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1-(5-Chloro-6-fluoro-1,3-benzothiazol-2-yl)hydrazine

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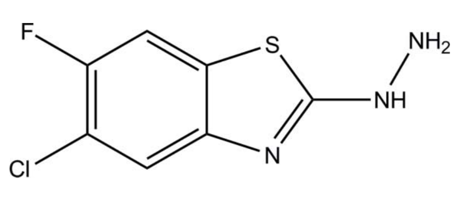
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.001 Å; R factor = 0.024; wR factor = 0.066; data-to-parameter ratio = 21.0.

In the title compound, $C_7H_5ClFN_3S$, the 1,3-benzothiazole ring system is nearly planar (r.m.s. deviation = 0.023 Å). In the crystal, molecules are linked *via* intermolecular N—H···N hydrogen bonds into a two-dimensional network parallel to (100).

Related literature

For general background to and the biological activities of benzothiazole derivatives, see: Yaseen *et al.* (2006); Kini *et al.* (2007); Munirajasekhar *et al.* (2011); Gurupadayya *et al.* (2008); Bowyer *et al.* (2007); Mittal *et al.* (2007); Pozas *et al.* (2005); Rana *et al.* (2008). For a related structure, see: Fun *et al.* (2012). For standard bond-length data, see: Allen *et al.* (1987). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_7H_5ClFN_3S$
M_r = 217.65
Monoclinic, $P2_1/c$
a = 11.1287 (6) Å
b = 5.6641 (3) Å
c = 13.3419 (7) Å
β = 108.552 (1)°
V = 797.29 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.70 mm^{-1}
T = 100 K
0.31 × 0.16 × 0.14 mm

Data collection

Bruker SMART APEXII DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.813, T_{max} = 0.908
9459 measured reflections
2899 independent reflections
2638 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.066
S = 1.07
2899 reflections
138 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1N2···N1^{i}	0.816 (16)	2.132 (16)	2.9478 (12)	176.9 (16)
N3—H2N3···N3^{ii}	0.850 (16)	2.443 (17)	3.1382 (12)	139.5 (14)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5255).

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§ Thomson Reuters ResearcherID: A-5525-2009.

supporting information

Acta Cryst. (2012). E**68**, o2459 [https://doi.org/10.1107/S160053681203156X]

1-(5-Chloro-6-fluoro-1,3-benzothiazol-2-yl)hydrazine

Hoong-Kun Fun, Ching Kheng Quah, B. K. Sarojini, B. J. Mohan and B. Narayana

S1. Comment

Benzothiazoles are very important bicyclic ring compounds which are of great interest because of their biological activities. The substituted benzothiazole derivatives have emerged as significant components in various diversified therapeutic applications. A literature review reveals that benzothiazoles and their derivatives show considerable activity, including potent inhibition of human immunodeficiency virus type 1 (HIV-1) replication by HIV-1 protease inhibition (Yaseen *et al.*, 2006), antitumor (Kini *et al.*, 2007), anthelmintic (Munirajasekhar *et al.*, 2011), analgesic and anti-inflammatory (Gurupadayya *et al.*, 2008), antimalarial (Bowyer *et al.*, 2007), antifungal (Mittal *et al.*, 2007), anticandidal activities (Pozas *et al.*, 2005) and various activities relating to the central nervous system (Rana *et al.*, 2008).

In the title molecule (Fig. 1), the benzo[d]thiazol-2-yl ring system (S1/N1/C1–C7) is nearly planar (r.m.s. deviation = 0.023). Bond lengths (Allen *et al.*, 1987) and angles are within normal ranges and are comparable with a related structure (Fun *et al.*, 2012).

In the crystal structure, Fig. 2, molecules are linked *via* intermolecular N2—H1N2···N1 and N3—H2N3···N3 hydrogen bonds (Table 1) into two-dimensional networks parallel to (100).

