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2-(1,3-Benzothiazol-2-yl)guanidin-2-ium acetate

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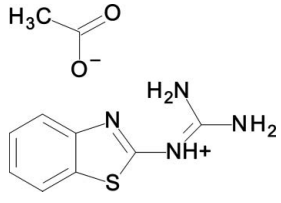
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.011 Å; R factor = 0.078; wR factor = 0.154; data-to-parameter ratio = 12.8.

In the title compound, $C_8H_9N_4S^+\cdot C_2H_3O_2^-$, the cation is essentially planar (r.m.s deviation = 0.037 Å) with the guanidine unit bent out of the plane of the fused-ring system by 4.6 (3)°. In the asymmetric unit, the cations and anions are linked into $R_2^2(8)$ motifs. In the crystal, further N—H···O and N—H···N hydrogen bonds link the components into a two-dimensional network.

Related literature

For the crystal structure of the neutral 2-(1,3-benzothiazol-2-yl)guanidine molecule, see: Mohamed *et al.* (2011). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_8H_9N_4S^+\cdot C_2H_3O_2^-$
M_r = 252.30
Orthorhombic, $Pca2_1$
a = 12.596 (2) Å
b = 11.276 (2) Å
c = 8.0936 (12) Å
V = 1149.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 120 K
0.14 × 0.10 × 0.02 mm

Data collection

Bruker–Nonius APEXII CCD camera on κ-goniostat diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.962, T_{max} = 0.995
7697 measured reflections
1991 independent reflections
1301 reflections with $I > 2\sigma(I)$
R_{int} = 0.116

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.154
S = 1.06
1991 reflections
155 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Absolute structure: Flack (1983), 897 Friedel pairs
Flack parameter: 0.3 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O12	0.88	1.82	2.671 (8)	162
N3—H3A···O11^{i}	0.88	2.06	2.760 (8)	136
N3—H3B···N1	0.88	2.05	2.713 (9)	131
N4—H4A···O12^{ii}	0.88	2.03	2.861 (7)	158
N4—H4B···O11	0.88	1.91	2.790 (8)	173

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

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2373).

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supporting information

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S1. Comment

The title compound was synthesized and exists as the acetate salt of benzothiazolo-2-guanidinium. The benzothiazolo-2-guanidinium cation is almost planar with the guanidine unit bent out of the plane of the fused-ring system by just 4.6 (3)°. In the asymmetric unit, The cations and anions are linked into $R^2_2(8)$ motif (Bernstein, *et al.*, 1995). The crystal packing is stabilized by intermolecular hydrogen bonds involving the cations and acetate counter-ions, Table 1, Fig.2.

S2. Experimental

A mixture of 1 mmol of 2-guanidyl benzothiazole with few drops of glacial acetic acid was heated in ethanol for 2 hours. The mixture was left at room temperature for two days to afford the shiny white crystals of benzothiazolo-2-guanidinium acetate in 94% yield. The single-crystal was obtained from a slow evaporation of the ethanolic solution of product over two days.

S3. Refinement

H atoms were positioned geometrically [C—H = 0.95 or 0.98 Å and N—H = 0.88 Å] and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or U_{iso}(H) = 1.5U_{eq}(C) respectively and U_{iso}(H) = 1.2U_{eq}(N).

