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(*E*)-3-Bromo-*N*′-(4-methoxybenzylidene)benzohydrazide methanol solvate

Guo-Biao Cao

Department of Chemistry, Ankang University, Ankang Shanxi 725000, People's Republic of China
Correspondence e-mail: guobiao_cao@126.com

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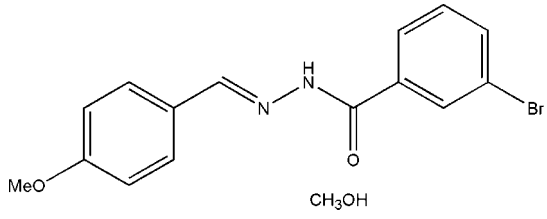
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.039; wR factor = 0.103; data-to-parameter ratio = 17.3.

The title compound, $C_{15}H_{13}BrN_2O_2\cdot CH_3OH$, was synthesized by the reaction of 4-methoxybenzaldehyde with an equimolar quantity of 3-bromobenzohydrazide in methanol. The benzohydrazide molecule displays an *E* configuration about the C=N bond. The dihedral angle between the two benzene rings is 4.0 (2)°. The benzohydrazide and methanol molecules are linked into a chain propagating along the *b* axis by O—H···O, O—H···N, N—H···O and C—H···O hydrogen bonds.

Related literature

For the crystal structures of hydrazone compounds, see: Mohd Lair *et al.* (2009); Fun *et al.* (2008); Li & Ban (2009); Zhu *et al.* (2009); Yang (2007); You *et al.* (2008). For hydrazone compounds reported previously by our group, see: Qu *et al.* (2008); Yang *et al.* (2008); Cao & Lu (2009*a*,*b*); Qu & Cao (2009); Cao & Wang (2009); Cao (2009).



Experimental

Crystal data

$C_{15}H_{13}BrN_2O_2\cdot CH_4O$
M_r = 365.23
Monoclinic, $P2_1/c$
a = 13.585 (1) Å
b = 6.715 (1) Å
c = 18.377 (1) Å
β = 104.429 (2)°
V = 1623.5 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.55 mm^{-1}
T = 298 K
0.20 × 0.20 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.630, T_{max} = 0.672
9539 measured reflections
3539 independent reflections
2132 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.103
S = 1.02
3539 reflections
205 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1	0.82	2.07	2.831 (3)	154
O3—H3···N2	0.82	2.60	3.211 (3)	132
N1—H1···O3^{i}	0.90 (1)	2.12 (1)	2.993 (3)	166 (3)
C6—H6···O3^{i}	0.93	2.49	3.406 (4)	168
C8—H8···O3^{i}	0.93	2.56	3.370 (3)	146

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

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*.

The Vital Foundation of Ankang University (project No. 2008AKXY012) and the Special Scientific Research Foundation of the Education Office of Shanxi Province (Project No. 02 J K202) are gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2872).

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supporting information

Acta Cryst. (2009). E**65**, o2086 [doi:10.1107/S1600536809030219]

(*E*)-3-Bromo-*N*′-(4-methoxybenzylidene)benzohydrazide methanol solvate

Guo-Biao Cao

S1. Comment

Study on the crystal structures of hydrazone derivatives is an interesting topic in structural chemistry. Recently, crystal structures of a number of hydrazone compounds have been reported (Mohd Lair *et al.*, 2009; Fun *et al.*, 2008; Li & Ban, 2009; Zhu *et al.*, 2009; Yang, 2007; You *et al.*, 2008). As a continuation of our work in this area (Qu *et al.*, 2008; Yang *et al.*, 2008; Cao & Lu, 2009a,b; Qu & Cao, 2009; Cao & Wang, 2009), the title new hydrazone compound derived from the reaction of 2-chlorobenzaldehyde with an equimolar quantity of 3-bromobenzohydrazide is reported.