S2. Experimental

Concentrated HCl (6 ml) was added drop-wise to hydrazine hydrate [6 ml, 0.12 mol] at 273–283 K followed by ethylene glycol (50 ml). To the above solution, 5-chloro-6-fluoro benzothiazol-2-amine [6.079 g, 0.03 mol] was added in portions. It was then refluxed for 3–4 h. A colourless solid was precipitated at the end of the reflux period. The mixture was cooled and the product was filtered and then washed with water several times. It was air dried and recrystallized using ethanol. The single crystals were grown by slow evaporation from solvent methanol (m.p. = 483–485 K).

S3. Refinement

All hydrogen atoms were located in a difference Fourier map and refined freely with N—H = 0.815 (16)–0.905 (15) Å and C—H = 0.951 (14) or 0.966 (15) Å.

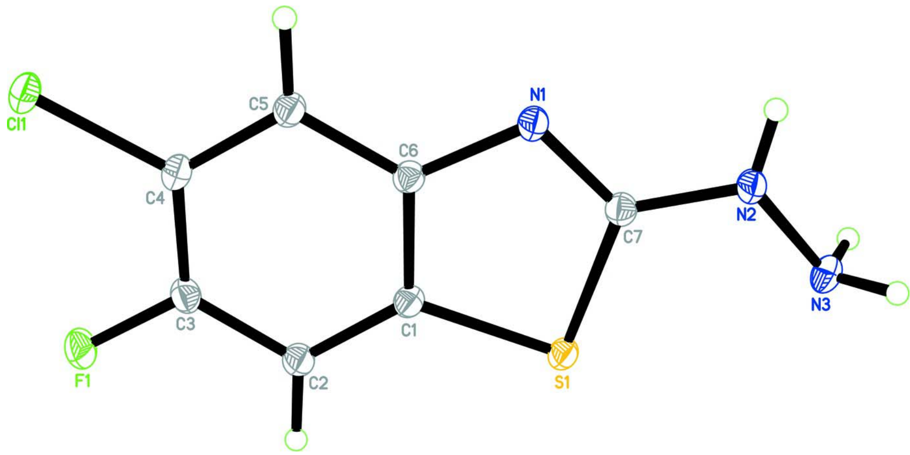

Figure 1

The molecular structure of the title compound showing 50% probability displacement ellipsoids for non-H atoms.

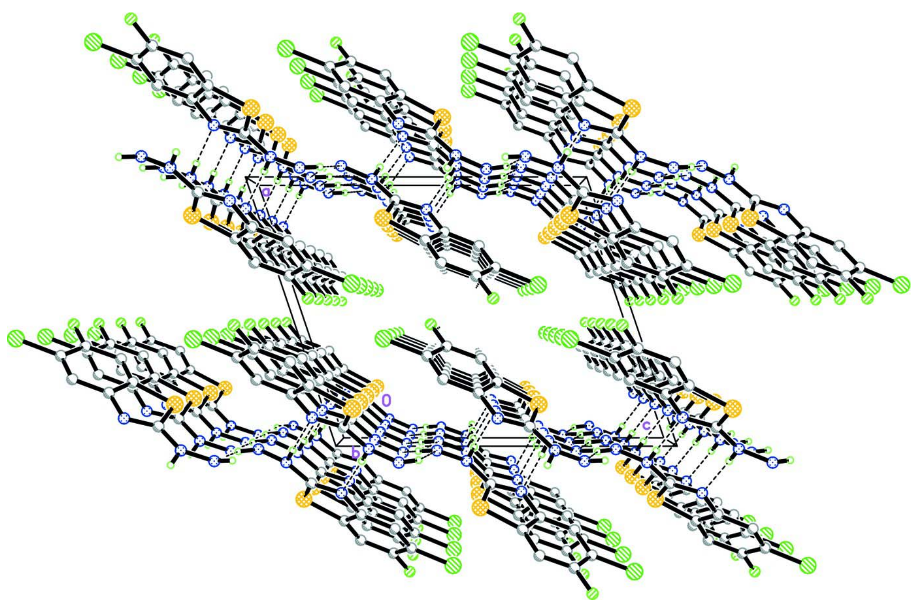

Figure 2

The crystal structure of the title compound, viewed along the *b* axis. H atoms not involved in hydrogen bonds (dashed lines) have been omitted for clarity.

