N′-(4-Bromobenzylidene)propano-hydrazide

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The title compound, $C_{10}H_{11}BrN_2O$, was prepared by the reaction of *p*-bromobenzaldehyde and propionylhydrazine. Centrosymmetric dimers are formed through N—H···O hydrogen bonds.

Related literature

For related literature, see: Allen *et al.* (1987); Qiu *et al.* (2006).
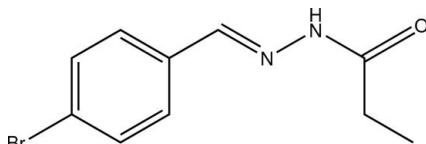
Experimental

Crystal data

$C_{10}H_{11}BrN_2O$
$M_r = 255.12$
Monoclinic, $P2_1/n$
$a = 4.7749 (10)$ Å
$b = 10.253 (2)$ Å
$c = 22.183 (4)$ Å
$\beta = 95.514 (4)°$

$V = 1081.0 (4)$ Å³
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 3.77$ mm^{-1}
$T = 294 (2)$ K
$0.28 \times 0.16 \times 0.14$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{min} = 0.418$, $T_{max} = 0.620$

5443 measured reflections
1906 independent reflections
1363 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.087$
$S = 1.00$
1906 reflections
129 parameters
6 restraints

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1[i]	0.86	2.09	2.951 (4)	174

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2094).

References

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supplementary materials

N'-(4-Bromobenzylidene)propanohydrazide

H.-M. Guo

Comment

As an extension of our work on the structural characterization of Schiff base compound, (I), is reported here. The title compound $C_{10}H_{11}BrN_2O$, is roughly planar. In (I), the bond lengths are within normal ranges (Allen *et al.*, 1987) (Fig.1). C7=N1 bond length of 1.279 (4) Å conforms to the value for a double bond and similar to the distance reported by Qiu *et al.*, 2006. The bond length of 1.346 (4) Å is greater than the value for a double bond, and less than the value for a single bond, because of conjugation effects in the molecule. The crystal structure is stabilized by intermolecular N—H—O hydrogen bonds (Table 1).

Scheme I

Experimental

A mixture of the *p*-bromobenzaldehyde(0.1 mol, 18,502 g,), and propionylhydrazine (0.1 mol, 8.810 g) was stirred in refluxing ethanol (30 ml) for 5 h to afford the title compound (0.087 mol, yield 81%). Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances=0.93–0.97 Å, N—H distances=0.8600 Å and with $U_{iso}=1.2–1.5U_{eq}$.

Figures
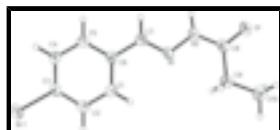
Fig. 1. The structure of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme.

N'-(4-Bromobenzylidene)propanohydrazide

Crystal data

$C_{10}H_{11}BrN_2O$	$Z = 4$
$M_r = 255.12$	$F_{000} = 512$
Monoclinic, $P2_1/n$	$D_x = 1.568$ Mg m^{-3}
Hall symbol: -P 2yn	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 4.7749$ (10) Å	$\theta = 1.8–25.0°$

$b = 10.253$ (2) Å

$c = 22.183$ (4) Å

$\beta = 95.514$ (4)°

$V = 1081.0$ (4) Å^3

$\mu = 3.77$ mm^{-1}

$T = 294$ (2) K

Block, colourless

$0.28 \times 0.16 \times 0.14$ mm

Data collection

CCD area-detector diffractometer	1906 independent reflections
Radiation source: fine-focus sealed tube	1363 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.035$
$T = 294(2)$ K	$\theta_{max} = 25.0°$
phi and ω scans	$\theta_{min} = 1.8°$
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -5 \rightarrow 5$
$T_{min} = 0.418$, $T_{max} = 0.620$	$k = -12 \rightarrow 9$
5443 measured reflections	$l = -23 \rightarrow 26$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.032$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.087$	$w = 1/[\sigma^2(F_o^2) + (0.0412P)^2 + 0.4567P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} < 0.001$
1906 reflections	$\Delta\rho_{max} = 0.41$ e Å^{-3}
129 parameters	$\Delta\rho_{min} = -0.34$ e Å^{-3}
6 restraints	Extinction correction: SHELXL, $Fc^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0194 (15)

