Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Bromo-5-methoxybenzohydrazide

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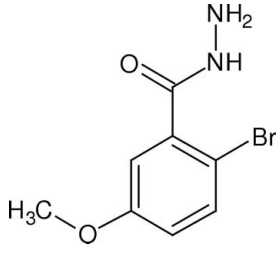
Received 15 October 2007; accepted 17 October 2007

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.006 Å; R factor = 0.046; wR factor = 0.116; data-to-parameter ratio = 16.6.

In the title compound, $C_8H_9BrN_2O_2$, the amide bond is in a *cis* configuation. The N—N—C=O group is planar [torsion angle 0.6 (6)°] and forms a dihedral angle of 46.4 (2)° with the aromatic ring. The crystal packing is stabilized by two strong and one rather weak N—H···O hydrogen bonds.

Related literature

For related literature, see: Bhat *et al.* (1974); Cajocorius *et al.* (1977); Katrusiak (1993); Liu *et al.* (2006); Muir & Morris (2003); Narayana, Ashalatha *et al.* (2005); Narayana, Vijayaraj *et al.* (2005); Sarojini *et al.* (2007); Swain (1959).



Experimental

Crystal data

$C_8H_9BrN_2O_2$
M_r = 245.08
Orthorhombic, $P2_12_12_1$
a = 5.0150 (5) Å
b = 11.4093 (10) Å
c = 16.1736 (14) Å
V = 925.42 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.41 mm^{-1}
T = 173 (2) K
0.32 × 0.27 × 0.23 mm

Data collection

Stoe IPDSII two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.263, T_{max} = 0.380
7646 measured reflections
2135 independent reflections
2008 reflections with $I > 2\sigma(I)$
R_{int} = 0.070

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.116
S = 1.03
2135 reflections
129 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.95 e Å^{-3}
$\Delta\rho_{min}$ = −0.94 e Å^{-3}
Absolute structure: Flack (1983), 862 Friedel pairs
Flack parameter: −0.014 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.91 (6)	1.95 (6)	2.783 (5)	152 (5)
N2—H2A···O2^{ii}	0.81 (6)	2.61 (6)	3.324 (5)	148 (6)
N2—H2B···O2^{iii}	0.92 (6)	2.29 (6)	3.156 (5)	156 (5)

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+\frac{3}{2}, -y+1, z+\frac{1}{2}$.

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: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

BKS and KM thank the Management of PA College of Engineering for the provision of research facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2037).

References

Bhat, T. N., Singh, T. P. & Vijayan, M. (1974). *Acta Cryst.* B**30**, 2921–2922.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Cajocorius, J., Cojocarius, Z. & Niester, C. (1977). *Rev. Chim.* **28**, 15–18.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Katrusiak, A. (1993). *Acta Cryst.* C**49**, 36–39.
Liu, F., Stephen, A. G., Adainson, C. S., Gousset, K., Aman, M. J., Freed, E. O., Fisher, R. J. & Burke, T. R. Jr (2006). *Org. Lett.* **8**, 5165–5168.
Muir, K. W. & Morris, D. G. (2003). *Acta Cryst.* E**59**, o490–o492.
Narayana, B., Ashalatha, B. V., Vijayaraj, K. K., Fernandes, J. & Sarojini, B. K. (2005). *Bioorg. Med. Chem.* **13**, 4638–4644.
Narayana, B., Vijayaraj, K. K., Ashalatha, B. V. & Suchetha Kumari, N. (2005). *Pharmazie*, **338**, 373–377.
Sarojini, B. K., Yathirajan, H. S., Sunil, K., Narayana, B. & Bolte, M. (2007). *Acta Cryst.* E**63**, o3487.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.
Swain, A. P. (1959). US Patent 2 883 391.

supplementary materials

Acta Cryst. (2007). E**63**, o4419 [**doi:10.1107/S1600536807051367**]

2-Bromo-5-methoxybenzohydrazide

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

Comment

Hydrazides are useful precursors in the synthesis of several heterocyclic systems (Narayana, Vijayaraj *et al.*, 2005; Narayana, Ashalatha *et al.*, 2005). Some substituted hydrazides are reported to exhibit carcinostatic activity (Cajocorius *et al.*, 1977) against several types of tumors and also possess antimicrobial activity (Swain, 1959 & Liu *et al.*, 2006). They are also used as intermediates in many pharmaceutically important compounds. Structures of maleic hydrazide (Katrusiak, 1993), isonicotinic acid hydrazide (Bhat *et al.*, 1974) and *N*,*N*-dimethyl-*N*'-(*o*-fluorobenzoyl)hydrazide (Muir & Morris, 2003) have been reported. Recently the structure of 2-bromo-*N*'-isopropylidene-5-methoxybenzohydrazide was reported (Sarojini *et al.*, 2007). In view of the importance of hydrazides, a new hydrazide, (I), $C_8H_9BrN_2O_2$, has been synthesized and its crystal structure is reported.

