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2-(1,3-Benzothiazol-2-yl)guanidinium chloride

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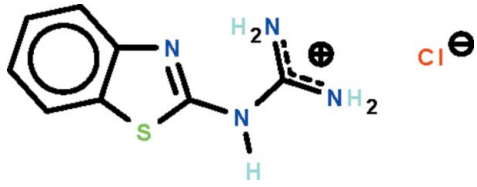
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.003 Å; R factor = 0.030; wR factor = 0.076; data-to-parameter ratio = 14.6.

The non-H atoms of the cation of the title salt, $C_8H_9N_4S^+\cdot Cl^-$, are approximately co-planar (r.m.s. deviation = 0.037 Å), with one amino group forming an intramolecular hydrogen bond to the tertiary N atom of the benzothiazole fused-ring system. The cations and anions are linked by cyclic $R_2^1(6)$ N—H···Cl hydrogen-bonding associations, generating helical chains running along the b-axis direction.

Related literature

For the synthesis, see: Takahashi & Niino (1943). For the structure of 2-(1,3-benzothiazol-2-yl)guanidine, see: Mohamed *et al.* (2011). For graph-set analysis, see: Etter *et al.* (1990).



Experimental

Crystal data

$C_8H_9N_4S^+\cdot Cl^-$
M_r = 228.71
Orthorhombic, $P2_12_12_1$
a = 3.8857 (5) Å
b = 11.0349 (17) Å
c = 22.186 (3) Å
V = 951.3 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.58 mm^{-1}
T = 120 K
0.12 × 0.03 × 0.01 mm

Data collection

Rigaku Saturn 724+ diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2011)
T_{min} = 0.933, T_{max} = 0.994
14016 measured reflections
2146 independent reflections
2117 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.076
S = 1.07
2146 reflections
147 parameters
5 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983), 839 Friedel pairs
Flack parameter: −0.01 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1···Cl1^{i}	0.88 (1)	2.21 (1)	3.074 (2)	165 (2)
N3—H2···Cl1	0.88 (1)	2.62 (2)	3.380 (2)	146 (2)
N4—H3···Cl1	0.88 (1)	2.31 (1)	3.157 (2)	160 (2)
N4—H4···N1	0.88 (1)	2.06 (2)	2.713 (2)	131 (2)

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

Data collection: *CrystalClear* (Rigaku, 2011); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2157).

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supporting information

Acta Cryst. (2011). E**67**, o3132 [doi:10.1107/S1600536811044643]

2-(1,3-Benzothiazol-2-yl)guanidinium chloride

Shaaban K. Mohamed, Peter N. Horton, Mahmoud A.A. El-Remaily and Seik Weng Ng

S1. Comment

A recent study (Mohamed *et al.*, 2011) describes the crystal structure of 2-(1,3-benzothiazol-2-yl)guanidine, which was synthesized by the reaction of 2-aminothiophenol and cyanoguanidine in 10% sulfuric acid medium. The product of the reaction is probably a sulfate or bisulfate salt that is then converted to the neutral compound upon treatment with sodium hydroxide. In the present study, 2-(1,3-benzothioazol-2-yl)guanidine is converted to the hydrochloride salt by treatment with hydrochloric acid. The non-H atoms of the cation of the title salt, $C_8H_9N_4S^+$ Cl^- (Scheme I), lie on a plane (r.m.s. deviation 0.037 Å), with one amino group forming an intramolecular hydrogen bond to the tertiary N atom of the benzothiazole fused-ring (Fig. 1). The cations and anions are linked by cyclic N—H···Cl hydrogen-bonding associations [graph set $R^1_2(6)$ (Etter *et al.*, 1990)] (Table 1), to generate helical chains running along the *b*-axis of the orthorhombic unit cell. This salt was first reported in 1943 (Takahashi & Niino, 1943).

S2. Experimental

2-(1,3-Benzothiazol-2-yl)guanidine (0.05 mol) was heated in ethanol (50 ml) in the presence of a few drops of hydrochloric acid for 3 h. The mixture was cooled and the product was collected and recrystallized from ethanol to give the title compound (m.p. 523 K) in 95% yield; .

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H = 0.95 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C). The amino H-atoms were located in a difference Fourier map, and were refined with a distance restraint of N—H = 0.88±0.01 Å with their isotropic displacement parameters freely refining.

