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2-Bromo-N-(4-bromophenyl)acetamide

B. Thimme Gowda,^{a*} Ingrid Svoboda,^b Sabine Foro,^b
P. A. Suchetan^a and Hartmut Fuess^b^aDepartment of Chemistry, Mangalore University, Mangalagangothri 574 199, Mangalore, India, and ^bInstitute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany

Correspondence e-mail: gowdabt@yahoo.com

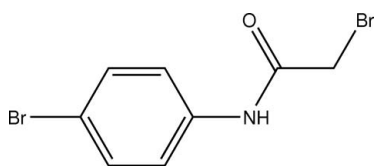
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Key indicators: single-crystal X-ray study; $T = 303$ K; mean $\sigma(\text{C}-\text{C}) = 0.009$ Å; R factor = 0.049; wR factor = 0.127; data-to-parameter ratio = 15.2.

In the title compound, $\text{C}_8\text{H}_7\text{Br}_2\text{NO}$, the conformation of the N—H bond is *anti* to both the carbonyl and C—Br bonds in the side chain. In the crystal structure, molecules are packed into supramolecular chains along the c axis by N—H \cdots O hydrogen bonds.

Related literature

For the preparation of the title compound, see: Gowda *et al.* (2003). For related structures, see: Andreotti *et al.* (1968); Gowda *et al.* (2007a,b,c).



Experimental

Crystal data

$\text{C}_8\text{H}_7\text{Br}_2\text{NO}$
 $M_r = 292.97$
 Monoclinic, $P2_1/n$
 $a = 4.4987$ (3) Å

$b = 23.152$ (1) Å
 $c = 9.1098$ (5) Å
 $\beta = 99.713$ (6)°
 $V = 935.22$ (9) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 8.62$ mm⁻¹

$T = 303$ K
 $0.50 \times 0.20 \times 0.14$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford)

Diffraction, 2009)
 $T_{\min} = 0.078$, $T_{\max} = 0.299$
 3065 measured reflections
 1661 independent reflections
 1415 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.127$
 $S = 0.99$
 1661 reflections

109 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.73$ e Å⁻³
 $\Delta\rho_{\min} = -0.68$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	0.86	2.11	2.925 (6)	157

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); 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: TK2505).

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supplementary materials

Acta Cryst. (2009). E65, o1955 [doi:10.1107/S1600536809028219]

2-Bromo-*N*-(4-bromophenyl)acetamide

B. T. Gowda, I. Svoboda, S. Foro, P. A. Suchetan and H. Fuess

Comment

As part of a study of the effect of the ring and the side chain substituents on the structures of *N*-aromatic amides (Gowda *et al.*, 2007*a, b, c*), in the present work, the structure of 2-bromo-*N*-(4-bromophenyl)acetamide (I) has been determined (Fig. 1). The conformation of the N—H bond is *anti* to both the C=O and the C—Br bonds in the side chain, similar to that observed in 2-chloro-*N*-(4-chlorophenyl)acetamide (Gowda *et al.*, 2007*c*), *N*-(4-bromophenyl)acetamide (Andreetti *et al.*, 1968), and other amides (Gowda *et al.*, 2007*a, b*).

The crystal packing shows N1—H1N···O1 hydrogen bonds (Table 1) that lead to the formation of molecular chain along the *c*-axis (Fig. 2).

Experimental

Compound (I) was prepared from 4-bromoaniline and bromoacetylchloride according to the literature method (Gowda *et al.*, 2003). Single crystals were obtained by slow evaporation of an ethanolic solution of (I) held at room temperature.

Refinement

The H atoms were positioned with idealized geometry using a riding model [N—H = 0.86 Å, C—H = 0.93—0.97 Å], and were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

Figures

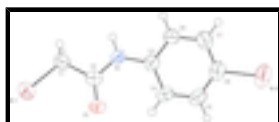


Fig. 1. Molecular structure of (I), showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented as small spheres of arbitrary radii.

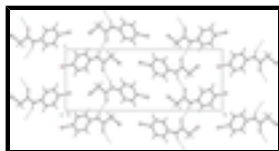


Fig. 2. Molecular packing of (I) with hydrogen bonds shown as dashed lines.