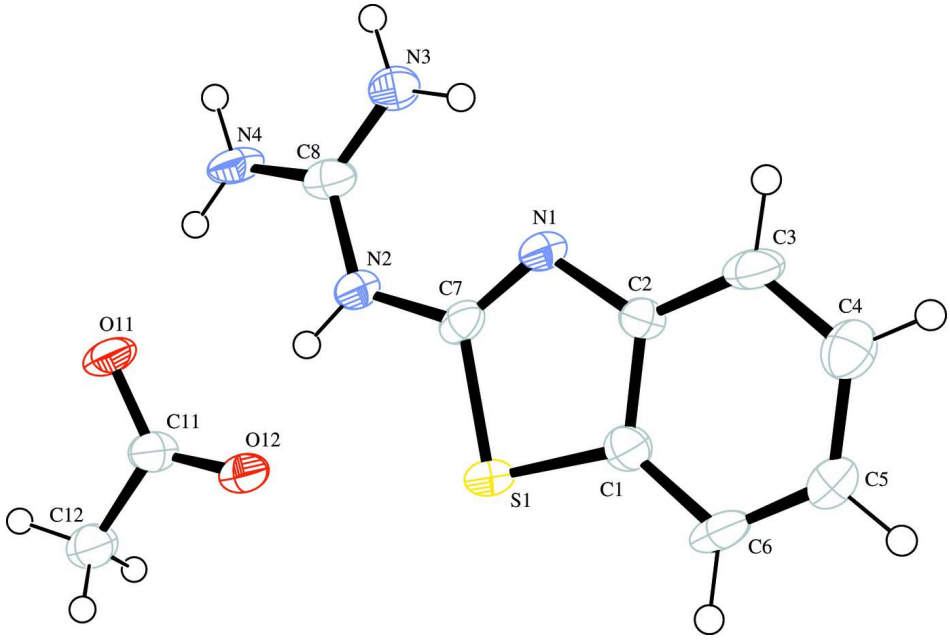

Figure 1

The asymmetric unit of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

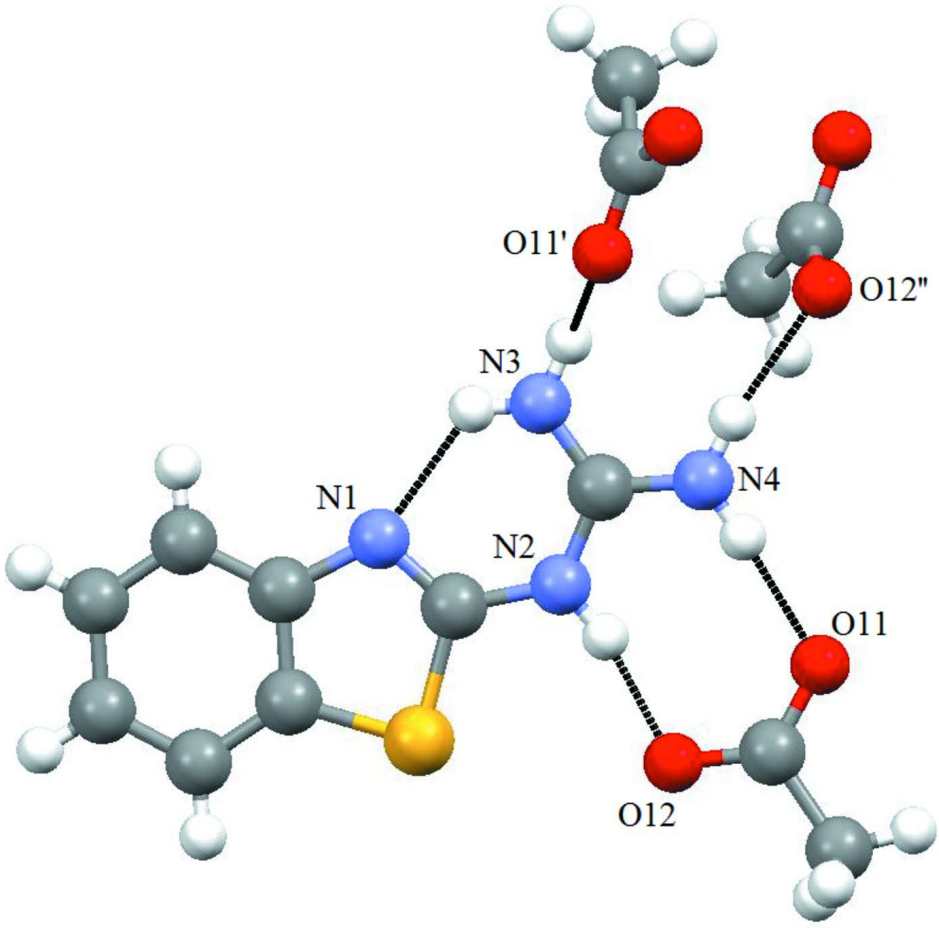

Figure 2

A partial packing diagram for (I), showing the intermolecular and intramolecular hydrogen bonding (symmetry codes: (i) : -x+1, y,z+1/2; (ii) x+1/2,-y,z)