1-(5-Chloro-6-fluoro-1,3-benzothiazol-2-yl)hydrazine

Crystal data

$C_7H_5ClFN_3S$
M_r = 217.65
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 11.1287 (6) Å
b = 5.6641 (3) Å
c = 13.3419 (7) Å
β = 108.552 (1)°
V = 797.29 (7) Å^3
Z = 4
$F(000)$ = 440
D_x = 1.813 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5638 reflections
θ = 3.9–32.6°
μ = 0.70 mm^{-1}
T = 100 K
Block, colourless
0.31 × 0.16 × 0.14 mm

Data collection

Bruker SMART APEXII DUO CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.813$, $T_{max} = 0.908$
9459 measured reflections
2899 independent reflections
2638 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 32.7°$, $\theta_{min} = 1.9°$
$h = -16 \rightarrow 16$
$k = -8 \rightarrow 8$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.066$
$S = 1.07$
2899 reflections
138 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0319P)^2 + 0.296P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.53$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
S1	0.17661 (2)	0.55946 (4)	0.127235 (17)	0.01252 (6)
F1	0.45495 (6)	0.83076 (11)	−0.09724 (5)	0.01932 (13)
Cl1	0.40693 (2)	0.42019 (4)	−0.236217 (18)	0.01702 (6)
N1	0.13780 (7)	0.19036 (15)	0.00140 (6)	0.01316 (14)
N2	0.02947 (8)	0.17861 (16)	0.12535 (6)	0.01531 (15)
N3	−0.01178 (8)	0.31566 (15)	0.19707 (6)	0.01435 (15)
C1	0.25396 (8)	0.54873 (16)	0.03242 (7)	0.01196 (15)
C2	0.33315 (8)	0.71802 (17)	0.01037 (7)	0.01342 (15)
C3	0.37807 (8)	0.67098 (17)	−0.07310 (7)	0.01365 (16)
C4	0.34704 (8)	0.46473 (17)	−0.13335 (7)	0.01302 (15)
C5	0.26897 (8)	0.29589 (17)	−0.11025 (7)	0.01283 (15)
C6	0.22136 (8)	0.33884 (16)	−0.02676 (7)	0.01158 (15)
C7	0.10783 (8)	0.28418 (17)	0.08008 (7)	0.01228 (15)
H2A	0.3595 (12)	0.862 (3)	0.0500 (11)	0.014 (3)*

H5A	0.2461 (12)	0.158 (3)	−0.1530 (11)	0.015 (3)*
H1N2	−0.0159 (14)	0.073 (3)	0.0922 (12)	0.023 (4)*
H1N3	−0.0937 (14)	0.360 (3)	0.1679 (12)	0.023 (4)*
H2N3	−0.0068 (14)	0.231 (3)	0.2508 (12)	0.025 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01537 (10)	0.01201 (11)	0.01173 (10)	−0.00092 (7)	0.00648 (7)	−0.00130 (7)
F1	0.0220 (3)	0.0177 (3)	0.0227 (3)	−0.0073 (2)	0.0133 (2)	−0.0019 (2)
Cl1	0.01930 (11)	0.01979 (12)	0.01571 (10)	−0.00142 (8)	0.01083 (8)	−0.00148 (8)
N1	0.0149 (3)	0.0130 (3)	0.0136 (3)	−0.0015 (3)	0.0075 (3)	−0.0013 (3)
N2	0.0196 (4)	0.0146 (4)	0.0155 (3)	−0.0048 (3)	0.0108 (3)	−0.0036 (3)
N3	0.0165 (3)	0.0160 (4)	0.0128 (3)	0.0018 (3)	0.0079 (3)	0.0001 (3)
C1	0.0131 (3)	0.0121 (4)	0.0113 (3)	0.0002 (3)	0.0048 (3)	−0.0004 (3)
C2	0.0149 (4)	0.0121 (4)	0.0142 (4)	−0.0013 (3)	0.0060 (3)	−0.0008 (3)
C3	0.0133 (3)	0.0136 (4)	0.0153 (4)	−0.0018 (3)	0.0062 (3)	0.0007 (3)
C4	0.0134 (3)	0.0150 (4)	0.0122 (3)	0.0010 (3)	0.0063 (3)	0.0002 (3)
C5	0.0134 (3)	0.0136 (4)	0.0124 (3)	0.0004 (3)	0.0054 (3)	−0.0007 (3)
C6	0.0124 (3)	0.0112 (4)	0.0118 (3)	0.0003 (3)	0.0047 (3)	−0.0002 (3)
C7	0.0133 (3)	0.0120 (4)	0.0121 (3)	−0.0004 (3)	0.0050 (3)	0.0003 (3)

