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2-Bromo-*N*′-isopropylidene-5-methoxybenzohydrazide

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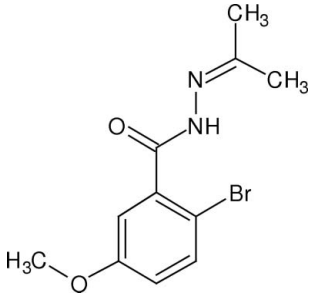
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.086; data-to-parameter ratio = 22.2.

The geometric parameters of the title compound, $C_{11}H_{13}BrN_2O_2$, are in the usual ranges. In the crystal structure, the molecules are connected by N—H···O hydrogen bonds to form centrosymmetric dimers.

Related literature

For related structures, see: Narayana, Sunil *et al.* (2007); Yathirajan, Sarojini *et al.* (2007); Yathirajan, Narayana *et al.* (2007).

For related literature, see: Swain (1959); Hodnett & Dunn (1970); Cajocorius *et al.* (1977); Misra *et al.* (1981); Agarwal *et al.* (1983); Varma *et al.* (1986); Singh & Dash (1988); Narayana, Vijayaraj *et al.* (2005); Narayana, Ashalatha *et al.* (2005); Liu *et al.* (2006).



Experimental

Crystal data

$C_{11}H_{13}BrN_2O_2$
M_r = 285.14
Monoclinic, $P2_1/c$
a = 7.8311 (5) Å
b = 13.6460 (7) Å
c = 11.3800 (8) Å
β = 92.005 (5)°
V = 1215.36 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.37 mm^{-1}
T = 173 (2) K
0.29 × 0.27 × 0.25 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.398, T_{max} = 0.438
27762 measured reflections
3404 independent reflections
2995 reflections with $I > 2\sigma(I)$
R_{int} = 0.080

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.086
S = 1.17
3404 reflections
153 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.85 (3)	2.08 (3)	2.916 (2)	168 (3)

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

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2187).

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supplementary materials

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2-Bromo-*N*'-isopropylidene-5-methoxybenzohydrazide

B. K. Sarojini, H. S. Yathirajan, K. Sunil, B. Narayana and M. Bolte

Comment

Hydrazides and their Schiff bases are useful precursors in the synthesis of several heterocyclic systems. Some substituted hydrazides and their Schiff bases are reported to exhibit carcinostatic activity against several types of tumors and also possess antimicrobial activity It is also used as an intermediate in many pharmaceutically important compounds. A new Schiff base of the hydrazide, $C_{11}H_{13}BrN_2O_2$ was synthesized and its crystal structure is reported.

Geometric parameters of the title compound (Fig. 1) are in the usual ranges. All seven non-H atoms of the acetone hydrazone moiety lie in a common plane (r.m.s. deviation 0.007 Å) which is almost perpendicular [88.52 (6)°] to the aromatic ring. In the crystal, the molecules are connected by N—H···O hydrogen bonds to form centrosymmetric dimers (Fig. 2).

Experimental

A mixture of 2-bromo-5-methoxybenzohydrazide (2.45 g, 0.01 mol) and acetone (1.2 g, 0.02 mol) in 15 ml of absolute ethanol containing 1 drop of dilute sulfuric acid was refluxed for about 4 h. On cooling, the solid that separated was filtered and recrystallized from ethyl acetate (m.p.: 404–406 K). Analysis for $C_{11}H_{13}BrN_2O_2$: Found (Calculated): C: 46.21 (46.33); H: 4.54 (4.60); N: 9.73% (9.82%).

Refinement

H atoms were found in a difference map, but they were refined using a riding model with C—H = 0.95Å and U_{iso}(H) = 1.2U_{eq}(C).

Figures

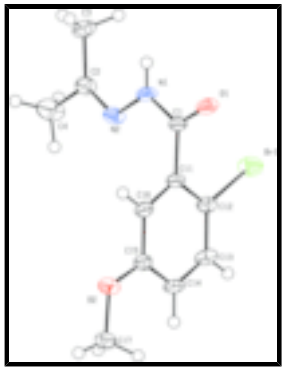
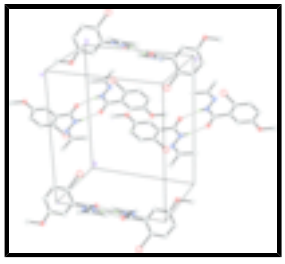


Fig. 1. Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level.



