organic compounds

6-Bromo-1,3-benzothiazol-2-amine

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The r.m.s. deviation from the mean plane for the non-H atoms in the title compound, $C_7H_5BrN_2S$, is 0.011 Å. In the crystal, the molecules are linked by N—H···N and N—H···Br hydrogen bonds to generate (010) sheets. Weak aromatic π–π stacking [centroid-to-centroid separation = 3.884 (10) Å] and possible C—H···Br interactions are also observed. The crystal studied was found to be an inversion twin.

Related literature

For a related structure and background to benzothiazole derivatives, see: Jin et al. (2012).
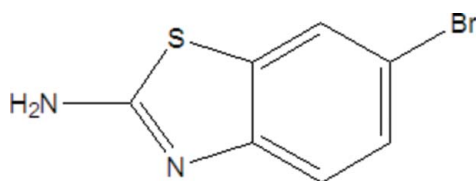
Experimental

Crystal data

$C_7H_5BrN_2S$
$M_r = 229.10$
Orthorhombic, $Pna2_1$
$a = 8.6268$ (7) Å

$b = 22.487$ (2) Å
$c = 4.0585$ (3) Å
$V = 787.30$ (11) Å^3
$Z = 4$

Mo $K\alpha$ radiation
$\mu = 5.41$ mm^{-1}
$T = 298$ K
$0.31 \times 0.25 \times 0.16$ mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2002)
$T_{min} = 0.215$, $T_{max} = 0.421$

3645 measured reflections
1340 independent reflections
929 reflections with $I > 2\sigma(I)$
$R_{int} = 0.087$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.095$
$wR(F^2) = 0.246$
$S = 1.07$
1340 reflections
100 parameters
1 restraint

H-atom parameters constrained
$\Delta\rho_{max} = 1.13$ e Å^{-3}
$\Delta\rho_{min} = -0.74$ e Å^{-3}
Absolute structure: Flack (1983), 539 Friedel pairs
Flack parameter: 0.42 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···Br1[i]	0.86	3.04	3.864 (17)	160
N2—H2A···N1[ii]	0.86	2.26	2.94 (2)	136
C4—H4···Br1[iii]	0.93	2.87	3.402 (17)	118

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

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); 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.

References

Bruker (2002). SADABS, SMART and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). Acta Cryst. A39, 876–881.
Jin, S. W., Yan, P. H., Wang, D. Q., Xu, Y. J., Jiang, Y. Y. & Hu, L. L. (2012). J. Mol. Struct. 1016, 55–63.
Sheldrick, G. M. (2008). Acta Cryst. A64, 112–122.

supporting information

6-Bromo-1,3-benzothiazol-2-amine

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S1. Comment

As an extension of our study of 2-aminoheterocyclic compounds (Jin *et al.*, 2012), herein we report the crystal structure of 6-bromobenzo[*d*]thiazol-2-amine.

The single crystals of the title compound (Fig.1) with the formula $C_7H_5BrN_2S$ was obtained by slow evaporating its methanol solution.

The 6-bromobenzo[*d*]thiazol-2-amine molecules (Fig. 1) are linked together in head to tail fashion *via* the N—H···Br association to form one-dimensional chain running along the direction that made a dihedral angle of *ca* 30° with the *a* axis direction. Two neighboring chains were held together by the CH—Br interaction with C—Br distance of 3.402 Å generating one-dimensional double chain (Fig.2). The double chains were stacked along the direction that is perpendicular with its extending direction by the CH—Br interaction with C—Br distance of 3.402 Å to form two-dimensional sheet extending parallel to the *ac* plane. The sheets were further stacked along the *b* axis direction by the intersheet N—H···N hydrogen bonds to form three-dimensional ABAB layer network structure.

S2. Experimental

The 6-bromobenzo[*d*]thiazol-2-amine (22.9 mg, 0.1 mmol) was dissolved in a methanol solution (8 ml). The solution was filtered into a test tube. The solution was left standing at room temperature for a month, light-yellow blocks were isolated after slow evaporation of the methanol solution to *ca* 3 ml in air.

