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(*E*)-4-Methoxy-*N*′-(4-nitrobenzylidene)-benzohydrazide methanol monosolvate

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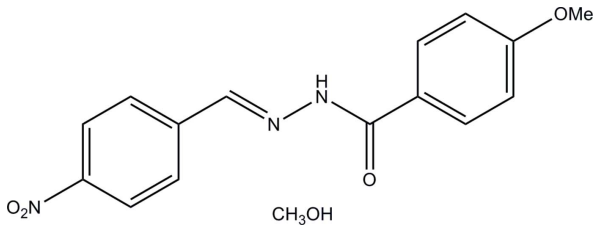
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.081; wR factor = 0.204; data-to-parameter ratio = 15.6.

The hydrazone molecule of the title compound, $C_{15}H_{13}N_3O_4\cdot CH_4O$, is nearly planar, with a dihedral angle between the two benzene rings of 1.2 (4)°. The molecule exists in a *trans* configuration with respect to the central methylidene unit. In the crystal, the benzohydrazide and methanol molecules are linked through intermolecular O—H···O, O—H···N and N—H···O hydrogen bonds, forming chains along the *a* axis.

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, Patil, Jebas *et al.*, 2008; Fun, Patil, Rao *et al.*, 2008; Yang *et al.* (2008); Ejsmont *et al.* (2008).



Experimental

Crystal data

$C_{15}H_{13}N_3O_4\cdot CH_4O$
M_r = 331.33
Monoclinic, $P2_1/n$
a = 6.6482 (14) Å
b = 17.730 (3) Å
c = 13.898 (2) Å
β = 95.004 (3)°
V = 1631.9 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 K
0.20 × 0.17 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008)
T_{min} = 0.980, T_{max} = 0.983
12876 measured reflections
3466 independent reflections
1184 reflections with $I > 2\sigma(I)$
R_{int} = 0.115

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.081
$wR(F^2)$ = 0.204
S = 0.94
3466 reflections
222 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O3	0.82	2.03	2.812 (4)	159
O5—H5···N2	0.82	2.61	3.194 (4)	129
N3—H3*A*···O5^{i}	0.90 (1)	2.02 (2)	2.900 (4)	166 (4)

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

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 data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2522).

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supporting information

Acta Cryst. (2010). E**66**, o3240 [https://doi.org/10.1107/S160053681004701X]

(*E*)-4-Methoxy-*N*′-(4-nitrobenzylidene)benzohydrazide methanol monosolvate

Hong-Yan Ban

S1. Comment

Hydrazone compounds 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 large number of hydrazone compounds have been synthesized and structurally characterized (Yehye *et al.*, 2008; Fun, Patil, Jebas *et al.*, 2008; Fun, Patil, Rao *et al.*, 2008; Yang *et al.*, 2008; Ejsmont *et al.*, 2008). Recently, we have reported a few such compounds (Ban & Li, 2008*a*,*b*; Li & Ban, 2009*a*,*b*). Herein the crystal structure of the title new compound is reported.

The asymmetric unit of the title compound consists of a hydrazone molecule and a methanol molecule (Fig. 1). The hydrazone molecule is nearly planar, the dihedral angle between the two benzene rings being 1.2 (4)°. The molecule exists in a *trans* configuration with respect to the central methylidene unit. In the crystal structure, the hydrazone molecules and the methanol molecules are linked through intermolecular O—H···O, O—H···N and N—H···O hydrogen bonds (Table 1), forming chains along the *a* axis (Fig. 2).

S2. Experimental

The title compound was prepared by refluxing 4-nitrobenzaldehyde (1.0 mol) with 4-methoxybenzohydrazide (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 H3A 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 = 0.93–0.96 Å and O—H = 0.82 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(O and methyl C).