The title compound (Fig. 1) consists of a hydrazone molecule and a methanol molecule of crystallization. The methanol molecule is linked to the hydrazone molecule through O—H···O and O—H···N hydrogen bonds (Table 1). The hydrazone molecule displays an *E* configuration about the C═N bond. The dihedral angle between the two benzene rings is 4.0 (2)°. In the crystal structure, molecules are linked through intermolecular N—H···O, O—H···O, O—H···N and C—H···O hydrogen bonds (Table 1) to form chains running along the *b* axis (Fig. 2).

S2. Experimental

The title compound was prepared by refluxing equimolar quantities of 4-methoxybenzaldehyde with 3-bromo-benzohydrazide in methanol. Colourless block-like crystals were formed by slow evaporation of the solution in air.

S3. Refinement

Atom H1 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 a O-H distance of 0.82 Å, C-H distances of 0.93-0.96 Å, and with U_{iso}(H) set at 1.2U_{eq}(C) and 1.5U_{eq}(methyl C).

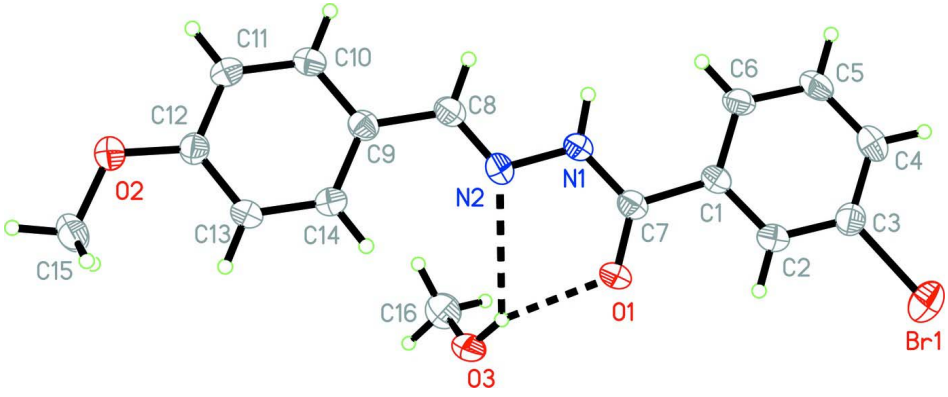

Figure 1

The asymmetric unit of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

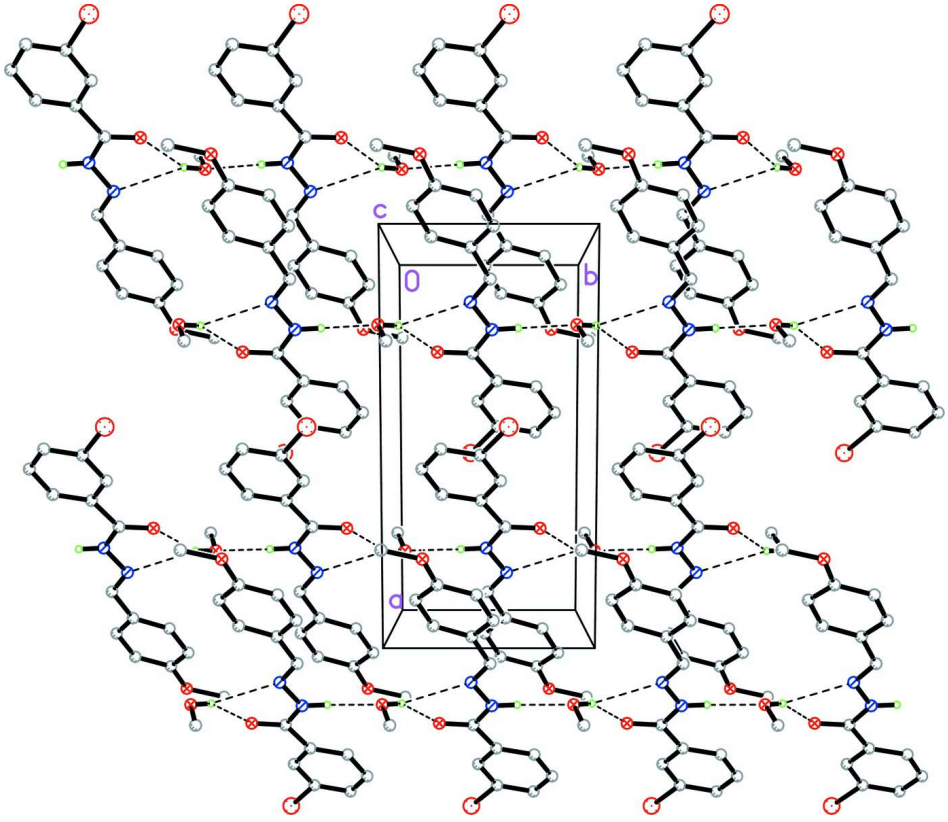

Figure 2

The crystal packing of the title compound, viewed along the *c* axis. Hydrogen bonds are shown as dashed lines. C-bound H atoms have been omitted for clarity.

(*E*)-3-Bromo-*N*′-(4-methoxybenzylidene)benzohydrazide methanol solvate

Crystal data

$C_{15}H_{13}BrN_2O_2{\cdot}CH_4O$
$M_r = 365.23$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.585\ (1)$ Å
$b = 6.715\ (1)$ Å
$c = 18.377\ (1)$ Å
$\beta = 104.429\ (2)°$
$V = 1623.5\ (3)$ Å^3
$Z = 4$
$F(000) = 744$
$D_x = 1.494$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2168 reflections
$\theta = 2.7–24.6°$
$\mu = 2.55$ mm^{-1}
$T = 298$ K
Block, colourless