S3. Refinement

H atoms bonded N atoms were located in a6-Bromo-1,3-benzothiazol-2-amine difference Fourier map and refined isotropically. Other H atoms were positioned geometrically with C—H = 0.93 Å, and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2Ueq(C)$.
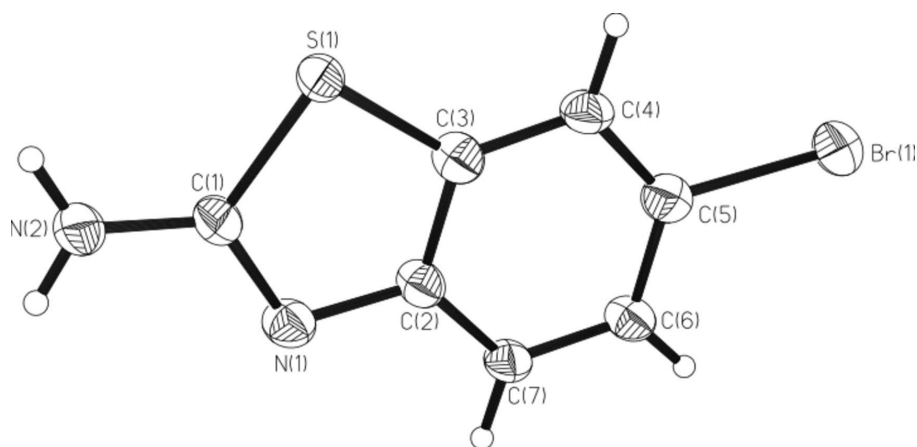
Figure 1

The structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level.
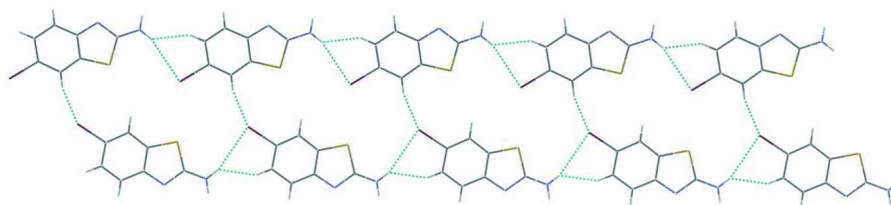

Figure 2

One-dimensional double chain structure.

6-Bromo-1,3-benzothiazol-2-amine

Crystal data

$C_7H_5BrN_2S$	$D_x = 1.933$ Mg m^{-3}
$M_r = 229.10$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Orthorhombic, $Pna2_1$	Cell parameters from 868 reflections
$a = 8.6268$ (7) Å	$\theta = 3.6$–21.4°
$b = 22.487$ (2) Å	$\mu = 5.41$ mm^{-1}
$c = 4.0585$ (3) Å	$T = 298$ K
$V = 787.30$ (11) Å^3	Block, colourless
$Z = 4$	0.31 × 0.25 × 0.16 mm
$F(000) = 448$	

Data collection

Bruker SMART CCD diffractometer	3645 measured reflections
Radiation source: fine-focus sealed tube	1340 independent reflections
Graphite monochromator	929 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.087$
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)	$\theta_{max} = 25.0°$, $\theta_{min} = 2.5°$
	$h = -6 \rightarrow 10$
$T_{min} = 0.215$, $T_{max} = 0.421$	$k = -26 \rightarrow 26$
	$l = -4 \rightarrow 4$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.095$	$w = 1/[\sigma^2(F_o^2) + (0.067P)^2 + 14.6225P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.246$	
$S = 1.07$	$(\Delta/\sigma)_{max} = 0.001$
1340 reflections	$\Delta\rho_{max} = 1.13$ e Å^{-3}
100 parameters	$\Delta\rho_{min} = -0.74$ e Å^{-3}
1 restraint	
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), **with how many Friedel pairs?**
Secondary atom site location: difference Fourier map	Absolute structure parameter: 0.42 (9)

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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
Br1	1.2451 (2)	0.69710 (7)	0.3819 (16)	0.0745 (8)
S1	0.6334 (4)	0.65747 (14)	0.8676 (16)	0.0407 (9)
N2	0.4231 (13)	0.5675 (5)	0.884 (6)	0.051 (3)
H2A	0.3952	0.5318	0.8379	0.061*
H2B	0.3608	0.5908	0.9878	0.061*
C7	0.9282 (17)	0.5595 (6)	0.404 (6)	0.044 (4)
H7	0.9250	0.5204	0.3294	0.052*
C6	1.0594 (18)	0.5939 (6)	0.352 (6)	0.043 (4)
H6	1.1456	0.5786	0.2435	0.052*
N1	0.6666 (18)	0.5508 (6)	0.627 (4)	0.047 (4)
C1	0.5630 (19)	0.5869 (7)	0.795 (4)	0.046 (5)
C2	0.802 (2)	0.5836 (7)	0.568 (4)	0.042 (4)
C4	0.933 (2)	0.6779 (7)	0.634 (5)	0.046 (4)
H4	0.9364	0.7170	0.7084	0.055*
C3	0.804 (2)	0.6421 (8)	0.682 (4)	0.045 (4)
C5	1.057 (2)	0.6520 (7)	0.469 (4)	0.052 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0513 (10)	0.0510 (9)	0.1212 (18)	−0.0117 (8)	0.005 (2)	0.005 (2)
S1	0.0394 (18)	0.0344 (16)	0.048 (2)	−0.0010 (15)	0.006 (3)	0.015 (3)
N2	0.041 (7)	0.041 (6)	0.071 (10)	−0.005 (5)	−0.012 (15)	−0.004 (11)