2-(1,3-Benzothiazol-2-yl)guanidin-2-ium acetate

Crystal data

$C_8H_9N_4S^+·C_2H_3O_2^-$
M_r = 252.30
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
a = 12.596 (2) Å
b = 11.276 (2) Å
c = 8.0936 (12) Å
V = 1149.6 (4) Å^3
Z = 4
F(000) = 528
D_x = 1.458 Mg m^{-3}
Melting point = 463–465 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2099 reflections
θ = 2.9–27.5°
μ = 0.28 mm^{-1}
T = 120 K
Plate, colourless
0.14 × 0.10 × 0.02 mm

Data collection

Bruker–Nonius APEXII CCD camera on κ-goniostat diffractometer
Radiation source: Bruker–Nonius FR591 rotating anode
10 cm confocal mirrors monochromator

Detector resolution: 4096x4096pixels / 62x62mm pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.962, T_{max} = 0.995
7697 measured reflections
1991 independent reflections
1301 reflections with $I > 2\sigma(I)$
R_{int} = 0.116
θ_{max} = 25.0°, θ_{min} = 3.2°
h = −14→14
k = −12→13
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.154
S = 1.06
1991 reflections
155 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.P)^2 + 4.3921P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Absolute structure: Flack (1983), 897 Friedel pairs
Absolute structure parameter: 0.3 (2)

Special details

Experimental. *SADABS* was used to perform the Absorption correction. Parameter refinement on 6249 reflections reduced *R*(int) from 0.1275 to 0.0768. Ratio of minimum to maximum apparent transmission: 0.627938. The given Tmin and Tmax were generated using the *SHELX* SIZE command

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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}
C1	0.1251 (6)	0.3579 (8)	0.7101 (8)	0.0287 (19)
C2	0.2293 (5)	0.3958 (7)	0.7510 (9)	0.0232 (17)
C3	0.2434 (6)	0.4986 (7)	0.8427 (10)	0.037 (2)
H3	0.3127	0.5226	0.8751	0.045*
C4	0.1569 (6)	0.5650 (8)	0.8861 (9)	0.037 (2)
H4	0.1667	0.6362	0.9469	0.044*
C5	0.0545 (6)	0.5299 (8)	0.8424 (9)	0.037 (2)
H5	−0.0043	0.5775	0.8740	0.044*
C6	0.0372 (6)	0.4267 (8)	0.7537 (9)	0.034 (2)
H6	−0.0326	0.4034	0.7233	0.040*
C7	0.2709 (5)	0.2345 (7)	0.6117 (11)	0.0262 (18)
C8	0.4331 (5)	0.1292 (8)	0.5459 (9)	0.030 (2)
N1	0.3104 (5)	0.3205 (6)	0.6934 (7)	0.0294 (16)
N2	0.3265 (4)	0.1462 (6)	0.5312 (7)	0.0295 (16)

H2	0.2908	0.0978	0.4663	0.035*
N3	0.4938 (5)	0.2037 (6)	0.6268 (7)	0.0331 (17)
H3A	0.5628	0.1920	0.6311	0.040*
H3B	0.4655	0.2654	0.6768	0.040*
N4	0.4741 (5)	0.0366 (6)	0.4707 (8)	0.0347 (17)
H4A	0.5430	0.0241	0.4743	0.042*
H4B	0.4326	−0.0128	0.4168	0.042*
S1	0.13101 (12)	0.22884 (17)	0.5956 (3)	0.0316 (5)
C11	0.2365 (6)	−0.0922 (8)	0.3103 (10)	0.035 (2)
C12	0.1654 (6)	−0.1819 (8)	0.2300 (10)	0.039 (2)
H12A	0.1089	−0.1410	0.1695	0.058*
H12B	0.1340	−0.2329	0.3149	0.058*
H12C	0.2069	−0.2304	0.1531	0.058*
O11	0.3349 (4)	−0.1038 (6)	0.2875 (7)	0.0417 (15)
O12	0.1938 (4)	−0.0085 (5)	0.3904 (7)	0.0372 (14)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.026 (4)	0.041 (6)	0.019 (4)	0.002 (4)	−0.003 (3)	0.003 (3)
C2	0.022 (4)	0.019 (4)	0.029 (4)	0.001 (3)	−0.004 (3)	0.000 (3)
C3	0.018 (3)	0.062 (6)	0.032 (4)	−0.006 (5)	0.002 (3)	0.004 (6)
C4	0.038 (5)	0.035 (6)	0.037 (5)	0.000 (4)	0.008 (4)	0.002 (4)
C5	0.026 (4)	0.045 (7)	0.040 (5)	0.002 (4)	0.007 (4)	−0.008 (4)
C6	0.016 (4)	0.049 (6)	0.035 (5)	0.007 (4)	0.003 (3)	0.010 (5)
C7	0.022 (3)	0.036 (5)	0.021 (4)	0.005 (3)	0.003 (4)	0.007 (4)
C8	0.022 (4)	0.037 (5)	0.031 (5)	−0.003 (4)	−0.001 (3)	0.010 (4)
N1	0.017 (3)	0.040 (5)	0.031 (4)	0.002 (3)	0.000 (3)	−0.003 (3)
N2	0.019 (3)	0.040 (5)	0.029 (4)	0.000 (3)	0.001 (3)	−0.002 (3)
N3	0.022 (3)	0.040 (5)	0.037 (4)	0.009 (3)	−0.006 (3)	−0.001 (4)
N4	0.015 (3)	0.037 (5)	0.052 (4)	0.003 (3)	0.001 (3)	0.001 (4)
S1	0.0167 (7)	0.0426 (13)	0.0355 (10)	−0.0010 (9)	−0.0014 (10)	−0.0046 (12)
C11	0.024 (5)	0.045 (6)	0.035 (5)	−0.002 (4)	−0.001 (4)	0.000 (4)
C12	0.029 (4)	0.041 (6)	0.046 (5)	−0.002 (4)	0.003 (4)	−0.006 (4)
O11	0.018 (3)	0.049 (4)	0.058 (4)	0.000 (3)	0.006 (2)	−0.015 (3)
O12	0.021 (3)	0.045 (4)	0.045 (3)	−0.003 (3)	0.002 (3)	−0.010 (3)

