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2-[(Prop-2-yn-1-yl)amino]anilinium chloride

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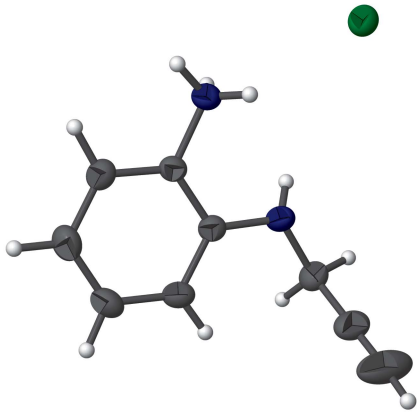
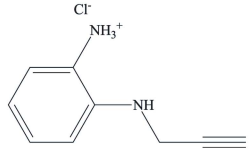
The title compound, $C_9H_{11}N_2^+\cdot Cl^-$, is an anilinium chloride salt, in which the $C_{ar}-N-C-C$ (ar = aromatic) torsion angle is $-84.95\,(18)^\circ$. In the crystal, a bilayer of cation–anion sheets runs parallel to (100), primarily through an extensive range of N—H···Cl hydrogen bonds. Weak offset π-stacking interactions between the benzene rings stack molecules along *c*.

3D view



Chemical scheme



Structure description

As a continuation of our studies of substituted 4-phenyl-1,5-benzodiazepin-2-one derivatives (Loughzail *et al.*, 2011; Ballo *et al.*, 2010), we have prepared the title compound (Fig. 1) by the action of hydroxylamine hydrochloride on 4-phenyl-1-(prop-2-yn-1-yl)-1*H*-1,5-benzodiazepin-2(3*H*)-one in ethanol.

In the title anilinium chloride salt, the N1 atom of the NH_3^+ substituent and the N2—H2*A* group lie in the plane of the benzene ring while the N2,C7,C8≡C9 substituent is inclined to the benzene ring at an angle of 81.57 (12)°.

In the crystal, the major packing interactions involve several N—H···Cl hydrogen bonds. Each of the H atoms of the NH_3^+ cations and the amine group form N—H···Cl hydrogen bonds with N1—H1*A* acting as a bifurcated donor while the N1—H1*B*···Cl1 contact is supported by a weaker C5—H5···Cl hydrogen bond, Table 1, Fig. 2. Weak, offset π-stacking interactions between the benzene rings stack molecules along *a* with centroid–centroid distances of 3.951 (2) Å and a dihedral angle of 7.02 (7)° between the rings. These interactions form bilayers running along the *c*axis direction (Fig. 3).

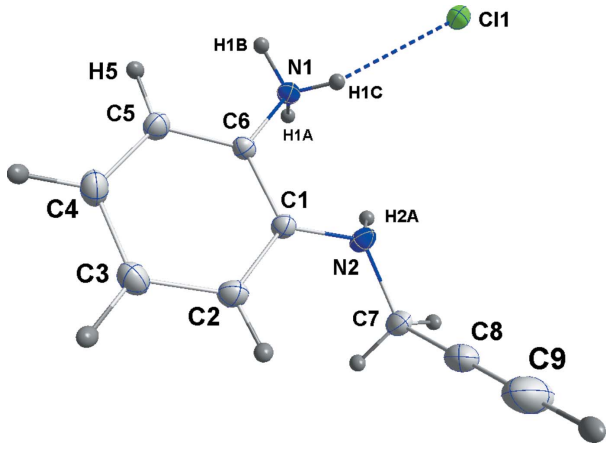


Figure 1
The title molecule with the atom-labelling scheme and 50% probability displacement ellipsoids.

Synthesis and crystallization

To a solution of 4-phenyl-1-(prop-2-yn-1-yl)-1*H*-1,5-benzodiazepin-2(3*H*)-one (10 mmol), was added hydroxylamine hydrochloride (20 mmol) in anhydrous ethanol (100 ml). The mixture was stirred at room temperature for 24 h. The solvent was removed under reduced pressure. The solid product was purified by recrystallization from ethanol solution to afford the title salt as colourless crystals.

