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(*E*)-*N*′-(5-Bromo-2-methoxybenzylidene)-4-methoxybenzohydrazide

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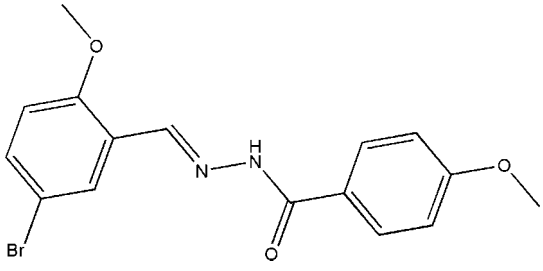
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.038; wR factor = 0.093; data-to-parameter ratio = 15.8.

In the title compound, $C_{16}H_{15}BrN_2O_3$, the benzohydrazide group is not planar and the molecule exists in a *trans* configuration with respect to the methylidene unit. The dihedral angle between the two substituted benzene rings is 75.6 (2)°. In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds involving carbonyl and amine functionalities, to form chains parallel to the *c* cell axis.

Related literature

For the biological activities of hydrazones, see: Zhong *et al.* (2007); Raj *et al.* (2007); Jimenez-Pulido *et al.* (2008). For related structures, see: 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

$C_{16}H_{15}BrN_2O_3$
M_r = 363.21
Monoclinic, $P2_1/c$
a = 12.438 (4) Å
b = 16.684 (6) Å
c = 7.863 (3) Å
β = 108.218 (6)°
V = 1549.8 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.67 mm^{-1}
T = 298 (2) K
0.20 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.593, T_{max} = 0.620
8697 measured reflections
3245 independent reflections
2068 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.093
S = 1.01
3245 reflections
205 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.890 (10)	1.966 (12)	2.835 (3)	165 (2)

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

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: BH2202).

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supplementary materials

Acta Cryst. (2008). E**64**, o2260 [**doi:10.1107/S1600536808035472**]

(*E*)-*N*'-(5-Bromo-2-methoxybenzylidene)-4-methoxybenzohydrazide

H.-Y. Ban and C.-M. Li

Comment

Hydrazones 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 great deal of hydrazones 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). We report herein the crystal structure of the title new compound, (I).

In the structure of the title compound (Fig. 1) the molecule exists in a *trans* configuration with respect to the methylidene unit. The dihedral angle between the two substituted benzene rings is 75.6 (2)°. In both the 5-bromo-2-methoxyphenyl unit and the 4-methoxyphenyl unit, the methoxy groups are nearly coplanar with the corresponding mean planes of the C1···C6 and C9···C14 rings, respectively. Atoms C16 and C15 deviate from their corresponding benzene rings by 0.058 (2) and 0.059 (2) Å respectively. The torsion angle of C7—N1—N2—C8 is 19.4 (3)°. The bond lengths and angles are found in expected ranges.

In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds involving carbonyl and amine groups (Table 1), to form chains parallel to the *c* axis (Fig. 2).

Experimental

The compound was prepared by refluxing 5-bromo-2-methoxybenzaldehyde (1.0 mmol) with 4-methoxybenzohydrazide (1.0 mmol) in methanol (100 ml). Excess methanol was removed from the mixture by distillation. The colorless solid product was filtered, and washed three times with methanol. Colorless block crystals of the title compound were obtained from a methanol solution by slow evaporation in air.

Refinement

H2 was located in a difference map and refined isotropically, with N2—H2 distance restrained to 0.90 (1) Å. Other H atoms were placed in calculated positions (C—H = 0.93–0.96 Å) and refined as riding atoms with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(methyl C). A rotating group model was used for the methyl groups.

Figures

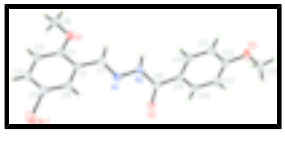

Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids for the non-hydrogen atoms.