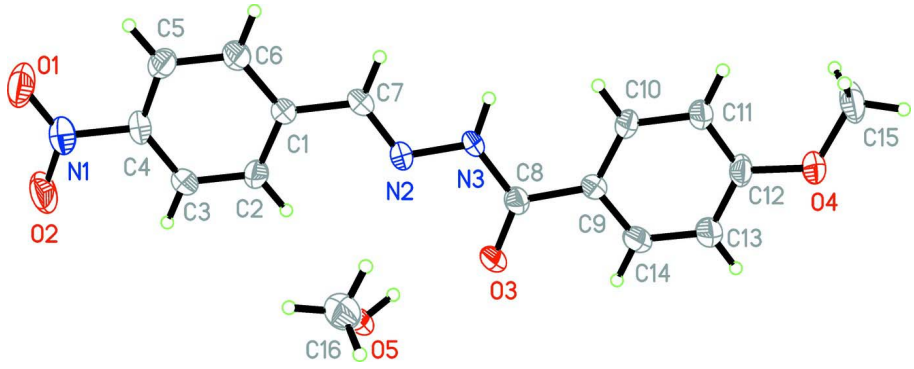

Figure 1

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

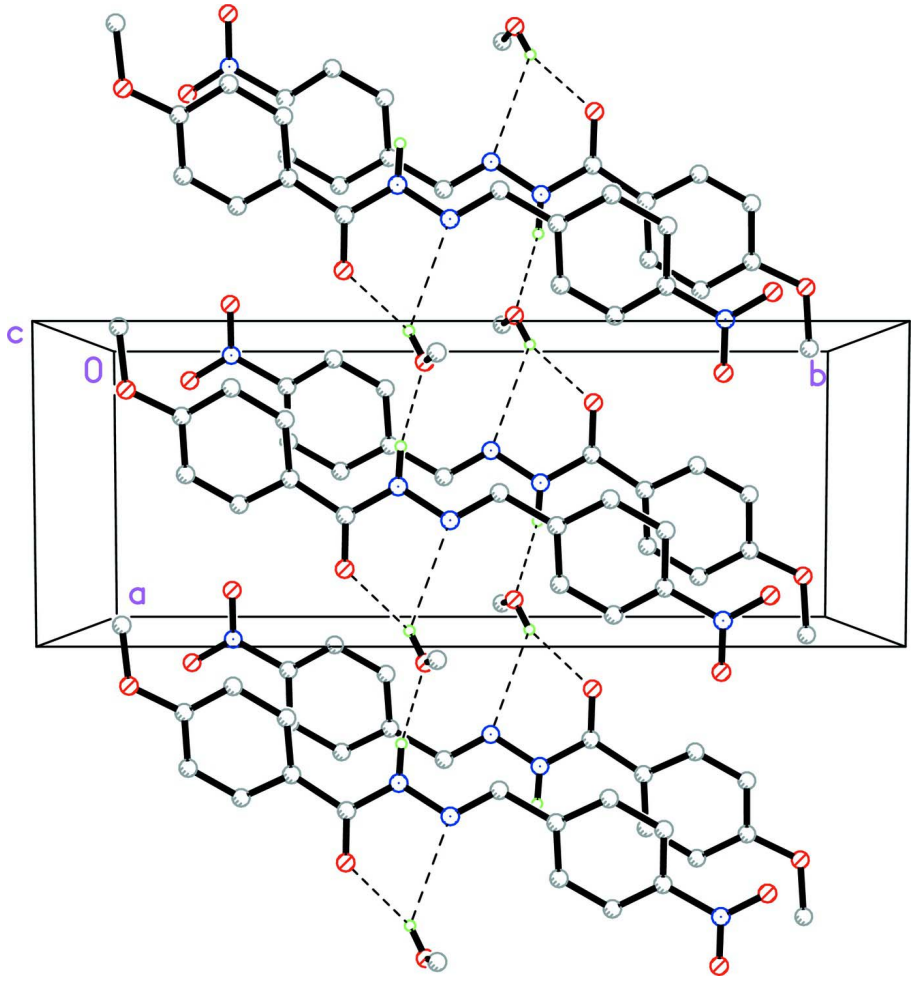

Figure 2

The packing diagram of the title compound, viewed along the *c* axis. Hydrogen bonds are shown as dashed lines. Hydrogen atoms not involved in hydrogen bonding are omitted for clarity.