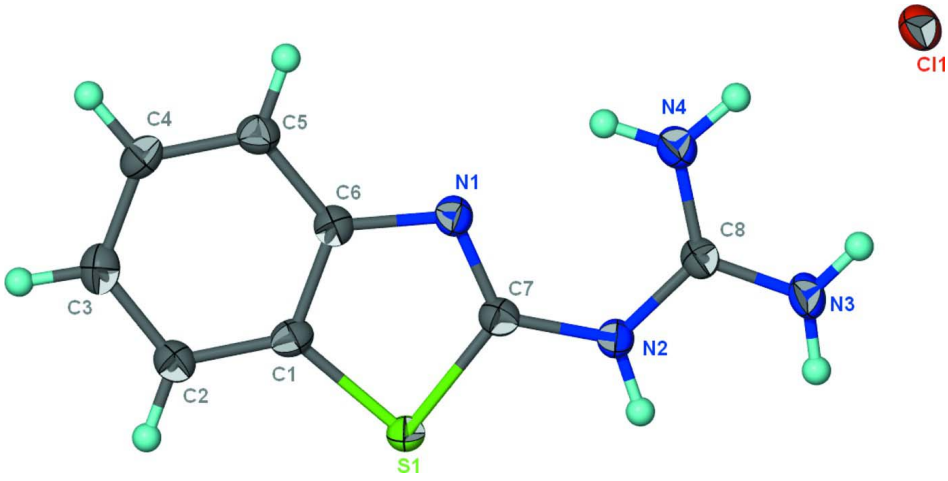

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_8H_9N_4S^+$ Cl^- at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

2-(1,3-Benzothiazol-2-yl)guanidinium chloride

Crystal data

$C_8H_9N_4S^+\cdot Cl^-$
M_r = 228.71
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
a = 3.8857 (5) Å
b = 11.0349 (17) Å
c = 22.186 (3) Å
V = 951.3 (2) Å^3
Z = 4
F(000) = 472
D_x = 1.597 Mg m^{-3}
Melting point: 523 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3321 reflections
θ = 2.1–31.0°
μ = 0.58 mm^{-1}
T = 120 K
Lath, colorless
0.12 × 0.03 × 0.01 mm

Data collection

Rigaku Saturn 724+ diffractometer
Radiation source: rotating anode
Confocal monochromator
Detector resolution: 28.5714 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2011)
T_{min} = 0.933, T_{max} = 0.994
14016 measured reflections
2146 independent reflections
2117 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.5°, θ_{min} = 2.1°
h = −4→4
k = −14→14
l = −28→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.076
S = 1.07
2146 reflections
147 parameters
5 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.3169P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Absolute structure: Flack (1983), 839 Friedel pairs
Absolute structure parameter: −0.01 (7)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.94400 (14)	0.19472 (4)	0.72036 (2)	0.02120 (13)
S1	0.32449 (13)	0.63436 (4)	0.96176 (2)	0.01619 (12)
N1	0.6155 (4)	0.42234 (14)	0.95141 (7)	0.0168 (4)
N2	0.4500 (5)	0.51363 (15)	0.85919 (7)	0.0175 (4)
H1	0.344 (7)	0.5752 (17)	0.8420 (12)	0.032 (7)*
N3	0.4832 (5)	0.43982 (16)	0.76315 (7)	0.0209 (4)
H2	0.539 (7)	0.3806 (17)	0.7388 (10)	0.027 (7)*
H5	0.390 (8)	0.5097 (17)	0.7530 (14)	0.053 (10)*
N4	0.7193 (5)	0.32801 (15)	0.83996 (8)	0.0211 (4)
H3	0.792 (7)	0.2752 (18)	0.8130 (9)	0.029 (7)*
H4	0.766 (7)	0.320 (2)	0.8786 (5)	0.023 (6)*
C1	0.4490 (5)	0.55994 (17)	1.02742 (9)	0.0168 (4)
C2	0.4162 (5)	0.59888 (18)	1.08674 (9)	0.0179 (4)
H2A	0.3142	0.6748	1.0962	0.021*
C3	0.5382 (5)	0.52252 (18)	1.13174 (9)	0.0195 (4)
H3A	0.5207	0.5467	1.1727	0.023*
C4	0.6856 (6)	0.41107 (18)	1.11753 (9)	0.0188 (4)
H4A	0.7657	0.3602	1.1491	0.023*
C5	0.7178 (6)	0.37284 (18)	1.05846 (9)	0.0186 (4)
H5A	0.8187	0.2967	1.0492	0.022*
C6	0.5993 (5)	0.44847 (17)	1.01311 (9)	0.0160 (4)
C7	0.4809 (5)	0.51068 (17)	0.92100 (9)	0.0158 (4)
C8	0.5560 (6)	0.42508 (17)	0.82086 (8)	0.0162 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0242 (3)	0.0190 (2)	0.0205 (2)	−0.0008 (2)	−0.00028 (19)	−0.00403 (19)
S1	0.0198 (2)	0.0132 (2)	0.0156 (2)	0.00165 (17)	−0.0007 (2)	0.00035 (18)
N1	0.0209 (9)	0.0139 (7)	0.0155 (8)	−0.0013 (6)	−0.0004 (7)	−0.0005 (6)
N2	0.0254 (9)	0.0127 (7)	0.0144 (8)	0.0006 (7)	−0.0006 (7)	0.0001 (6)
N3	0.0310 (10)	0.0157 (8)	0.0159 (8)	−0.0017 (8)	−0.0003 (8)	−0.0036 (7)
N4	0.0290 (10)	0.0167 (8)	0.0174 (8)	0.0022 (8)	0.0016 (8)	−0.0009 (7)
C1	0.0172 (9)	0.0141 (8)	0.0191 (9)	−0.0010 (8)	−0.0012 (8)	0.0029 (7)
C2	0.0183 (10)	0.0164 (9)	0.0189 (9)	−0.0014 (8)	0.0021 (8)	−0.0008 (7)