Geometric parameters of the title compound are in the usual ranges. The amide bond is in a *cis* conformation. The N—N—C=O moiety is planar [torsion angle N2—N1—C7—O1 0.6 (6)°] and forms a dihedral angle of 46.4 (2)° with the aromatic ring. The crystal packing is stabilized by two strong and one rather weak N—H···O hydrogen bonds.

Experimental

A mixture of ethyl 2-bromo-5-methoxybenzoate (25.9 g, 0.1 mol) and 5.5 ml of hydrazine hydrate in 100 ml of ethanol was refluxed over water bath for 4 h. The precipitate formed was filtered and recrystallized from a mixture (8:2) of ethanol and toluene (m.p.:433–435 K). Analysis for $C_8H_9BrN_2O_2$: Found (Calculated): C: 39.14 (39.21); H: 3.66 (3.70); N: 11.39% (11.43%).

Refinement

H atoms were refined with fixed individual displacement parameters [U(H) = 1.2 U_{eq}(C,*N*) or U(H) = 1.5 U_{eq}(C_{methyl})] using a riding model with C—H(aromatic) = 0.95Å or *C*—*H*(methyl) = 0.98 Å, respectively. The methyl group was allowed to rotate but not to tip. The coordinates of the amino H atoms were refined.

Figures

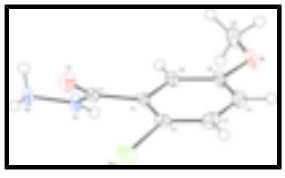


Fig. 1. Perspective view of the title compound with the atom numbering; displacement ellipsoids are plotted at the 50% probability level.

2-Bromo-5-methoxybenzohydrazide

Crystal data

$C_8H_9BrN_2O_2$	F_{000} = 488
M_r = 245.08	D_x = 1.759 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 6028 reflections
a = 5.0150 (5) Å	θ = 3.7–27.1º
b = 11.4093 (10) Å	μ = 4.41 mm^{-1}
c = 16.1736 (14) Å	T = 173 (2) K
V = 925.42 (15) Å^3	Block, colourless
Z = 4	0.32 × 0.27 × 0.23 mm

Data collection

Stoe IPDSII two-circle diffractometer	2135 independent reflections
Radiation source: fine-focus sealed tube	2008 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.070
T = 173(2) K	θ_{max} = 27.6º
ω scans	θ_{min} = 3.6º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	h = −6→5
T_{min} = 0.263, T_{max} = 0.380	k = −14→14
7646 measured reflections	l = −21→21

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0796P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.046	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.116	$\Delta\rho_{max}$ = 0.95 e Å^{-3}
S = 1.04	$\Delta\rho_{min}$ = −0.94 e Å^{-3}
2135 reflections	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
129 parameters	Extinction coefficient: 0.025 (4)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 862 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: −0.014 (17)
Hydrogen site location: inferred from neighbouring sites	

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	1.19431 (9)	0.27433 (4)	0.41643 (3)	0.02729 (19)
O1	1.0833 (6)	0.5462 (3)	0.4178 (2)	0.0235 (6)
O2	0.4447 (7)	0.3871 (3)	0.13391 (18)	0.0249 (7)
N1	0.6358 (7)	0.5633 (3)	0.4286 (2)	0.0173 (7)
H1	0.471 (12)	0.537 (5)	0.414 (4)	0.021*
N2	0.6506 (8)	0.6559 (3)	0.4876 (2)	0.0207 (7)
H2A	0.694 (13)	0.714 (5)	0.462 (4)	0.025*
H2B	0.799 (13)	0.634 (5)	0.518 (3)	0.025*
C1	0.8142 (9)	0.4236 (3)	0.3314 (2)	0.0163 (7)
C2	0.9609 (9)	0.3205 (4)	0.3285 (3)	0.0211 (8)
C3	0.9408 (10)	0.2434 (4)	0.2620 (3)	0.0250 (9)
H3	1.0455	0.1740	0.2604	0.030*
C4	0.7651 (9)	0.2693 (4)	0.1979 (3)	0.0240 (9)
H4	0.7510	0.2179	0.1519	0.029*
C5	0.6095 (9)	0.3707 (4)	0.2009 (2)	0.0196 (8)
C6	0.6336 (8)	0.4478 (4)	0.2672 (2)	0.0168 (8)
H6	0.5279	0.5168	0.2690	0.020*
C7	0.8561 (9)	0.5152 (4)	0.3976 (2)	0.0165 (8)
C8	0.2815 (11)	0.4903 (4)	0.1332 (3)	0.0266 (10)
H8A	0.1511	0.4860	0.1782	0.040*
H8B	0.1878	0.4958	0.0801	0.040*
H8C	0.3941	0.5597	0.1407	0.040*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0261 (3)	0.0310 (3)	0.0248 (2)	0.00662 (19)	−0.00015 (18)	0.00917 (16)
O1	0.0107 (13)	0.0321 (15)	0.0278 (15)	−0.0018 (12)	0.0003 (14)	−0.0065 (14)
O2	0.0269 (18)	0.0347 (17)	0.0130 (13)	0.0016 (15)	−0.0039 (12)	−0.0064 (12)
N1	0.0085 (17)	0.0267 (17)	0.0167 (15)	−0.0009 (13)	−0.0030 (12)	−0.0070 (13)
N2	0.0200 (19)	0.0257 (18)	0.0163 (15)	−0.0004 (15)	−0.0033 (14)	−0.0070 (13)