2-Bromo-*N*-(4-bromophenyl)acetamide

Crystal data

C₈H₇Br₂NO

$M_r = 292.97$

Monoclinic, $P2_1/n$

$F_{000} = 560$

$D_x = 2.081 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

supplementary materials

Hall symbol: -P 2₁n

$a = 4.4987$ (3) Å

$b = 23.152$ (1) Å

$c = 9.1098$ (5) Å

$\beta = 99.713$ (6)°

$V = 935.22$ (9) Å³

$Z = 4$

Cell parameters from 2250 reflections

$\theta = 2.9$ – 27.8 °

$\mu = 8.62$ mm⁻¹

$T = 303$ K

Needle, colourless

$0.50 \times 0.20 \times 0.14$ mm

Data collection

Oxford Diffraction Xcalibur
diffractometer with a Sapphire CCD detector

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 303$ K

Rotation method data acquisition using ω and φ scans $\theta_{\min} = 2.9$ °

Absorption correction: multi-scan
(CrysAlis RED; Oxford Diffraction, 2009)

$T_{\min} = 0.078$, $T_{\max} = 0.299$

3065 measured reflections

1661 independent reflections

1415 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\max} = 25.4$ °

$h = -5 \rightarrow 4$

$k = -16 \rightarrow 27$

$l = -10 \rightarrow 9$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.049$

$wR(F^2) = 0.127$

$S = 0.99$

1661 reflections

109 parameters

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 4.1384P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.73$ e Å⁻³

$\Delta\rho_{\min} = -0.68$ e Å⁻³

Extinction correction: none

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.7460 (14)	0.2109 (3)	0.3871 (6)	0.0489 (15)
H1A	0.6432	0.2287	0.4610	0.059*
H1B	0.9218	0.1909	0.4395	0.059*
C2	0.8475 (13)	0.2577 (3)	0.2905 (6)	0.0400 (13)
C3	1.1568 (13)	0.3474 (2)	0.3295 (6)	0.0388 (12)
C4	1.0573 (14)	0.3743 (3)	0.1927 (6)	0.0445 (14)
H4	0.9070	0.3573	0.1236	0.053*
C5	1.1831 (16)	0.4263 (3)	0.1603 (7)	0.0535 (16)
H5	1.1158	0.4444	0.0697	0.064*
C6	1.4056 (14)	0.4510 (3)	0.2609 (7)	0.0473 (15)
C7	1.5067 (15)	0.4251 (3)	0.3949 (7)	0.0538 (16)
H7	1.6576	0.4424	0.4631	0.065*
C8	1.3832 (14)	0.3731 (3)	0.4283 (7)	0.0476 (14)
H8	1.4542	0.3553	0.5189	0.057*
N1	1.0306 (11)	0.2954 (2)	0.3747 (5)	0.0397 (11)
H1N	1.0770	0.2870	0.4677	0.048*
O1	0.7725 (10)	0.26109 (19)	0.1567 (4)	0.0529 (11)
Br1	0.48101 (16)	0.15590 (3)	0.27451 (8)	0.0576 (3)
Br2	1.5669 (2)	0.52368 (3)	0.21830 (9)	0.0722 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.048 (3)	0.063 (4)	0.033 (3)	-0.015 (3)	0.000 (3)	-0.002 (3)
C2	0.038 (3)	0.044 (3)	0.038 (3)	0.007 (3)	0.007 (2)	-0.001 (3)
C3	0.044 (3)	0.037 (3)	0.037 (3)	0.004 (2)	0.012 (2)	-0.005 (2)
C4	0.053 (4)	0.050 (3)	0.029 (3)	0.004 (3)	0.004 (3)	0.003 (3)
C5	0.072 (4)	0.048 (4)	0.042 (3)	0.007 (3)	0.014 (3)	0.006 (3)
C6	0.058 (4)	0.038 (3)	0.053 (4)	-0.001 (3)	0.027 (3)	-0.003 (3)
C7	0.054 (4)	0.057 (4)	0.052 (4)	-0.012 (3)	0.012 (3)	-0.008 (3)
C8	0.048 (3)	0.052 (4)	0.041 (3)	-0.001 (3)	0.003 (3)	0.001 (3)
N1	0.051 (3)	0.039 (2)	0.028 (2)	-0.001 (2)	0.007 (2)	0.006 (2)
O1	0.072 (3)	0.054 (3)	0.030 (2)	-0.012 (2)	-0.001 (2)	-0.0005 (19)
Br1	0.0632 (5)	0.0541 (4)	0.0528 (4)	-0.0127 (3)	0.0024 (3)	-0.0073 (3)
Br2	0.1010 (7)	0.0456 (4)	0.0785 (6)	-0.0099 (4)	0.0392 (5)	-0.0006 (3)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.513 (8)	C4—H4	0.9300
C1—Br1	1.919 (6)	C5—C6	1.363 (9)
C1—H1A	0.9700	C5—H5	0.9300
C1—H1B	0.9700	C6—C7	1.367 (9)
C2—O1	1.211 (7)	C6—Br2	1.898 (6)
C2—N1	1.349 (7)	C7—C8	1.381 (9)
C3—C8	1.376 (8)	C7—H7	0.9300