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$^2 > 2$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	0.77061 (8)	0.21453 (4)	0.196101 (18)	0.0605 (2)
N1	−0.0531 (6)	0.1549 (3)	0.43391 (13)	0.0441 (7)
O1	−0.4993 (5)	0.1484 (3)	0.54910 (11)	0.0573 (7)
N2	−0.2420 (6)	0.1110 (3)	0.47212 (13)	0.0471 (8)
H2	−0.3080	0.0331	0.4679	0.057*
C1	0.3146 (7)	0.2337 (3)	0.34522 (16)	0.0465 (9)
H1	0.2713	0.2969	0.3729	0.056*
C2	0.4859 (8)	0.2653 (4)	0.30103 (17)	0.0477 (9)
H2A	0.5590	0.3491	0.2989	0.057*
C3	0.5480 (7)	0.1711 (4)	0.25989 (15)	0.0429 (9)
C4	0.4499 (7)	0.0465 (4)	0.26351 (17)	0.0500 (9)
H4	0.4975	−0.0166	0.2362	0.060*
C5	0.2792 (7)	0.0152 (4)	0.30818 (16)	0.0501 (10)
H5	0.2130	−0.0697	0.3109	0.060*
C6	0.2049 (7)	0.1087 (3)	0.34909 (14)	0.0396 (8)
C7	0.0139 (7)	0.0733 (3)	0.39412 (15)	0.0427 (8)
H7	−0.0606	−0.0105	0.3938	0.051*
C8	−0.3254 (7)	0.1882 (3)	0.51608 (16)	0.0446 (9)
C9	−0.1936 (9)	0.3205 (4)	0.5226 (2)	0.0647 (12)
H9A	−0.2210	0.3649	0.4839	0.078*
H9B	0.0074	0.3101	0.5327	0.078*
C10	−0.3091 (10)	0.4053 (4)	0.5702 (2)	0.0781 (14)
H10A	−0.2727	0.3648	0.6092	0.117*
H10B	−0.5083	0.4158	0.5608	0.117*
H10C	−0.2195	0.4892	0.5708	0.117*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0552 (3)	0.0728 (4)	0.0571 (3)	−0.0027 (2)	0.02439 (19)	0.0049 (2)
N1	0.0437 (16)	0.0492 (18)	0.0415 (17)	−0.0020 (14)	0.0153 (14)	0.0055 (14)
O1	0.0602 (16)	0.0590 (17)	0.0574 (16)	−0.0039 (14)	0.0288 (14)	0.0030 (13)
N2	0.0523 (17)	0.0448 (19)	0.0473 (17)	−0.0056 (14)	0.0199 (15)	0.0033 (14)
C1	0.052 (2)	0.042 (2)	0.046 (2)	0.0005 (17)	0.0097 (17)	−0.0070 (17)
C2	0.053 (2)	0.039 (2)	0.053 (2)	−0.0070 (17)	0.0108 (18)	−0.0007 (17)
C3	0.0373 (18)	0.051 (2)	0.041 (2)	0.0030 (16)	0.0108 (16)	0.0058 (16)
C4	0.056 (2)	0.041 (2)	0.056 (2)	0.0054 (18)	0.0200 (18)	−0.0042 (18)
C5	0.053 (2)	0.038 (2)	0.062 (2)	−0.0045 (17)	0.0194 (19)	−0.0004 (18)
C6	0.0382 (18)	0.042 (2)	0.0391 (19)	0.0023 (16)	0.0053 (15)	0.0014 (16)
C7	0.0431 (19)	0.041 (2)	0.045 (2)	−0.0002 (16)	0.0081 (16)	0.0037 (16)
C8	0.045 (2)	0.047 (2)	0.043 (2)	0.0059 (16)	0.0118 (17)	0.0063 (17)
C9	0.074 (3)	0.053 (3)	0.072 (3)	−0.006 (2)	0.033 (2)	−0.007 (2)
C10	0.083 (3)	0.069 (3)	0.087 (3)	−0.006 (2)	0.030 (3)	−0.019 (2)