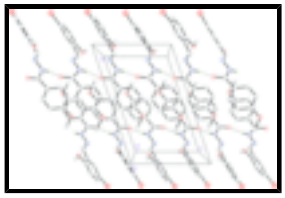

Fig. 2. The packing diagram of (I), viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

(*E*)-*N*'-(5-Bromo-2-methoxybenzylidene)-4-methoxybenzohydrazide

Crystal data

$C_{16}H_{15}BrN_2O_3$	F_{000} = 736
M_r = 363.21	D_x = 1.557 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2110 reflections
a = 12.438 (4) Å	θ = 2.4–24.5º
b = 16.684 (6) Å	μ = 2.67 mm^{-1}
c = 7.863 (3) Å	T = 298 (2) K
β = 108.218 (6)º	Block, colorless
V = 1549.8 (9) Å^3	0.20 × 0.20 × 0.18 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3245 independent reflections
Radiation source: fine-focus sealed tube	2068 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.034
T = 298(2) K	θ_{max} = 26.7º
ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	h = −14→15
T_{min} = 0.593, T_{max} = 0.620	k = −20→21
8697 measured reflections	l = −9→9

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.038	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.093	$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2 + 0.1883P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max}$ = 0.001
3245 reflections	$\Delta\rho_{max}$ = 0.50 e Å^{-3}
205 parameters	$\Delta\rho_{min}$ = −0.47 e Å^{-3}

1 restraint
Primary atom site location: structure-invariant direct methods
Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	−0.31436 (3)	0.22534 (2)	0.94364 (5)	0.07161 (17)
N1	0.10628 (17)	0.23762 (13)	0.8539 (3)	0.0380 (5)
N2	0.19688 (18)	0.26531 (13)	0.8053 (3)	0.0384 (5)
O1	0.01300 (15)	0.01461 (11)	0.7292 (3)	0.0509 (5)
O2	0.26258 (15)	0.33509 (11)	1.0641 (2)	0.0455 (5)
O3	0.61120 (16)	0.44804 (12)	0.6741 (3)	0.0603 (6)
C1	−0.0401 (2)	0.14085 (16)	0.8049 (3)	0.0383 (6)
C2	−0.0634 (2)	0.05851 (16)	0.7802 (3)	0.0414 (7)
C3	−0.1589 (2)	0.02734 (18)	0.8110 (4)	0.0490 (7)
H3	−0.1738	−0.0273	0.7967	0.059*
C4	−0.2318 (2)	0.07627 (19)	0.8625 (4)	0.0498 (8)
H4	−0.2957	0.0548	0.8828	0.060*
C5	−0.2101 (2)	0.15743 (18)	0.8841 (4)	0.0460 (7)
C6	−0.1148 (2)	0.18901 (17)	0.8578 (3)	0.0429 (7)
H6	−0.1000	0.2435	0.8757	0.051*
C7	0.0603 (2)	0.17374 (16)	0.7739 (3)	0.0389 (6)
H7	0.0915	0.1481	0.6953	0.047*
C8	0.2703 (2)	0.31655 (15)	0.9165 (4)	0.0369 (6)
C9	0.3602 (2)	0.34885 (15)	0.8498 (3)	0.0354 (6)
C10	0.3494 (2)	0.35720 (16)	0.6705 (4)	0.0425 (7)
H10	0.2833	0.3400	0.5852	0.051*
C11	0.4338 (2)	0.39027 (18)	0.6149 (4)	0.0493 (7)
H11	0.4246	0.3954	0.4934	0.059*
C12	0.5326 (2)	0.41596 (16)	0.7408 (4)	0.0432 (7)
C13	0.5452 (2)	0.40845 (18)	0.9203 (4)	0.0482 (7)
H13	0.6115	0.4256	1.0054	0.058*
C14	0.4593 (2)	0.37549 (17)	0.9738 (4)	0.0462 (7)
H14	0.4681	0.3711	1.0953	0.055*
C15	0.7161 (3)	0.4720 (2)	0.7978 (5)	0.0739 (11)
H15A	0.7031	0.5129	0.8749	0.111*
H15B	0.7644	0.4926	0.7338	0.111*
H15C	0.7517	0.4267	0.8684	0.111*
C16	−0.0095 (3)	−0.06880 (17)	0.6972 (4)	0.0563 (8)
H16A	−0.0110	−0.0943	0.8059	0.084*
H16B	0.0488	−0.0925	0.6575	0.084*
H16C	−0.0814	−0.0757	0.6068	0.084*
H2	0.212 (2)	0.2407 (14)	0.715 (3)	0.044 (8)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0574 (2)	0.0821 (3)	0.0863 (3)	0.00286 (18)	0.0383 (2)	−0.0118 (2)