C3	0.0190 (10)	0.0228 (10)	0.0167 (9)	−0.0059 (9)	0.0013 (8)	0.0004 (8)
C4	0.0181 (9)	0.0204 (9)	0.0178 (9)	−0.0017 (9)	−0.0013 (8)	0.0053 (7)
C5	0.0205 (10)	0.0155 (9)	0.0199 (9)	0.0007 (8)	0.0004 (8)	0.0007 (7)
C6	0.0158 (10)	0.0154 (9)	0.0169 (9)	−0.0022 (7)	0.0009 (7)	0.0000 (7)
C7	0.0161 (10)	0.0134 (8)	0.0179 (9)	−0.0015 (7)	0.0011 (8)	0.0006 (7)
C8	0.0191 (9)	0.0142 (8)	0.0154 (9)	−0.0024 (8)	0.0033 (8)	0.0008 (7)

Geometric parameters (Å, º)

S1—C1	1.7409 (19)	N4—H4	0.882 (10)
S1—C7	1.746 (2)	C1—C2	1.390 (3)
N1—C7	1.296 (2)	C1—C6	1.398 (3)
N1—C6	1.400 (2)	C2—C3	1.390 (3)
N2—C8	1.359 (2)	C2—H2A	0.9500
N2—C7	1.377 (2)	C3—C4	1.393 (3)
N2—H1	0.882 (10)	C3—H3A	0.9500
N3—C8	1.321 (3)	C4—C5	1.382 (3)
N3—H2	0.876 (10)	C4—H4A	0.9500
N3—H5	0.880 (10)	C5—C6	1.386 (3)
N4—C8	1.315 (3)	C5—H5A	0.9500
N4—H3	0.883 (10)		
C1—S1—C7	88.16 (9)	C2—C3—H3A	119.6
C7—N1—C6	109.63 (17)	C4—C3—H3A	119.6
C8—N2—C7	125.43 (17)	C5—C4—C3	121.41 (19)
C8—N2—H1	115.2 (19)	C5—C4—H4A	119.3
C7—N2—H1	119.3 (19)	C3—C4—H4A	119.3
C8—N3—H2	116.9 (18)	C4—C5—C6	118.31 (19)
C8—N3—H5	116 (2)	C4—C5—H5A	120.8
H2—N3—H5	127 (3)	C6—C5—H5A	120.8
C8—N4—H3	118.3 (16)	C5—C6—C1	120.25 (18)
C8—N4—H4	119.6 (16)	C5—C6—N1	124.80 (18)
H3—N4—H4	122 (2)	C1—C6—N1	114.95 (17)
C2—C1—C6	121.68 (18)	N1—C7—N2	124.85 (18)
C2—C1—S1	128.38 (15)	N1—C7—S1	117.31 (15)
C6—C1—S1	109.95 (14)	N2—C7—S1	117.84 (14)
C3—C2—C1	117.47 (18)	N4—C8—N3	121.06 (18)
C3—C2—H2A	121.3	N4—C8—N2	122.02 (18)
C1—C2—H2A	121.3	N3—C8—N2	116.92 (18)
C2—C3—C4	120.87 (19)		
C7—S1—C1—C2	179.9 (2)	S1—C1—C6—N1	−0.1 (2)
C7—S1—C1—C6	0.28 (16)	C7—N1—C6—C5	179.4 (2)
C6—C1—C2—C3	−0.1 (3)	C7—N1—C6—C1	−0.3 (2)
S1—C1—C2—C3	−179.68 (17)	C6—N1—C7—N2	−178.94 (19)
C1—C2—C3—C4	−0.3 (3)	C6—N1—C7—S1	0.5 (2)
C2—C3—C4—C5	0.4 (3)	C8—N2—C7—N1	−0.2 (3)
C3—C4—C5—C6	0.0 (3)	C8—N2—C7—S1	−179.64 (17)

C4—C5—C6—C1	−0.5 (3)	C1—S1—C7—N1	−0.50 (16)
C4—C5—C6—N1	179.77 (19)	C1—S1—C7—N2	179.02 (17)
C2—C1—C6—C5	0.6 (3)	C7—N2—C8—N4	−3.7 (3)
S1—C1—C6—C5	−179.81 (16)	C7—N2—C8—N3	175.3 (2)
C2—C1—C6—N1	−179.68 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···Cl1^{i}	0.88 (1)	2.21 (1)	3.074 (2)	165 (2)
N3—H2···Cl1	0.88 (1)	2.62 (2)	3.380 (2)	146 (2)
N4—H3···Cl1	0.88 (1)	2.31 (1)	3.157 (2)	160 (2)
N4—H4···N1	0.88 (1)	2.06 (2)	2.713 (2)	131 (2)

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+3/2.