C7	0.045 (8)	0.033 (7)	0.053 (11)	0.000 (6)	0.007 (13)	−0.012 (11)
C6	0.052 (9)	0.038 (7)	0.040 (10)	−0.002 (6)	0.014 (14)	−0.007 (11)
N1	0.054 (9)	0.040 (7)	0.047 (9)	−0.001 (7)	0.010 (8)	−0.006 (7)
C1	0.043 (9)	0.037 (8)	0.059 (15)	−0.007 (7)	0.000 (9)	−0.009 (8)
C2	0.047 (10)	0.038 (9)	0.041 (10)	−0.005 (8)	0.011 (8)	−0.004 (8)
C4	0.052 (11)	0.035 (8)	0.051 (11)	0.002 (8)	0.005 (9)	−0.008 (8)
C3	0.050 (11)	0.044 (9)	0.040 (10)	−0.004 (8)	0.005 (8)	−0.001 (8)
C5	0.051 (10)	0.044 (9)	0.060 (16)	−0.001 (8)	0.004 (9)	0.002 (8)

Geometric parameters (Å, º)

Br1—C5	1.944 (18)	C6—C5	1.39 (2)
S1—C3	1.686 (19)	C6—H6	0.9300
S1—C1	1.725 (16)	N1—C1	1.39 (2)
N2—C1	1.33 (2)	N1—C2	1.40 (2)
N2—H2A	0.8600	C2—C3	1.39 (2)
N2—H2B	0.8600	C4—C3	1.39 (3)
C7—C6	1.39 (2)	C4—C5	1.39 (2)
C7—C2	1.39 (2)	C4—H4	0.9300
C7—H7	0.9300		
C3—S1—C1	92.4 (9)	N1—C1—S1	113.3 (12)
C1—N2—H2A	120.0	C7—C2—C3	121.1 (16)
C1—N2—H2B	120.0	C7—C2—N1	122.0 (15)
H2A—N2—H2B	120.0	C3—C2—N1	116.9 (16)
C6—C7—C2	119.7 (14)	C3—C4—C5	116.3 (15)
C6—C7—H7	120.1	C3—C4—H4	121.8
C2—C7—H7	120.1	C5—C4—H4	121.8
C7—C6—C5	117.5 (15)	C4—C3—C2	120.7 (17)
C7—C6—H6	121.2	C4—C3—S1	130.0 (14)
C5—C6—H6	121.2	C2—C3—S1	109.2 (14)
C1—N1—C2	108.1 (14)	C6—C5—C4	124.6 (16)
N2—C1—N1	121.7 (14)	C6—C5—Br1	114.7 (13)
N2—C1—S1	125.0 (13)	C4—C5—Br1	120.7 (12)
C2—C7—C6—C5	0 (3)	C7—C2—C3—C4	0 (3)
C2—N1—C1—N2	179.8 (19)	N1—C2—C3—C4	−180.0 (17)
C2—N1—C1—S1	0.3 (18)	C7—C2—C3—S1	179.8 (17)
C3—S1—C1—N2	−179.9 (18)	N1—C2—C3—S1	0 (2)
C3—S1—C1—N1	−0.4 (15)	C1—S1—C3—C4	−180 (2)
C6—C7—C2—C3	0 (3)	C1—S1—C3—C2	0.4 (15)
C6—C7—C2—N1	−179.9 (19)	C7—C6—C5—C4	0 (3)
C1—N1—C2—C7	180 (2)	C7—C6—C5—Br1	−179.5 (15)
C1—N1—C2—C3	0 (2)	C3—C4—C5—C6	0 (3)
C5—C4—C3—C2	0 (3)	C3—C4—C5—Br1	179.6 (14)
C5—C4—C3—S1	−179.7 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···Br1[i]	0.86	3.04	3.864 (17)	160
N2—H2*A*···N1[ii]	0.86	2.26	2.94 (2)	136
C4—H4···Br1[iii]	0.93	2.87	3.402 (17)	118

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