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4-Aminopyridinium azide 4-aminopyridine solvate

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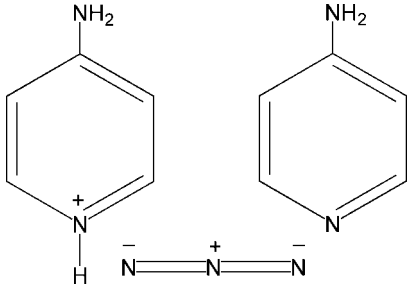
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.105; data-to-parameter ratio = 13.7.

In the title compound, $C_5H_7N_2^+\cdot N_3^-\cdot C_5H_6N_2$, all N atoms of the azide anion are situated on a twofold rotational axis, so the 4-aminopyridinium cation and 4-aminopyridine molecule, being related by symmetry, occupy one position in the asymmetric unit. Intermolecular N—H···N hydrogen bonds generate a three-dimensional hydrogen-bonding network which consolidates the crystal packing.

Related literature

For a related compound, see: Teulon *et al.* (1985).



Experimental

Crystal data

$C_5H_7N_2^+\cdot N_3^-\cdot C_5H_6N_2$ M_r = 231.27
Monoclinic, $C2/c$
a = 7.507 (3) Å
b = 12.247 (5) Å
c = 13.634 (5) Å
β = 99.278 (5)°
V = 1237.0 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 291 K
0.14 × 0.11 × 0.10 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.988, T_{max} = 0.992
3027 measured reflections
1096 independent reflections
852 reflections with $I > 2\sigma(I)$
R_{int} = 0.072

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.105
S = 1.08
1096 reflections
80 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N5^{i}	0.86	2.15	3.008 (2)	174
N1—H1B···N3^{ii}	0.86	2.14	2.9942 (18)	172
N2—H2A···N2^{iii}	0.86	1.84	2.689 (3)	169

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2787).

References

Bruker (2000). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Teulon, P., Delaplane, R. G., Olovsson, I. & Rozière, J. (1985). *Acta Cryst.* C**41**, 479–483.

supporting information

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S1. Comment

The crystal structure of 4-aminopyridine hemiperchlorate, has been previously reported (Teulon *et al.*, 1985). In this paper, we report the X-ray single-crystal structure of 4-aminopyridinium azide 4-aminopyridine (I).

The molecular structure of (I) is illustrated in Fig. 1. All N atoms of the azide anions are situated on a twofold rotational axis, so 4-aminopyridinium cation and 4-aminopyridine molecule being related by symmetry occupy one position in the asymmetric unit. Intermolecular N—H···N hydrogen bonds (Table 1) generate a three-dimensional hydrogen-bonding network which consolidate the crystal packing.

S2. Experimental

The title compound (I) was prepared by the treatment of 4-aminopyridine (0.5 mmol, 0.041 g) and excess sodium azide (NaN_3) in 20 ml methanol with a few drops of acetate acid (HOAc). Colourless single crystals suitable for X-ray diffraction measurement were grown from its methanol solution after five days′ slow evaporation at room temperature in air. Anal. Calcd. for $C_{10}H_{13}N_7$: C, 51.94; H, 5.66; N, 42.40%. Found: C, 51.85; H, 5.81; N, 42.29%. FT–IR (KBr pellets, cm^{-1}): 3447 (*vs*), 2057 (*s*), 1645 (*s*), 1463 (*m*), 1202 (w), 1202 (w), 840 (w), and 590 (w).

S3. Refinement

One restraint (DELU 0.001 C1 C2) was used to reduce the components of the anisotropic displacement parameters along chemical C—C bond. The H atoms were placed in geometrically idealized positions and refined as riding, with C—H = 0.93 Å and N—H = 0.86 Å, U_{iso}(H) = 1.2U_{eq}(C) and U_{iso}(H) = 1.2U_{eq}(N).