N1	0.0331 (12)	0.0430 (14)	0.0405 (13)	−0.0059 (10)	0.0154 (10)	−0.0001 (10)
N2	0.0398 (13)	0.0409 (13)	0.0403 (14)	−0.0086 (11)	0.0208 (11)	−0.0065 (11)
O1	0.0498 (12)	0.0413 (11)	0.0670 (14)	−0.0080 (9)	0.0260 (11)	−0.0072 (10)
O2	0.0488 (11)	0.0473 (11)	0.0470 (12)	−0.0117 (9)	0.0244 (10)	−0.0083 (9)
O3	0.0472 (13)	0.0689 (14)	0.0728 (15)	−0.0201 (11)	0.0300 (11)	−0.0025 (12)
C1	0.0363 (15)	0.0414 (16)	0.0361 (15)	−0.0074 (12)	0.0098 (13)	−0.0008 (12)
C2	0.0401 (16)	0.0453 (17)	0.0371 (16)	−0.0058 (13)	0.0098 (13)	−0.0017 (13)
C3	0.0483 (17)	0.0462 (17)	0.0525 (19)	−0.0146 (14)	0.0158 (15)	−0.0021 (14)
C4	0.0411 (17)	0.060 (2)	0.0503 (18)	−0.0150 (15)	0.0176 (15)	0.0009 (15)
C5	0.0404 (16)	0.058 (2)	0.0410 (17)	−0.0019 (14)	0.0145 (14)	0.0009 (14)
C6	0.0437 (16)	0.0433 (16)	0.0412 (17)	−0.0049 (13)	0.0125 (14)	−0.0019 (13)
C7	0.0372 (15)	0.0417 (16)	0.0404 (16)	−0.0035 (13)	0.0158 (13)	−0.0020 (13)
C8	0.0368 (15)	0.0318 (14)	0.0436 (17)	0.0025 (12)	0.0147 (13)	0.0013 (12)
C9	0.0328 (14)	0.0328 (14)	0.0422 (17)	−0.0033 (11)	0.0142 (13)	−0.0010 (12)
C10	0.0350 (15)	0.0479 (17)	0.0422 (17)	−0.0075 (13)	0.0086 (13)	−0.0016 (13)
C11	0.0504 (18)	0.0560 (19)	0.0450 (17)	−0.0098 (14)	0.0201 (15)	0.0023 (14)
C12	0.0365 (16)	0.0385 (15)	0.057 (2)	−0.0043 (12)	0.0184 (15)	−0.0021 (13)
C13	0.0340 (16)	0.0556 (19)	0.054 (2)	−0.0106 (13)	0.0128 (14)	−0.0093 (15)
C14	0.0453 (17)	0.0554 (18)	0.0400 (17)	−0.0063 (14)	0.0165 (14)	−0.0055 (14)
C15	0.048 (2)	0.087 (3)	0.096 (3)	−0.0268 (18)	0.036 (2)	−0.017 (2)
C16	0.062 (2)	0.0428 (18)	0.067 (2)	−0.0070 (15)	0.0239 (17)	−0.0084 (15)

Geometric parameters (Å, °)

Br1—C5	1.888 (3)	C6—H6	0.9300
N1—C7	1.278 (3)	C7—H7	0.9300
N1—N2	1.378 (3)	C8—C9	1.477 (3)
N2—C8	1.352 (3)	C9—C10	1.381 (4)
N2—H2	0.890 (10)	C9—C14	1.385 (4)
O1—C2	1.355 (3)	C10—C11	1.372 (4)
O1—C16	1.426 (3)	C10—H10	0.9300
O2—C8	1.233 (3)	C11—C12	1.383 (4)
O3—C12	1.355 (3)	C11—H11	0.9300
O3—C15	1.419 (4)	C12—C13	1.377 (4)
C1—C6	1.386 (4)	C13—C14	1.379 (4)
C1—C2	1.405 (4)	C13—H13	0.9300
C1—C7	1.453 (3)	C14—H14	0.9300
C2—C3	1.386 (4)	C15—H15A	0.9600
C3—C4	1.371 (4)	C15—H15B	0.9600
C3—H3	0.9300	C15—H15C	0.9600
C4—C5	1.381 (4)	C16—H16A	0.9600
C4—H4	0.9300	C16—H16B	0.9600
C5—C6	1.371 (4)	C16—H16C	0.9600
C7—N1—N2	115.0 (2)	C10—C9—C14	117.8 (2)
C8—N2—N1	118.5 (2)	C10—C9—C8	123.9 (2)
C8—N2—H2	122.6 (17)	C14—C9—C8	118.2 (2)
N1—N2—H2	117.5 (17)	C11—C10—C9	121.8 (3)
C2—O1—C16	117.6 (2)	C11—C10—H10	119.1
C12—O3—C15	117.8 (2)	C9—C10—H10	119.1