0.20 × 0.20 × 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*; Bruker, 2001)
$T_{min} = 0.630$, $T_{max} = 0.672$
9539 measured reflections
3539 independent reflections
2132 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 27.0°$, $\theta_{min} = 1.6°$
$h = -17\rightarrow17$
$k = -8\rightarrow8$
$l = -23\rightarrow20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.103$
$S = 1.02$
3539 reflections
205 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
$w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 0.516P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.52$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2$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}
Br1	0.53424 (3)	0.09750 (6)	−0.18341 (2)	0.09185 (19)
O1	0.26855 (15)	0.2642 (3)	−0.02385 (11)	0.0608 (5)
O2	−0.19847 (15)	0.3318 (3)	0.21672 (11)	0.0595 (5)
O3	0.20352 (18)	0.5573 (3)	0.06465 (12)	0.0676 (6)
H3	0.2041	0.4585	0.0385	0.101*
N1	0.21308 (16)	−0.0152 (3)	0.02088 (12)	0.0460 (5)
N2	0.14486 (16)	0.0932 (3)	0.04946 (12)	0.0477 (5)
C1	0.34289 (18)	−0.0397 (4)	−0.04807 (14)	0.0410 (6)
C2	0.3955 (2)	0.0578 (4)	−0.09241 (14)	0.0470 (6)
H2	0.3858	0.1937	−0.1011	0.056*
C3	0.4622 (2)	−0.0442 (4)	−0.12395 (15)	0.0504 (7)
C4	0.4778 (2)	−0.2432 (4)	−0.11248 (16)	0.0573 (8)
H4	0.5230	−0.3111	−0.1341	0.069*
C5	0.4257 (2)	−0.3414 (4)	−0.06842 (17)	0.0560 (7)
H5	0.4360	−0.4773	−0.0602	0.067*
C6	0.3584 (2)	−0.2429 (4)	−0.03613 (15)	0.0488 (7)
H6	0.3236	−0.3120	−0.0065	0.059*
C7	0.27195 (19)	0.0826 (4)	−0.01586 (14)	0.0446 (6)
C8	0.0896 (2)	−0.0058 (4)	0.08234 (15)	0.0485 (6)
H8	0.0978	−0.1432	0.0862	0.058*
C9	0.01344 (18)	0.0883 (4)	0.11444 (14)	0.0430 (6)
C10	−0.0341 (2)	−0.0209 (4)	0.15986 (15)	0.0488 (7)
H10	−0.0179	−0.1549	0.1683	0.059*
C11	−0.1040 (2)	0.0628 (4)	0.19264 (15)	0.0519 (7)