2-Bromo-*N*'-isopropylidene-5-methoxybenzohydrazide

Crystal data

$C_{11}H_{13}BrN_2O_2$	F_{000} = 576
M_r = 285.14	D_x = 1.558 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 13368 reflections
a = 7.8311 (5) Å	θ = 3.6–29.5º
b = 13.6460 (7) Å	μ = 3.37 mm^{-1}
c = 11.3800 (8) Å	T = 173 (2) K
β = 92.005 (5)º	Block, colourless
V = 1215.36 (13) Å^3	0.29 × 0.27 × 0.25 mm
Z = 4	

Data collection

Stoe IPDS II two-circle-diffractometer	3404 independent reflections
Radiation source: fine-focus sealed tube	2995 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.080
T = 173(2) K	θ_{max} = 29.6º
ω scans	θ_{min} = 3.6º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	h = −10→10
T_{min} = 0.398, T_{max} = 0.438	k = −18→16
27762 measured reflections	l = −15→15

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.040	$w = 1/[\sigma^2(F_o^2) + (0.0262P)^2 + 0.9527P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.086	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.17	$\Delta\rho_{max}$ = 0.47 e Å^{-3}

3404 reflections	$\Delta\rho_{min} = -0.53$ e Å^{-3}
153 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0108 (9)
Secondary atom site location: difference Fourier map	

Special details

Experimental. ;

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 $F^2 > 2\text{sigma}(F^2)$ 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	−0.02400 (3)	0.755039 (17)	0.73418 (2)	0.03436 (10)
N1	0.1881 (2)	0.56917 (15)	0.92316 (15)	0.0241 (4)
H1	0.163 (4)	0.553 (2)	0.992 (2)	0.029 (7)*
N2	0.3432 (2)	0.61293 (15)	0.89571 (15)	0.0263 (4)
O1	−0.0623 (2)	0.50527 (13)	0.85582 (13)	0.0299 (4)
O2	0.3225 (2)	0.42571 (13)	0.48012 (14)	0.0322 (4)
C1	0.0758 (3)	0.54460 (15)	0.83589 (17)	0.0213 (4)
C2	0.4445 (3)	0.63696 (17)	0.98164 (19)	0.0266 (4)
C3	0.4144 (3)	0.6235 (2)	1.11038 (19)	0.0342 (5)
H3A	0.3039	0.6521	1.1292	0.051*
H3B	0.5056	0.6561	1.1568	0.051*
H3C	0.4141	0.5534	1.1291	0.051*
C4	0.6117 (3)	0.6825 (2)	0.9500 (2)	0.0433 (7)
H4A	0.6176	0.6864	0.8642	0.065*
H4B	0.7062	0.6422	0.9814	0.065*
H4C	0.6201	0.7485	0.9836	0.065*
C11	0.1243 (2)	0.56409 (16)	0.71060 (16)	0.0200 (4)
C12	0.0821 (3)	0.65109 (16)	0.65255 (18)	0.0229 (4)
C13	0.1178 (3)	0.66386 (17)	0.53468 (18)	0.0274 (4)
H13	0.0881	0.7235	0.4961	0.033*
C14	0.1971 (3)	0.58937 (17)	0.47296 (17)	0.0264 (4)
H14	0.2215	0.5980	0.3924	0.032*
C15	0.2403 (3)	0.50254 (16)	0.52987 (17)	0.0230 (4)
C16	0.2025 (3)	0.48971 (16)	0.64868 (17)	0.0226 (4)
H16	0.2306	0.4297	0.6870	0.027*

C17	0.3679 (3)	0.4361 (2)	0.35960 (19)	0.0350 (5)
H17A	0.2639	0.4419	0.3097	0.053*
H17B	0.4329	0.3785	0.3358	0.053*
H17C	0.4379	0.4951	0.3511	0.053*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.04829 (16)	0.02903 (13)	0.02596 (13)	0.00271 (11)	0.00392 (9)	−0.00287 (9)
N1	0.0230 (8)	0.0368 (10)	0.0127 (7)	−0.0085 (7)	0.0021 (6)	0.0033 (7)
N2	0.0242 (9)	0.0351 (10)	0.0199 (8)	−0.0105 (7)	0.0052 (7)	−0.0027 (7)
O1	0.0236 (8)	0.0489 (10)	0.0173 (7)	−0.0125 (7)	−0.0005 (6)	0.0073 (7)
O2	0.0361 (9)	0.0403 (9)	0.0204 (7)	0.0059 (7)	0.0062 (6)	−0.0018 (7)
C1	0.0222 (9)	0.0275 (10)	0.0142 (8)	−0.0030 (7)	0.0018 (7)	0.0038 (7)
C2	0.0251 (10)	0.0322 (11)	0.0226 (10)	−0.0060 (8)	0.0032 (8)	−0.0028 (8)
C3	0.0315 (12)	0.0510 (15)	0.0199 (10)	−0.0089 (11)	−0.0038 (8)	0.0015 (10)
C4	0.0359 (13)	0.0602 (18)	0.0341 (13)	−0.0239 (13)	0.0062 (11)	−0.0121 (12)
C11	0.0192 (9)	0.0281 (10)	0.0129 (8)	−0.0068 (7)	0.0004 (7)	0.0020 (7)
C12	0.0263 (10)	0.0255 (10)	0.0169 (9)	−0.0041 (8)	0.0005 (7)	−0.0020 (7)
C13	0.0375 (12)	0.0279 (10)	0.0166 (9)	−0.0039 (9)	−0.0007 (8)	0.0055 (8)
C14	0.0298 (11)	0.0376 (12)	0.0119 (8)	−0.0059 (9)	0.0012 (7)	0.0032 (8)
C15	0.0205 (9)	0.0321 (11)	0.0164 (8)	−0.0039 (8)	−0.0002 (7)	−0.0009 (8)
C16	0.0225 (10)	0.0282 (10)	0.0168 (9)	−0.0028 (8)	−0.0006 (7)	0.0045 (7)
C17	0.0291 (11)	0.0572 (16)	0.0189 (10)	0.0024 (11)	0.0031 (8)	−0.0072 (10)