supplementary materials

C3—C4	1.398 (8)	C8—H8	0.9300
C3—N1	1.421 (7)	N1—H1N	0.8600
C4—C5	1.382 (9)		
C2—C1—Br1	112.6 (4)	C6—C5—C4	120.2 (6)
C2—C1—H1A	109.1	C6—C5—H5	119.9
Br1—C1—H1A	109.1	C4—C5—H5	119.9
C2—C1—H1B	109.1	C5—C6—C7	120.8 (6)
Br1—C1—H1B	109.1	C5—C6—Br2	119.8 (5)
H1A—C1—H1B	107.8	C7—C6—Br2	119.3 (5)
O1—C2—N1	124.7 (6)	C6—C7—C8	119.6 (6)
O1—C2—C1	124.9 (5)	C6—C7—H7	120.2
N1—C2—C1	110.4 (5)	C8—C7—H7	120.2
C8—C3—C4	118.8 (6)	C3—C8—C7	120.9 (6)
C8—C3—N1	117.7 (5)	C3—C8—H8	119.6
C4—C3—N1	123.5 (5)	C7—C8—H8	119.6
C5—C4—C3	119.8 (6)	C2—N1—C3	128.3 (5)
C5—C4—H4	120.1	C2—N1—H1N	115.8
C3—C4—H4	120.1	C3—N1—H1N	115.8
Br1—C1—C2—O1	0.2 (8)	Br2—C6—C7—C8	178.0 (5)
Br1—C1—C2—N1	179.5 (4)	C4—C3—C8—C7	1.3 (9)
C8—C3—C4—C5	-1.2 (9)	N1—C3—C8—C7	-176.9 (6)
N1—C3—C4—C5	177.0 (5)	C6—C7—C8—C3	-0.9 (10)
C3—C4—C5—C6	0.6 (9)	O1—C2—N1—C3	4.1 (9)
C4—C5—C6—C7	-0.3 (10)	C1—C2—N1—C3	-175.1 (5)
C4—C5—C6—Br2	-177.8 (5)	C8—C3—N1—C2	-167.5 (6)
C5—C6—C7—C8	0.4 (10)	C4—C3—N1—C2	14.3 (9)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N \cdots O1 ⁱ	0.86	2.11	2.925 (6)	157

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

Fig. 1

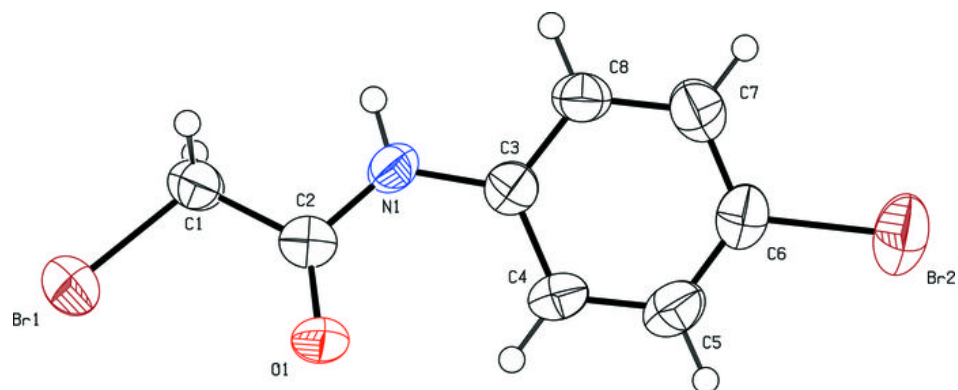


Fig. 2