H11	−0.1342	−0.0137	0.2232	0.062*
C12	−0.12967 (19)	0.2614 (4)	0.18031 (14)	0.0451 (6)
C13	−0.0856 (2)	0.3731 (4)	0.13385 (15)	0.0474 (6)
H13	−0.1038	0.5058	0.1242	0.057*
C14	−0.0142 (2)	0.2866 (4)	0.10182 (15)	0.0474 (6)
H14	0.0159	0.3630	0.0711	0.057*
C15	−0.2213 (2)	0.5384 (4)	0.21096 (18)	0.0662 (9)
H15A	−0.1611	0.6134	0.2330	0.099*
H15B	−0.2728	0.5674	0.2370	0.099*
H15C	−0.2455	0.5742	0.1590	0.099*
C16	0.2481 (3)	0.5111 (5)	0.13973 (19)	0.0749 (9)
H16A	0.3160	0.4636	0.1445	0.112*
H16B	0.2088	0.4098	0.1563	0.112*
H16C	0.2500	0.6283	0.1700	0.112*
H1	0.210 (2)	−0.1474 (16)	0.0258 (17)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1033 (3)	0.0942 (3)	0.0980 (3)	−0.0125 (2)	0.0627 (2)	0.0055 (2)
O1	0.0810 (14)	0.0302 (11)	0.0831 (14)	0.0074 (9)	0.0428 (11)	0.0037 (9)
O2	0.0673 (12)	0.0512 (11)	0.0699 (13)	0.0065 (9)	0.0358 (10)	0.0029 (10)
O3	0.1030 (16)	0.0346 (11)	0.0763 (15)	0.0028 (11)	0.0434 (13)	−0.0005 (10)
N1	0.0513 (13)	0.0327 (11)	0.0584 (14)	0.0025 (10)	0.0218 (11)	−0.0012 (11)
N2	0.0514 (13)	0.0417 (12)	0.0538 (14)	0.0046 (10)	0.0202 (11)	−0.0054 (11)
C1	0.0431 (14)	0.0351 (14)	0.0431 (15)	0.0001 (11)	0.0075 (12)	−0.0043 (11)
C2	0.0519 (16)	0.0369 (14)	0.0515 (16)	−0.0012 (12)	0.0112 (13)	0.0010 (12)
C3	0.0512 (16)	0.0540 (18)	0.0481 (16)	−0.0026 (13)	0.0163 (13)	−0.0029 (13)
C4	0.0560 (18)	0.0532 (18)	0.0635 (19)	0.0122 (14)	0.0167 (15)	−0.0099 (15)
C5	0.0638 (19)	0.0358 (15)	0.0686 (19)	0.0104 (13)	0.0172 (16)	−0.0023 (14)
C6	0.0544 (17)	0.0328 (14)	0.0599 (17)	0.0010 (12)	0.0153 (14)	0.0017 (12)
C7	0.0483 (15)	0.0376 (16)	0.0471 (15)	0.0051 (12)	0.0105 (12)	−0.0003 (12)
C8	0.0520 (17)	0.0381 (14)	0.0564 (17)	0.0032 (13)	0.0153 (14)	−0.0028 (13)
C9	0.0411 (14)	0.0406 (15)	0.0453 (15)	−0.0004 (12)	0.0071 (12)	−0.0041 (12)
C10	0.0567 (17)	0.0361 (14)	0.0548 (16)	0.0020 (12)	0.0161 (14)	0.0028 (13)
C11	0.0606 (18)	0.0443 (16)	0.0555 (17)	−0.0027 (13)	0.0231 (14)	0.0082 (13)
C12	0.0454 (15)	0.0463 (16)	0.0459 (15)	−0.0012 (12)	0.0158 (12)	−0.0027 (12)
C13	0.0538 (16)	0.0339 (14)	0.0559 (17)	0.0038 (12)	0.0165 (13)	0.0013 (12)
C14	0.0537 (16)	0.0397 (15)	0.0520 (16)	−0.0033 (12)	0.0189 (13)	0.0029 (13)
C15	0.073 (2)	0.059 (2)	0.073 (2)	0.0194 (16)	0.0303 (17)	−0.0001 (16)
C16	0.086 (2)	0.064 (2)	0.078 (3)	−0.0032 (18)	0.028 (2)	−0.0082 (19)