Geometric parameters (Å, º)

C1—C6	1.398 (10)	C8—N3	1.311 (9)
C1—C2	1.420 (10)	C8—N4	1.314 (9)
C1—S1	1.727 (8)	C8—N2	1.361 (8)
C2—C3	1.388 (11)	N2—H2	0.8800
C2—N1	1.408 (9)	N3—H3A	0.8800
C3—C4	1.368 (10)	N3—H3B	0.8800
C3—H3	0.9500	N4—H4A	0.8800
C4—C5	1.395 (11)	N4—H4B	0.8800
C4—H4	0.9500	C11—O11	1.260 (9)

C5—C6	1.384 (11)	C11—O12	1.265 (10)
C5—H5	0.9500	C11—C12	1.499 (10)
C6—H6	0.9500	C12—H12A	0.9800
C7—N1	1.275 (10)	C12—H12B	0.9800
C7—N2	1.381 (10)	C12—H12C	0.9800
C7—S1	1.768 (6)		
C6—C1—C2	120.4 (7)	N3—C8—N2	121.9 (8)
C6—C1—S1	129.6 (6)	N4—C8—N2	117.3 (7)
C2—C1—S1	109.8 (6)	C7—N1—C2	110.3 (6)
C3—C2—N1	126.0 (7)	C8—N2—C7	124.1 (7)
C3—C2—C1	119.7 (7)	C8—N2—H2	118.0
N1—C2—C1	114.3 (7)	C7—N2—H2	118.0
C4—C3—C2	119.5 (8)	C8—N3—H3A	120.0
C4—C3—H3	120.3	C8—N3—H3B	120.0
C2—C3—H3	120.3	H3A—N3—H3B	120.0
C3—C4—C5	121.1 (8)	C8—N4—H4A	120.0
C3—C4—H4	119.5	C8—N4—H4B	120.0
C5—C4—H4	119.5	H4A—N4—H4B	120.0
C6—C5—C4	121.1 (8)	C1—S1—C7	88.5 (4)
C6—C5—H5	119.5	O11—C11—O12	124.8 (8)
C4—C5—H5	119.5	O11—C11—C12	117.0 (8)
C5—C6—C1	118.2 (7)	O12—C11—C12	118.2 (7)
C5—C6—H6	120.9	C11—C12—H12A	109.5
C1—C6—H6	120.9	C11—C12—H12B	109.5
N1—C7—N2	126.5 (6)	H12A—C12—H12B	109.5
N1—C7—S1	117.0 (6)	C11—C12—H12C	109.5
N2—C7—S1	116.4 (6)	H12A—C12—H12C	109.5
N3—C8—N4	120.7 (7)	H12B—C12—H12C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O12	0.88	1.82	2.671 (8)	162
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