(*E*)-4-Methoxy-*N*′-(4-nitrobenzylidene)benzohydrazide methanol monosolvate

Crystal data

$C_{15}H_{13}N_3O_4·CH_4O$
M_r = 331.33
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 6.6482 (14) Å
b = 17.730 (3) Å
c = 13.898 (2) Å
β = 95.004 (3)°
V = 1631.9 (5) Å^3
Z = 4
$F(000)$ = 696
D_x = 1.349 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 794 reflections
θ = 2.7–26.5°
μ = 0.10 mm^{-1}
T = 298 K
Block, colourless
0.20 × 0.17 × 0.17 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, 2008)
T_{min} = 0.980, T_{max} = 0.983
12876 measured reflections
3466 independent reflections
1184 reflections with $I > 2\sigma(I)$
R_{int} = 0.115
θ_{max} = 27.0°, θ_{min} = 1.9°
h = −8→8
k = −22→22
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.081
$wR(F^2)$ = 0.204
S = 0.94
3466 reflections
222 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
$w = 1/[\sigma^2(F_o^2) + (0.0757P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 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	0.4536 (7)	−0.3135 (2)	0.1424 (3)	0.0634 (12)
N2	0.1193 (5)	0.0260 (2)	0.1132 (2)	0.0453 (9)
N3	0.0063 (5)	0.0908 (2)	0.1112 (3)	0.0460 (9)

O1	0.3670 (6)	−0.3698 (2)	0.1665 (3)	0.0923 (13)
O2	0.6283 (6)	−0.3132 (2)	0.1217 (3)	0.0930 (13)
O3	0.2843 (4)	0.15868 (16)	0.0864 (2)	0.0650 (10)
O4	−0.3188 (4)	0.43167 (16)	0.0795 (2)	0.0709 (10)
O5	0.5933 (4)	0.05685 (17)	0.1482 (3)	0.0642 (10)
H5	0.4898	0.0767	0.1237	0.096*
C1	0.1378 (6)	−0.1068 (2)	0.1293 (3)	0.0424 (11)
C2	0.3344 (6)	−0.1112 (2)	0.1018 (3)	0.0529 (12)
H2	0.3964	−0.0682	0.0801	0.063*
C3	0.4373 (6)	−0.1786 (3)	0.1064 (3)	0.0549 (13)
H3	0.5680	−0.1817	0.0876	0.066*
C4	0.3437 (7)	−0.2407 (2)	0.1391 (3)	0.0493 (12)
C5	0.1521 (7)	−0.2403 (3)	0.1657 (3)	0.0586 (13)
H5A	0.0922	−0.2840	0.1866	0.070*
C6	0.0480 (6)	−0.1719 (3)	0.1607 (3)	0.0563 (13)
H6	−0.0834	−0.1699	0.1786	0.068*
C7	0.0276 (6)	−0.0354 (3)	0.1262 (3)	0.0495 (12)
H7	−0.1100	−0.0350	0.1338	0.059*
C8	0.1041 (7)	0.1569 (2)	0.0971 (3)	0.0461 (11)
C9	−0.0195 (6)	0.2263 (2)	0.0959 (3)	0.0449 (11)
C10	−0.2157 (6)	0.2311 (2)	0.1201 (3)	0.0508 (12)
H10	−0.2785	0.1877	0.1403	0.061*
C11	−0.3223 (6)	0.2981 (2)	0.1153 (3)	0.0535 (12)
H11	−0.4551	0.2998	0.1314	0.064*
C12	−0.2287 (7)	0.3619 (2)	0.0864 (3)	0.0540 (12)
C13	−0.0345 (7)	0.3593 (3)	0.0605 (4)	0.0829 (18)
H13	0.0277	0.4028	0.0402	0.100*
C14	0.0666 (7)	0.2920 (3)	0.0649 (4)	0.0745 (16)
H14	0.1977	0.2904	0.0465	0.089*
C15	−0.5263 (8)	0.4384 (3)	0.0967 (4)	0.0833 (17)
H15A	−0.6078	0.4115	0.0477	0.125*
H15B	−0.5642	0.4907	0.0951	0.125*
H15C	−0.5467	0.4177	0.1589	0.125*
C16	0.5721 (7)	0.0414 (3)	0.2467 (4)	0.0823 (17)
H16A	0.6181	−0.0089	0.2617	0.123*
H16B	0.4327	0.0459	0.2588	0.123*
H16C	0.6512	0.0767	0.2863	0.123*
H3A	−0.128 (2)	0.087 (2)	0.116 (3)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.077 (3)	0.054 (3)	0.060 (3)	0.021 (3)	0.006 (2)	0.000 (2)
N2	0.041 (2)	0.038 (2)	0.056 (2)	0.0098 (19)	0.0024 (17)	0.0028 (18)
N3	0.030 (2)	0.039 (2)	0.069 (3)	0.0102 (19)	0.0049 (19)	0.0039 (19)
O1	0.117 (3)	0.047 (2)	0.115 (3)	0.022 (2)	0.025 (2)	0.015 (2)
O2	0.078 (3)	0.077 (3)	0.128 (3)	0.043 (2)	0.031 (2)	0.021 (2)
O3	0.0322 (17)	0.053 (2)	0.111 (3)	0.0070 (15)	0.0143 (17)	0.0145 (18)