Geometric parameters (Å, º)

Br1—C3	1.895 (3)	C6—H6	0.93
O1—C7	1.227 (3)	C8—C9	1.456 (3)
O2—C12	1.363 (3)	C8—H8	0.93
O2—C15	1.420 (3)	C9—C10	1.385 (4)

O3—C16	1.396 (4)	C9—C14	1.387 (3)
O3—H3	0.82	C10—C11	1.366 (4)
N1—C7	1.340 (3)	C10—H10	0.93
N1—N2	1.381 (3)	C11—C12	1.382 (4)
N1—H1	0.895 (10)	C11—H11	0.93
N2—C8	1.264 (3)	C12—C13	1.380 (3)
C1—C2	1.376 (3)	C13—C14	1.381 (3)
C1—C6	1.390 (4)	C13—H13	0.93
C1—C7	1.496 (3)	C14—H14	0.93
C2—C3	1.374 (3)	C15—H15A	0.96
C2—H2	0.93	C15—H15B	0.96
C3—C4	1.361 (4)	C15—H15C	0.96
C4—C5	1.370 (4)	C16—H16A	0.96
C4—H4	0.93	C16—H16B	0.96
C5—C6	1.376 (4)	C16—H16C	0.96
C5—H5	0.93		

C12—O2—C15	117.8 (2)	C10—C9—C14	117.5 (2)
C16—O3—H3	109.5	C10—C9—C8	119.9 (2)
C7—N1—N2	118.3 (2)	C14—C9—C8	122.6 (2)
C7—N1—H1	126 (2)	C11—C10—C9	121.8 (2)
N2—N1—H1	115 (2)	C11—C10—H10	119.1
C8—N2—N1	116.1 (2)	C9—C10—H10	119.1
C2—C1—C6	118.7 (2)	C10—C11—C12	120.0 (2)
C2—C1—C7	117.0 (2)	C10—C11—H11	120.0
C6—C1—C7	124.4 (2)	C12—C11—H11	120.0
C3—C2—C1	120.4 (2)	O2—C12—C13	124.9 (2)
C3—C2—H2	119.8	O2—C12—C11	115.5 (2)
C1—C2—H2	119.8	C13—C12—C11	119.6 (2)
C4—C3—C2	121.2 (2)	C12—C13—C14	119.6 (2)
C4—C3—Br1	119.9 (2)	C12—C13—H13	120.2
C2—C3—Br1	118.9 (2)	C14—C13—H13	120.2
C3—C4—C5	118.7 (2)	C13—C14—C9	121.5 (2)
C3—C4—H4	120.7	C13—C14—H14	119.3
C5—C4—H4	120.7	C9—C14—H14	119.3
C4—C5—C6	121.3 (3)	O2—C15—H15A	109.5
C4—C5—H5	119.3	O2—C15—H15B	109.5
C6—C5—H5	119.3	H15A—C15—H15B	109.5
C5—C6—C1	119.6 (3)	O2—C15—H15C	109.5
C5—C6—H6	120.2	H15A—C15—H15C	109.5
C1—C6—H6	120.2	H15B—C15—H15C	109.5
O1—C7—N1	122.5 (2)	O3—C16—H16A	109.5
O1—C7—C1	120.5 (2)	O3—C16—H16B	109.5
N1—C7—C1	117.0 (2)	H16A—C16—H16B	109.5
N2—C8—C9	122.1 (2)	O3—C16—H16C	109.5
N2—C8—H8	118.9	H16A—C16—H16C	109.5
C9—C8—H8	118.9	H16B—C16—H16C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O1	0.82	2.07	2.831 (3)	154
O3—H3···N2	0.82	2.60	3.211 (3)	132
N1—H1···O3^{i}	0.90 (1)	2.12 (1)	2.993 (3)	166 (3)
C6—H6···O3^{i}	0.93	2.49	3.406 (4)	168
C8—H8···O3^{i}	0.93	2.56	3.370 (3)	146

Symmetry code: (i) *x*, *y*−1, *z*.