Geometric parameters (Å, °)

Br1—C3	1.903 (3)	C4—H4	0.9300
N1—C7	1.279 (4)	C5—C6	1.389 (5)
N1—N2	1.372 (3)	C5—H5	0.9300
O1—C8	1.228 (4)	C6—C7	1.462 (4)
N2—C8	1.346 (4)	C7—H7	0.9300
N2—H2	0.8600	C8—C9	1.497 (5)
C1—C2	1.374 (5)	C9—C10	1.513 (5)
C1—C6	1.391 (5)	C9—H9A	0.9700
C1—H1	0.9300	C9—H9B	0.9700
C2—C3	1.381 (5)	C10—H10A	0.9600
C2—H2A	0.9300	C10—H10B	0.9600
C3—C4	1.366 (5)	C10—H10C	0.9600
C4—C5	1.380 (4)		
C7—N1—N2	115.8 (3)	C5—C6—C7	119.4 (3)
C8—N2—N1	120.8 (3)	C1—C6—C7	122.3 (3)
C8—N2—H2	119.6	N1—C7—C6	121.6 (3)
N1—N2—H2	119.6	N1—C7—H7	119.2
C2—C1—C6	121.0 (3)	C6—C7—H7	119.2
C2—C1—H1	119.5	O1—C8—N2	120.1 (3)
C6—C1—H1	119.5	O1—C8—C9	123.0 (3)
C1—C2—C3	119.1 (3)	N2—C8—C9	116.9 (3)
C1—C2—H2A	120.4	C8—C9—C10	114.3 (3)
C3—C2—H2A	120.4	C8—C9—H9A	108.7
C4—C3—C2	121.3 (3)	C10—C9—H9A	108.7
C4—C3—Br1	118.8 (3)	C8—C9—H9B	108.7
C2—C3—Br1	119.9 (3)	C10—C9—H9B	108.7
C3—C4—C5	119.3 (3)	H9A—C9—H9B	107.6
C3—C4—H4	120.4	C9—C10—H10A	109.5
C5—C4—H4	120.4	C9—C10—H10B	109.5
C4—C5—C6	121.0 (3)	H10A—C10—H10B	109.5
C4—C5—H5	119.5	C9—C10—H10C	109.5
C6—C5—H5	119.5	H10A—C10—H10C	109.5
C5—C6—C1	118.3 (3)	H10B—C10—H10C	109.5
C7—N1—N2—C8	179.4 (3)	C2—C1—C6—C5	1.8 (5)
C6—C1—C2—C3	0.3 (5)	C2—C1—C6—C7	−177.7 (3)
C1—C2—C3—C4	−2.1 (5)	N2—N1—C7—C6	177.7 (3)
C1—C2—C3—Br1	177.2 (3)	C5—C6—C7—N1	177.7 (3)
C2—C3—C4—C5	1.8 (6)	C1—C6—C7—N1	−2.8 (5)
Br1—C3—C4—C5	−177.5 (3)	N1—N2—C8—O1	178.4 (3)
C3—C4—C5—C6	0.4 (5)	N1—N2—C8—C9	−2.5 (5)
C4—C5—C6—C1	−2.1 (5)	O1—C8—C9—C10	−4.5 (6)
C4—C5—C6—C7	177.4 (3)	N2—C8—C9—C10	176.5 (4)

Hydrogen-bond geometry (Å, °)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*

| N2—H2···O1[i] | 0.86 | 2.09 | 2.951 (4) | 174 |

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

Fig. 1