Geometric parameters (Å, °)

Br1—C12	1.902 (2)	C4—H4B	0.9800
N1—C1	1.346 (3)	C4—H4C	0.9800
N1—N2	1.398 (2)	C11—C16	1.390 (3)
N1—H1	0.85 (3)	C11—C12	1.393 (3)
N2—C2	1.280 (3)	C12—C13	1.391 (3)
O1—C1	1.235 (2)	C13—C14	1.394 (3)
O2—C15	1.364 (3)	C13—H13	0.9500
O2—C17	1.436 (3)	C14—C15	1.386 (3)
C1—C11	1.512 (3)	C14—H14	0.9500
C2—C3	1.503 (3)	C15—C16	1.405 (3)
C2—C4	1.505 (3)	C16—H16	0.9500
C3—H3A	0.9800	C17—H17A	0.9800
C3—H3B	0.9800	C17—H17B	0.9800
C3—H3C	0.9800	C17—H17C	0.9800
C4—H4A	0.9800		
C1—N1—N2	119.46 (16)	C16—C11—C1	118.64 (18)
C1—N1—H1	116.7 (19)	C12—C11—C1	122.28 (18)
N2—N1—H1	123.7 (19)	C13—C12—C11	120.8 (2)
C2—N2—N1	117.28 (17)	C13—C12—Br1	118.92 (17)
C15—O2—C17	117.17 (19)	C11—C12—Br1	120.29 (15)
O1—C1—N1	121.77 (18)	C12—C13—C14	120.2 (2)

O1—C1—C11	120.00 (18)	C12—C13—H13	119.9
N1—C1—C11	118.21 (17)	C14—C13—H13	119.9
N2—C2—C3	126.8 (2)	C15—C14—C13	119.57 (18)
N2—C2—C4	116.3 (2)	C15—C14—H14	120.2
C3—C2—C4	116.8 (2)	C13—C14—H14	120.2
C2—C3—H3A	109.5	O2—C15—C14	125.03 (18)
C2—C3—H3B	109.5	O2—C15—C16	114.97 (19)
H3A—C3—H3B	109.5	C14—C15—C16	120.0 (2)
C2—C3—H3C	109.5	C11—C16—C15	120.54 (19)
H3A—C3—H3C	109.5	C11—C16—H16	119.7
H3B—C3—H3C	109.5	C15—C16—H16	119.7
C2—C4—H4A	109.5	O2—C17—H17A	109.5
C2—C4—H4B	109.5	O2—C17—H17B	109.5
H4A—C4—H4B	109.5	H17A—C17—H17B	109.5
C2—C4—H4C	109.5	O2—C17—H17C	109.5
H4A—C4—H4C	109.5	H17A—C17—H17C	109.5
H4B—C4—H4C	109.5	H17B—C17—H17C	109.5
C16—C11—C12	118.91 (17)		
C1—N1—N2—C2	178.5 (2)	C1—C11—C12—Br1	5.2 (3)
N2—N1—C1—O1	179.5 (2)	C11—C12—C13—C14	−0.2 (3)
N2—N1—C1—C11	1.0 (3)	Br1—C12—C13—C14	179.32 (17)
N1—N2—C2—C3	−0.2 (4)	C12—C13—C14—C15	−0.1 (3)
N1—N2—C2—C4	179.1 (2)	C17—O2—C15—C14	0.2 (3)
O1—C1—C11—C16	−85.5 (3)	C17—O2—C15—C16	−178.48 (19)
N1—C1—C11—C16	93.0 (2)	C13—C14—C15—O2	−178.0 (2)
O1—C1—C11—C12	89.7 (3)	C13—C14—C15—C16	0.6 (3)
N1—C1—C11—C12	−91.8 (3)	C12—C11—C16—C15	0.8 (3)
C16—C11—C12—C13	−0.2 (3)	C1—C11—C16—C15	176.12 (18)
C1—C11—C12—C13	−175.3 (2)	O2—C15—C16—C11	177.74 (19)
C16—C11—C12—Br1	−179.67 (15)	C14—C15—C16—C11	−1.0 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.85 (3)	2.08 (3)	2.916 (2)	168 (3)

