (9)	C11—C12	1.394 (2)
N2—C8	1.2916 (19)	C12—C13	1.373 (3)
N3—C2	1.4705 (19)	C12—H12	0.93
C1—C6	1.389 (2)	C13—C14	1.374 (3)
C1—C2	1.392 (2)	C13—H13	0.93
C1—C7	1.5083 (19)	C14—C15	1.379 (3)
C2—C3	1.383 (2)	C14—H14	0.93
C3—C4	1.380 (2)	C15—H15	0.93
C3—H3	0.93	C16—H16A	0.96
C4—C5	1.384 (2)	C16—H16B	0.96
C4—H4A	0.93	C16—H16C	0.96
C5—C6	1.383 (2)		
C11—O4—H4	109.5	C10—C8—C9	120.59 (13)
C16—O5—H5	109.5	C8—C9—H9A	109.5
C7—N1—N2	117.46 (12)	C8—C9—H9B	109.5
C7—N1—H1	119.7 (12)	H9A—C9—H9B	109.5
N2—N1—H1	122.2 (12)	C8—C9—H9C	109.5
C8—N2—N1	119.43 (12)	H9A—C9—H9C	109.5
O2—N3—O3	123.74 (15)	H9B—C9—H9C	109.5
O2—N3—C2	118.16 (15)	C15—C10—C11	117.60 (15)
O3—N3—C2	118.08 (14)	C15—C10—C8	120.27 (15)
C6—C1—C2	117.14 (13)	C11—C10—C8	122.11 (14)
C6—C1—C7	117.87 (13)	O4—C11—C12	116.92 (16)
C2—C1—C7	124.98 (13)	O4—C11—C10	123.35 (14)
C3—C2—C1	122.71 (14)	C12—C11—C10	119.73 (16)
C3—C2—N3	117.65 (14)	C13—C12—C11	120.8 (2)
C1—C2—N3	119.55 (13)	C13—C12—H12	119.6
C4—C3—C2	118.57 (15)	C11—C12—H12	119.6
C4—C3—H3	120.7	C12—C13—C14	120.37 (18)
C2—C3—H3	120.7	C12—C13—H13	119.8
C3—C4—C5	120.24 (14)	C14—C13—H13	119.8
C3—C4—H4A	119.9	C13—C14—C15	119.77 (18)
C5—C4—H4A	119.9	C13—C14—H14	120.1
C6—C5—C4	120.21 (14)	C15—C14—H14	120.1
C6—C5—H5A	119.9	C14—C15—C10	121.72 (19)
C4—C5—H5A	119.9	C14—C15—H15	119.1
C5—C6—C1	121.08 (14)	C10—C15—H15	119.1
C5—C6—H6	119.5	O5—C16—H16A	109.5
C1—C6—H6	119.5	O5—C16—H16B	109.5
O1—C7—N1	123.95 (14)	H16A—C16—H16B	109.5
O1—C7—C1	121.23 (13)	O5—C16—H16C	109.5
N1—C7—C1	114.72 (12)	H16A—C16—H16C	109.5

N2—C8—C10	115.28 (13)	H16B—C16—H16C	109.5
N2—C8—C9	124.13 (14)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O5^{i}	0.893 (9)	2.08 (1)	2.9563 (17)	165 (2)
O5—H5···O1	0.82	1.94	2.7451 (16)	168
O4—H4···N2	0.82	1.85	2.5612 (17)	144

Symmetry codes: (i) *x*−1, *y*, *z*.

Fig. 1

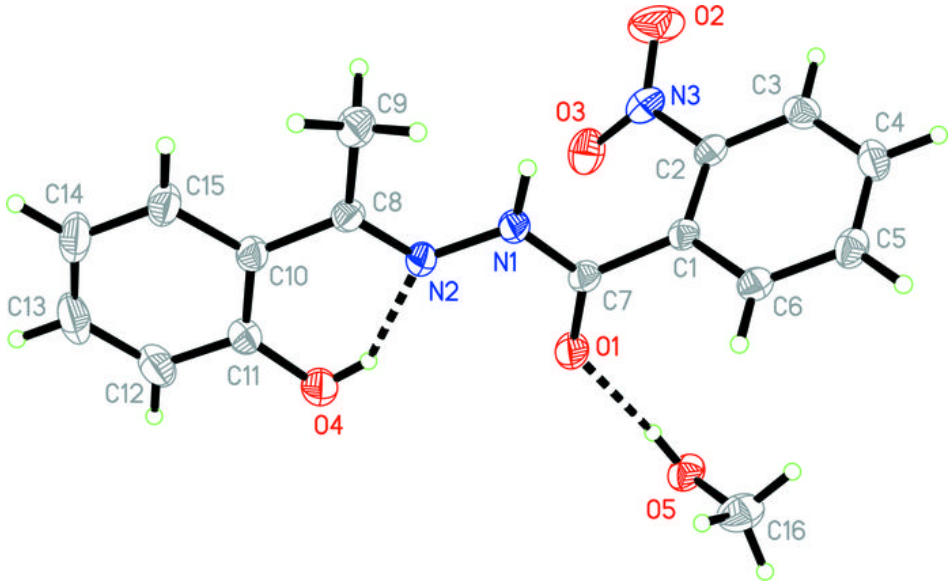
O2
N3
O3
C3
C4
C9
C2
C14
C15
C8
C5
N1
C1
C10
N2
C7
C13
C6
O1
C11
C12
O4
O5
C16

Fig. 2