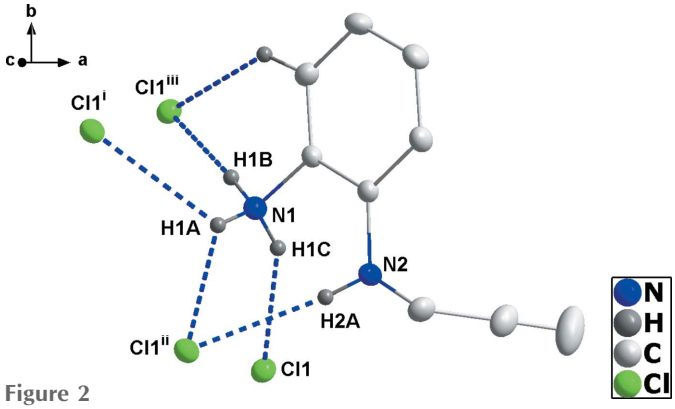


Figure 2
Details of the cation–anion interactions, with hydrogen bonds shown as dashed lines and symmetry operations as in Table 1.

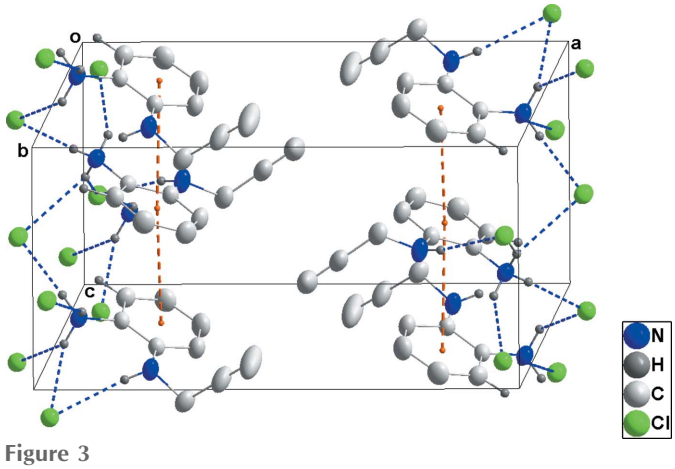


Figure 3
Overall packing viewed along the *b*-axis direction with π–π stacking interactions shown as dashed orange lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Cl1^{i}	0.90 (2)	2.72 (2)	3.2854 (17)	122.2 (17)
N1—H1A···Cl1^{ii}	0.90 (2)	2.56 (2)	3.2903 (18)	138.6 (18)
N1—H1B···Cl1^{iii}	0.92 (2)	2.29 (2)	3.2011 (16)	174.6 (16)
N1—H1C···Cl1	0.86 (2)	2.44 (2)	3.2064 (17)	148.2 (16)
N2—H2A···Cl1^{ii}	0.832 (17)	2.492 (17)	3.3148 (18)	170.1 (15)
C5—H5···Cl1^{iii}	0.996 (17)	2.935 (16)	3.7479 (19)	139.4 (12)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_9H_{11}N_2^+\cdot Cl^-$
M_r	182.65
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	14.736 (6), 7.955 (3), 7.843 (3)
β (°)	94.502 (5)
V (Å^3)	916.6 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.36
Crystal size (mm)	0.30 × 0.29 × 0.08
Data collection	
Diffractometer	Bruker SMART APEX CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.84, 0.97
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8431, 2311, 1732
R_{int}	0.033
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.677
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.106, 1.00
No. of reflections	2311
No. of parameters	153
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.50, −0.18

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

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2-[(Prop-2-yn-1-yl)amino]anilinium chloride

Crystal data

$C_9H_{11}N_2^+\cdot Cl^-$
M_r = 182.65
Monoclinic, $P2_1/c$
a = 14.736 (6) Å
b = 7.955 (3) Å
c = 7.843 (3) Å
β = 94.502 (5)°
V = 916.6 (7) Å^3
Z = 4
$F(000)$ = 384
D_x = 1.324 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3191 reflections
θ = 2.8–28.1°
μ = 0.36 mm^{-1}
T = 296 K
Plate, colourless
0.30 × 0.29 × 0.08 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.84, T_{max} = 0.97
8431 measured reflections
2311 independent reflections
1732 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 28.7°, θ_{min} = 1.4°
h = −19→19
k = −10→10
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.106
S = 1.00
2311 reflections
153 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0677P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

Experimental. The diffraction data were collected in three sets of 363 frames (0.5° width in ω) at φ = 0, 120 and 240°. A scan time of 25 sec/frame was used.