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

Fig. 1

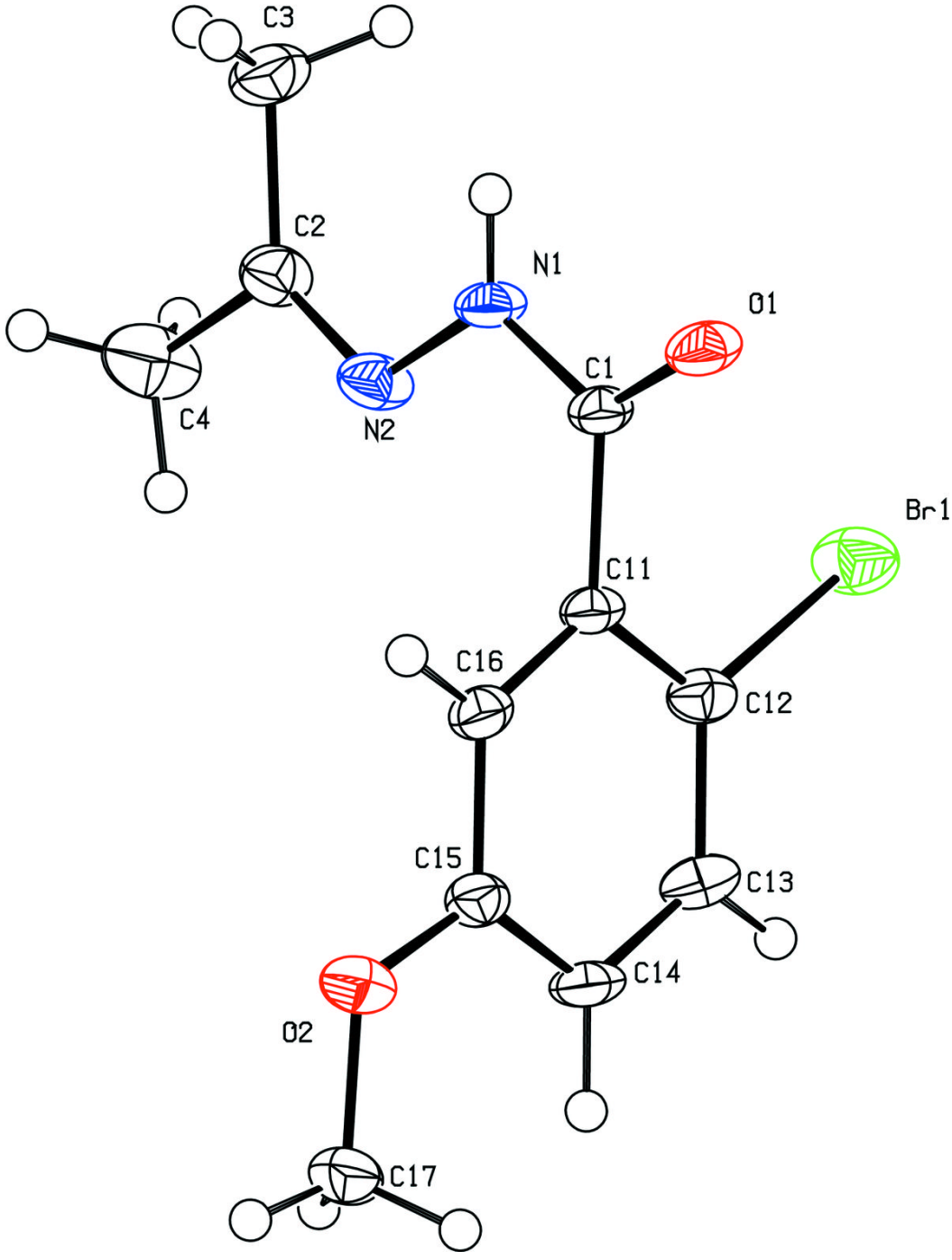


Fig. 2