O4	0.056 (2)	0.0407 (19)	0.115 (3)	0.0141 (17)	−0.0002 (19)	−0.0034 (19)
O5	0.0343 (18)	0.056 (2)	0.102 (3)	0.0096 (16)	0.0027 (17)	0.017 (2)
C1	0.037 (3)	0.040 (3)	0.049 (3)	0.002 (2)	0.004 (2)	0.001 (2)
C2	0.050 (3)	0.036 (3)	0.073 (3)	0.004 (2)	0.013 (2)	0.000 (2)
C3	0.037 (3)	0.050 (3)	0.077 (4)	0.005 (2)	0.006 (2)	0.002 (3)
C4	0.058 (3)	0.042 (3)	0.048 (3)	0.017 (2)	0.003 (2)	0.003 (2)
C5	0.061 (3)	0.044 (3)	0.073 (3)	−0.002 (3)	0.017 (3)	0.008 (2)
C6	0.048 (3)	0.052 (3)	0.070 (4)	0.007 (3)	0.017 (2)	0.002 (3)
C7	0.034 (2)	0.051 (3)	0.063 (3)	0.007 (2)	0.004 (2)	0.002 (2)
C8	0.042 (3)	0.044 (3)	0.053 (3)	0.007 (2)	0.001 (2)	0.009 (2)
C9	0.035 (3)	0.044 (3)	0.056 (3)	0.001 (2)	−0.001 (2)	0.002 (2)
C10	0.052 (3)	0.031 (3)	0.071 (3)	0.003 (2)	0.010 (2)	0.007 (2)
C11	0.049 (3)	0.039 (3)	0.074 (3)	0.008 (2)	0.013 (2)	0.009 (2)
C12	0.059 (3)	0.034 (3)	0.067 (3)	0.013 (2)	−0.005 (3)	0.002 (2)
C13	0.051 (3)	0.047 (3)	0.152 (5)	0.005 (3)	0.020 (3)	0.015 (3)
C14	0.037 (3)	0.057 (3)	0.131 (5)	0.003 (3)	0.012 (3)	0.020 (3)
C15	0.091 (4)	0.057 (3)	0.106 (4)	0.036 (3)	0.029 (3)	0.009 (3)
C16	0.069 (4)	0.082 (4)	0.094 (5)	0.006 (3)	−0.009 (3)	−0.002 (3)

Geometric parameters (Å, º)