C6—C1—C2	118.7 (2)	C10—C11—C12	119.6 (3)
C6—C1—C7	121.5 (2)	C10—C11—H11	120.2
C2—C1—C7	119.8 (2)	C12—C11—H11	120.2
O1—C2—C3	124.6 (3)	O3—C12—C13	124.6 (2)
O1—C2—C1	115.9 (2)	O3—C12—C11	115.6 (3)
C3—C2—C1	119.5 (3)	C13—C12—C11	119.7 (3)
C4—C3—C2	120.7 (3)	C12—C13—C14	119.9 (3)
C4—C3—H3	119.7	C12—C13—H13	120.0
C2—C3—H3	119.7	C14—C13—H13	120.0
C3—C4—C5	119.9 (3)	C13—C14—C9	121.2 (3)
C3—C4—H4	120.0	C13—C14—H14	119.4
C5—C4—H4	120.0	C9—C14—H14	119.4
C6—C5—C4	120.2 (3)	O3—C15—H15A	109.5
C6—C5—Br1	120.0 (2)	O3—C15—H15B	109.5
C4—C5—Br1	119.8 (2)	H15A—C15—H15B	109.5
C5—C6—C1	121.0 (3)	O3—C15—H15C	109.5
C5—C6—H6	119.5	H15A—C15—H15C	109.5
C1—C6—H6	119.5	H15B—C15—H15C	109.5
N1—C7—C1	120.5 (2)	O1—C16—H16A	109.5
N1—C7—H7	119.7	O1—C16—H16B	109.5
C1—C7—H7	119.7	H16A—C16—H16B	109.5
O2—C8—N2	122.1 (2)	O1—C16—H16C	109.5
O2—C8—C9	122.2 (2)	H16A—C16—H16C	109.5
N2—C8—C9	115.7 (2)	H16B—C16—H16C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{i}	0.890 (10)	1.966 (12)	2.835 (3)	165 (2)

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2.

Fig. 1

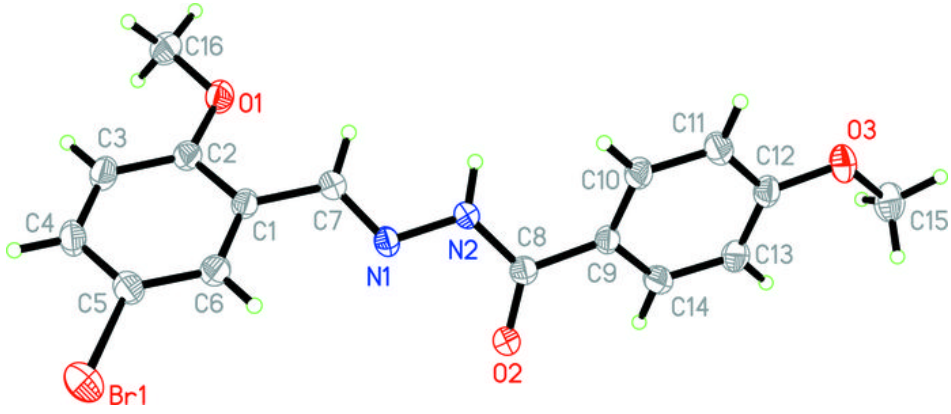
C16
O1
C3
C2
C4
C1
C7
N1
N2
C8
C5
C6
O2
Br1
C11
O3
C10
C12
C15
C9
C13
C14

Fig. 2