Geometric parameters (Å, º)

S1—C1	1.7429 (9)	N3—H2N3	0.849 (17)
S1—C7	1.7625 (10)	C1—C2	1.3957 (13)
F1—C3	1.3529 (11)	C1—C6	1.4093 (13)
Cl1—C4	1.7243 (9)	C2—C3	1.3839 (12)
N1—C7	1.3109 (11)	C2—H2A	0.966 (15)
N1—C6	1.3912 (11)	C3—C4	1.3977 (13)
N2—C7	1.3483 (11)	C4—C5	1.3910 (13)
N2—N3	1.4172 (11)	C5—C6	1.3986 (12)
N2—H1N2	0.815 (16)	C5—H5A	0.951 (14)
N3—H1N3	0.905 (15)		
C1—S1—C7	88.21 (4)	F1—C3—C4	118.78 (8)
C7—N1—C6	109.48 (8)	C2—C3—C4	122.51 (9)
C7—N2—N3	117.01 (8)	C5—C4—C3	120.30 (8)
C7—N2—H1N2	117.3 (11)	C5—C4—Cl1	120.18 (7)
N3—N2—H1N2	119.3 (11)	C3—C4—Cl1	119.52 (7)
N2—N3—H1N3	111.1 (10)	C4—C5—C6	118.59 (8)
N2—N3—H2N3	108.3 (11)	C4—C5—H5A	119.8 (8)
H1N3—N3—H2N3	107.7 (14)	C6—C5—H5A	121.5 (8)
C2—C1—C6	121.92 (8)	N1—C6—C5	124.41 (8)
C2—C1—S1	128.31 (7)	N1—C6—C1	115.70 (8)
C6—C1—S1	109.73 (7)	C5—C6—C1	119.85 (8)
C3—C2—C1	116.83 (9)	N1—C7—N2	122.99 (9)
C3—C2—H2A	118.6 (8)	N1—C7—S1	116.89 (7)

C1—C2—H2A	124.6 (8)	N2—C7—S1	120.11 (7)
F1—C3—C2	118.72 (8)		

C7—S1—C1—C2	177.55 (9)	C7—N1—C6—C1	−0.18 (11)
C7—S1—C1—C6	0.05 (7)	C4—C5—C6—N1	176.84 (8)
C6—C1—C2—C3	0.32 (13)	C4—C5—C6—C1	−0.63 (13)
S1—C1—C2—C3	−176.91 (7)	C2—C1—C6—N1	−177.63 (8)
C1—C2—C3—F1	179.88 (8)	S1—C1—C6—N1	0.06 (10)
C1—C2—C3—C4	−0.13 (14)	C2—C1—C6—C5	0.06 (14)
F1—C3—C4—C5	179.54 (8)	S1—C1—C6—C5	177.75 (7)
C2—C3—C4—C5	−0.45 (14)	C6—N1—C7—N2	−179.09 (8)
F1—C3—C4—Cl1	−0.12 (12)	C6—N1—C7—S1	0.22 (10)
C2—C3—C4—Cl1	179.89 (7)	N3—N2—C7—N1	−169.63 (8)
C3—C4—C5—C6	0.82 (13)	N3—N2—C7—S1	11.09 (11)
Cl1—C4—C5—C6	−179.52 (7)	C1—S1—C7—N1	−0.16 (8)
C7—N1—C6—C5	−177.75 (8)	C1—S1—C7—N2	179.17 (8)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1*N*2···N1^{i}	0.816 (16)	2.132 (16)	2.9478 (12)	176.9 (16)
N3—H2*N*3···N3^{ii}	0.850 (16)	2.443 (17)	3.1382 (12)	139.5 (14)

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