C1	0.0131 (18)	0.0242 (18)	0.0114 (15)	−0.0030 (17)	0.0014 (16)	0.0010 (13)
C2	0.022 (2)	0.0241 (19)	0.0177 (17)	−0.0023 (17)	0.0042 (16)	0.0059 (16)
C3	0.028 (2)	0.0171 (19)	0.030 (2)	0.0057 (16)	0.0073 (19)	0.0016 (16)
C4	0.025 (2)	0.0252 (19)	0.0213 (18)	−0.0005 (18)	0.0056 (16)	−0.0063 (16)
C5	0.017 (2)	0.028 (2)	0.0132 (16)	−0.0017 (17)	0.0026 (14)	−0.0052 (15)
C6	0.014 (2)	0.0214 (18)	0.0152 (17)	0.0000 (14)	0.0047 (14)	−0.0030 (14)
C7	0.015 (2)	0.0218 (18)	0.0132 (17)	0.0011 (15)	0.0011 (14)	0.0016 (13)
C8	0.025 (2)	0.034 (2)	0.0202 (18)	0.002 (2)	−0.0069 (18)	−0.0035 (16)

Geometric parameters (Å, °)

Br1—C2	1.916 (4)	C1—C7	1.511 (5)
O1—C7	1.237 (6)	C2—C3	1.393 (6)
O2—C5	1.376 (5)	C3—C4	1.393 (6)
O2—C8	1.434 (6)	C3—H3	0.9500
N1—C7	1.332 (5)	C4—C5	1.396 (6)
N1—N2	1.426 (5)	C4—H4	0.9500
N1—H1	0.91 (6)	C5—C6	1.391 (5)
N2—H2A	0.81 (6)	C6—H6	0.9500
N2—H2B	0.92 (6)	C8—H8A	0.9800
C1—C2	1.388 (6)	C8—H8B	0.9800
C1—C6	1.405 (6)	C8—H8C	0.9800
C5—O2—C8	117.5 (3)	C3—C4—H4	119.9
C7—N1—N2	121.0 (3)	C5—C4—H4	119.9
C7—N1—H1	121 (4)	O2—C5—C6	125.0 (4)
N2—N1—H1	118 (4)	O2—C5—C4	114.9 (4)
N1—N2—H2A	107 (4)	C6—C5—C4	120.1 (4)
N1—N2—H2B	101 (3)	C5—C6—C1	120.1 (4)
H2A—N2—H2B	106 (6)	C5—C6—H6	120.0
C2—C1—C6	118.9 (4)	C1—C6—H6	120.0
C2—C1—C7	122.5 (4)	O1—C7—N1	123.1 (4)
C6—C1—C7	118.5 (4)	O1—C7—C1	120.9 (4)
C1—C2—C3	121.6 (4)	N1—C7—C1	115.9 (4)
C1—C2—Br1	122.1 (3)	O2—C8—H8A	109.5
C3—C2—Br1	116.3 (3)	O2—C8—H8B	109.5
C2—C3—C4	119.1 (4)	H8A—C8—H8B	109.5
C2—C3—H3	120.5	O2—C8—H8C	109.5
C4—C3—H3	120.5	H8A—C8—H8C	109.5
C3—C4—C5	120.2 (4)	H8B—C8—H8C	109.5
C6—C1—C2—C3	2.7 (6)	O2—C5—C6—C1	−178.2 (4)
C7—C1—C2—C3	−172.7 (4)	C4—C5—C6—C1	−0.2 (6)
C6—C1—C2—Br1	−175.7 (3)	C2—C1—C6—C5	−1.9 (6)
C7—C1—C2—Br1	9.0 (6)	C7—C1—C6—C5	173.7 (4)
C1—C2—C3—C4	−1.4 (7)	N2—N1—C7—O1	0.6 (6)
Br1—C2—C3—C4	177.1 (3)	N2—N1—C7—C1	−176.0 (3)
C2—C3—C4—C5	−0.7 (7)	C2—C1—C7—O1	45.0 (6)
C8—O2—C5—C6	−1.4 (6)	C6—C1—C7—O1	−130.4 (4)
C8—O2—C5—C4	−179.5 (4)	C2—C1—C7—N1	−138.3 (4)
C3—C4—C5—O2	179.7 (4)	C6—C1—C7—N1	46.3 (5)

C3—C4—C5—C6	1.5 (7)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.91 (6)	1.95 (6)	2.783 (5)	152 (5)
N2—H2A···O2^{ii}	0.81 (6)	2.61 (6)	3.324 (5)	148 (6)
N2—H2B···O2^{iii}	0.92 (6)	2.29 (6)	3.156 (5)	156 (5)

Symmetry codes: (i) $x-1$, y, z; (ii) $-x+1$, $y+1/2$, $-z+1/2$; (iii) $-x+3/2$, $-y+1$, $z+1/2$.

Fig. 1