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.08712 (9)	0.56842 (17)	0.23286 (19)	0.0323 (3)
H1A	0.0601 (16)	0.533 (3)	0.325 (3)	0.079 (7)*
H1B	0.0470 (13)	0.633 (3)	0.166 (2)	0.056 (5)*
H1C	0.1026 (12)	0.485 (3)	0.172 (3)	0.055 (6)*
N2	0.24881 (9)	0.42987 (16)	0.38629 (18)	0.0373 (3)
H2A	0.2005 (12)	0.383 (2)	0.407 (2)	0.040 (5)*
C1	0.24303 (9)	0.60397 (17)	0.36781 (18)	0.0296 (3)
C2	0.31409 (11)	0.7126 (2)	0.4199 (2)	0.0379 (4)
H2	0.3669 (12)	0.663 (2)	0.465 (2)	0.043 (4)*
C3	0.30463 (11)	0.8851 (2)	0.4016 (2)	0.0433 (4)
H3	0.3552 (12)	0.953 (2)	0.438 (2)	0.049 (5)*
C4	0.22475 (12)	0.9538 (2)	0.3278 (2)	0.0435 (4)
H4	0.2185 (13)	1.075 (2)	0.317 (2)	0.060 (5)*
C5	0.15423 (10)	0.84824 (19)	0.27104 (19)	0.0355 (3)
H5	0.0949 (11)	0.890 (2)	0.217 (2)	0.042 (4)*
C6	0.16362 (9)	0.67665 (17)	0.29098 (17)	0.0275 (3)
C7	0.32679 (10)	0.3572 (2)	0.4861 (2)	0.0412 (4)
H7A	0.3111 (15)	0.243 (3)	0.526 (3)	0.066 (6)*
H7B	0.3429 (11)	0.419 (2)	0.591 (2)	0.043 (4)*
C8	0.40509 (11)	0.3334 (2)	0.3858 (2)	0.0513 (5)
C9	0.46786 (16)	0.3128 (4)	0.3068 (3)	0.0850 (8)
H9	0.516 (3)	0.307 (5)	0.237 (5)	0.147 (13)*
Cl1	0.05771 (2)	0.22948 (5)	0.01428 (5)	0.03535 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0282 (6)	0.0321 (7)	0.0357 (7)	−0.0017 (5)	−0.0030 (5)	−0.0011 (6)
N2	0.0259 (6)	0.0294 (6)	0.0555 (8)	−0.0006 (5)	−0.0033 (6)	0.0061 (6)
C1	0.0260 (7)	0.0292 (7)	0.0337 (7)	−0.0007 (5)	0.0026 (5)	0.0011 (6)
C2	0.0275 (7)	0.0388 (8)	0.0466 (9)	−0.0024 (6)	−0.0026 (7)	0.0015 (7)
C3	0.0375 (9)	0.0367 (8)	0.0552 (10)	−0.0101 (7)	−0.0001 (7)	−0.0047 (7)
C4	0.0468 (9)	0.0279 (8)	0.0561 (10)	−0.0035 (7)	0.0049 (8)	−0.0003 (7)
C5	0.0330 (8)	0.0324 (8)	0.0408 (8)	0.0041 (6)	0.0015 (6)	0.0014 (6)