N1—O1	1.214 (4)	C5—H5A	0.9300
N1—O2	1.221 (5)	C6—H6	0.9300
N1—C4	1.482 (5)	C7—H7	0.9300
N2—C7	1.269 (5)	C8—C9	1.479 (5)
N2—N3	1.372 (4)	C9—C10	1.377 (5)
N3—C8	1.363 (5)	C9—C14	1.383 (5)
N3—H3A	0.902 (10)	C10—C11	1.383 (5)
O3—C8	1.221 (4)	C10—H10	0.9300
O4—C12	1.374 (5)	C11—C12	1.367 (5)
O4—C15	1.426 (5)	C11—H11	0.9300
O5—C16	1.415 (5)	C12—C13	1.371 (6)
O5—H5	0.8200	C13—C14	1.369 (6)
C1—C6	1.386 (5)	C13—H13	0.9300
C1—C2	1.395 (5)	C14—H14	0.9300
C1—C7	1.461 (5)	C15—H15A	0.9600
C2—C3	1.376 (5)	C15—H15B	0.9600
C2—H2	0.9300	C15—H15C	0.9600
C3—C4	1.362 (5)	C16—H16A	0.9600
C3—H3	0.9300	C16—H16B	0.9600
C4—C5	1.357 (5)	C16—H16C	0.9600
C5—C6	1.395 (5)		

O1—N1—O2	123.5 (4)	N3—C8—C9	116.5 (4)
O1—N1—C4	118.7 (4)	C10—C9—C14	116.8 (4)
O2—N1—C4	117.7 (5)	C10—C9—C8	125.8 (4)
C7—N2—N3	116.9 (3)	C14—C9—C8	117.4 (4)
C8—N3—N2	117.2 (3)	C9—C10—C11	122.2 (4)

C8—N3—H3A	124 (3)	C9—C10—H10	118.9
N2—N3—H3A	119 (3)	C11—C10—H10	118.9
C12—O4—C15	119.1 (4)	C12—C11—C10	118.7 (4)
C16—O5—H5	109.5	C12—C11—H11	120.7
C6—C1—C2	118.6 (4)	C10—C11—H11	120.6
C6—C1—C7	120.1 (4)	C11—C12—C13	120.9 (4)
C2—C1—C7	121.3 (4)	C11—C12—O4	123.9 (4)
C3—C2—C1	120.6 (4)	C13—C12—O4	115.2 (4)
C3—C2—H2	119.7	C14—C13—C12	119.2 (5)
C1—C2—H2	119.7	C14—C13—H13	120.4
C4—C3—C2	118.5 (4)	C12—C13—H13	120.4
C4—C3—H3	120.7	C13—C14—C9	122.2 (4)
C2—C3—H3	120.7	C13—C14—H14	118.9
C5—C4—C3	123.6 (4)	C9—C14—H14	118.9
C5—C4—N1	118.0 (4)	O4—C15—H15A	109.5
C3—C4—N1	118.4 (4)	O4—C15—H15B	109.5
C4—C5—C6	117.7 (4)	H15A—C15—H15B	109.5
C4—C5—H5A	121.2	O4—C15—H15C	109.5
C6—C5—H5A	121.2	H15A—C15—H15C	109.5
C1—C6—C5	120.9 (4)	H15B—C15—H15C	109.5
C1—C6—H6	119.5	O5—C16—H16A	109.5
C5—C6—H6	119.5	O5—C16—H16B	109.5
N2—C7—C1	120.1 (4)	H16A—C16—H16B	109.5
N2—C7—H7	120.0	O5—C16—H16C	109.5
C1—C7—H7	120.0	H16A—C16—H16C	109.5
O3—C8—N3	121.7 (4)	H16B—C16—H16C	109.5
O3—C8—C9	121.8 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O3	0.82	2.03	2.812 (4)	159
O5—H5···N2	0.82	2.61	3.194 (4)	129
N3—H3*A*···O5^{i}	0.90 (1)	2.02 (2)	2.900 (4)	166 (4)

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