C6	0.0246 (6)	0.0287 (7)	0.0293 (7)	−0.0022 (5)	0.0028 (5)	−0.0016 (5)
C7	0.0364 (8)	0.0358 (9)	0.0498 (10)	0.0057 (7)	−0.0059 (7)	0.0011 (8)
C8	0.0362 (9)	0.0601 (11)	0.0553 (11)	0.0059 (8)	−0.0113 (8)	−0.0156 (9)
C9	0.0405 (11)	0.142 (2)	0.0713 (16)	0.0057 (13)	−0.0047 (11)	−0.0393 (16)
Cl1	0.0343 (2)	0.0347 (2)	0.0365 (2)	0.00367 (14)	−0.00021 (15)	−0.00251 (14)

Geometric parameters (Å, º)

N1—C6	1.4626 (18)	C3—C4	1.383 (2)
N1—H1A	0.90 (2)	C3—H3	0.948 (19)
N1—H1B	0.92 (2)	C4—C5	1.382 (2)
N1—H1C	0.86 (2)	C4—H4	0.968 (19)
N2—C1	1.3945 (19)	C5—C6	1.380 (2)
N2—C7	1.4580 (19)	C5—H5	0.996 (17)
N2—H2A	0.832 (17)	C7—C8	1.459 (2)
C1—C2	1.394 (2)	C7—H7A	0.99 (2)
C1—C6	1.3986 (18)	C7—H7B	0.972 (17)
C2—C3	1.385 (2)	C8—C9	1.164 (3)
C2—H2	0.919 (17)	C9—H9	0.93 (4)
C6—N1—H1A	108.4 (15)	C5—C4—C3	119.21 (15)
C6—N1—H1B	107.3 (11)	C5—C4—H4	120.7 (12)
H1A—N1—H1B	109.3 (18)	C3—C4—H4	120.1 (12)
C6—N1—H1C	113.3 (12)	C6—C5—C4	119.90 (13)
H1A—N1—H1C	111.1 (19)	C6—C5—H5	116.9 (9)
H1B—N1—H1C	107.4 (17)	C4—C5—H5	123.2 (9)
C1—N2—C7	119.25 (13)	C5—C6—C1	122.07 (12)
C1—N2—H2A	115.0 (12)	C5—C6—N1	118.63 (12)
C7—N2—H2A	111.5 (11)	C1—C6—N1	119.29 (13)
C2—C1—N2	123.16 (13)	N2—C7—C8	112.68 (15)
C2—C1—C6	116.99 (13)	N2—C7—H7A	109.8 (13)
N2—C1—C6	119.84 (12)	C8—C7—H7A	105.2 (13)
C3—C2—C1	121.09 (14)	N2—C7—H7B	112.6 (10)
C3—C2—H2	122.9 (11)	C8—C7—H7B	111.8 (9)
C1—C2—H2	116.0 (11)	H7A—C7—H7B	104.1 (17)
C4—C3—C2	120.70 (15)	C9—C8—C7	179.3 (3)
C4—C3—H3	121.5 (11)	C8—C9—H9	174 (2)
C2—C3—H3	117.8 (11)		
C7—N2—C1—C2	9.2 (2)	C4—C5—C6—C1	0.0 (2)
C7—N2—C1—C6	−172.05 (13)	C4—C5—C6—N1	178.74 (14)
N2—C1—C2—C3	−178.87 (14)	C2—C1—C6—C5	−1.7 (2)
C6—C1—C2—C3	2.3 (2)	N2—C1—C6—C5	179.43 (13)
C1—C2—C3—C4	−1.3 (2)	C2—C1—C6—N1	179.61 (13)
C2—C3—C4—C5	−0.5 (2)	N2—C1—C6—N1	0.7 (2)
C3—C4—C5—C6	1.1 (2)	C1—N2—C7—C8	−84.95 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Cl1^{i}	0.90 (2)	2.72 (2)	3.2854 (17)	122.2 (17)
N1—H1*A*···Cl1^{ii}	0.90 (2)	2.56 (2)	3.2903 (18)	138.6 (18)
N1—H1*B*···Cl1^{iii}	0.92 (2)	2.29 (2)	3.2011 (16)	174.6 (16)
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Symmetry codes: (i) −*x*, *y*+1/2, −*z*+1/2; (ii) *x*, −*y*+1/2, *z*+1/2; (iii) −*x*, −*y*+1, −*z*.