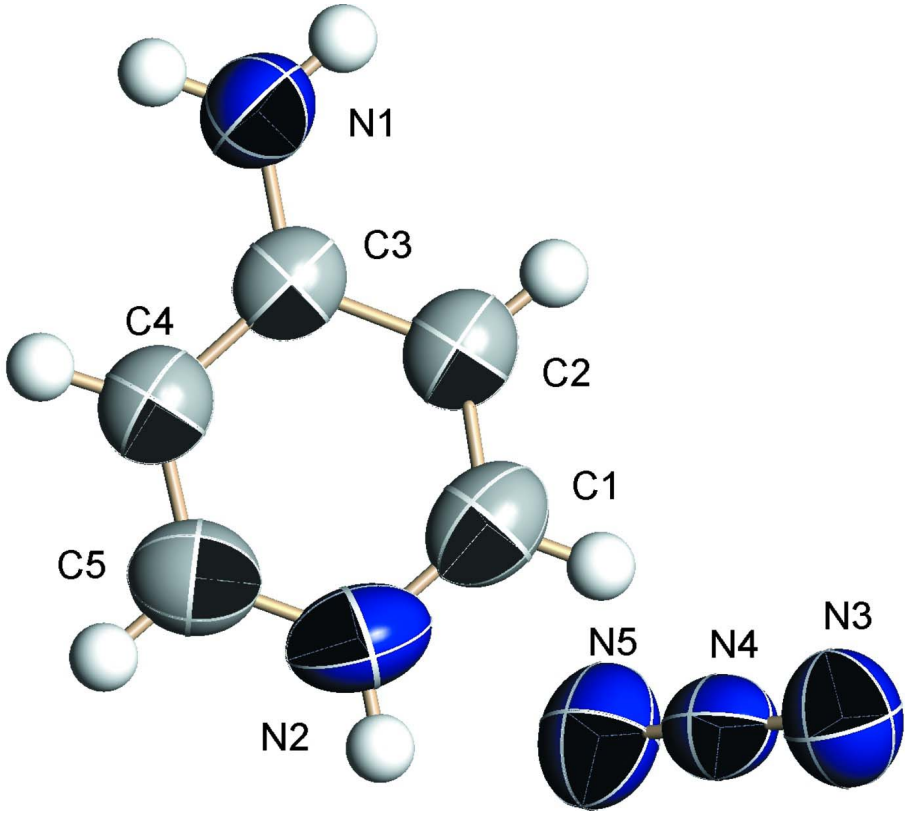

Figure 1

Molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

4-Aminopyridinium azide 4-aminopyridine solvate

Crystal data

$C_5H_7N_2^+\cdot N_3^-\cdot C_5H_6N_2$
$M_r = 231.27$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 7.507\ (3)$ Å
$b = 12.247\ (5)$ Å
$c = 13.634\ (5)$ Å
$\beta = 99.278\ (5)°$
$V = 1237.0\ (8)$ Å^3
$Z = 4$
$F(000) = 488$
$D_x = 1.242$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1359 reflections
$\theta = 3.0–25.4°$
$\mu = 0.08$ mm^{-1}
$T = 291$ K
Block, colourless
0.14 × 0.11 × 0.10 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.988$, $T_{max} = 0.992$
3027 measured reflections
1096 independent reflections
852 reflections with $I > 2\sigma(I)$
$R_{int} = 0.072$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.0°$
$h = -8\rightarrow8$
$k = -12\rightarrow14$
$l = -16\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.105$
$S = 1.08$
1096 reflections
80 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
$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.0478P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.11$ e Å^{-3}
$\Delta\rho_{min} = -0.11$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.042 (5)

Special details

Experimental. The structure was solved by direct methods (Bruker, 2000) and successive difference Fourier syntheses.
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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.1721 (2)	0.05406 (12)	−0.09812 (12)	0.0879 (5)	
H1	0.2321	0.0033	−0.1316	0.106*	
C2	0.22248 (18)	0.06466 (10)	0.00146 (11)	0.0780 (4)	
H2	0.3145	0.0214	0.0350	0.094*	
C3	0.13523 (16)	0.14103 (10)	0.05345 (9)	0.0699 (4)	
C4	−0.00162 (18)	0.20282 (11)	−0.00180 (11)	0.0791 (4)	
H4	−0.0635	0.2547	0.0294	0.095*	
C5	−0.0439 (2)	0.18672 (13)	−0.10131 (12)	0.0932 (5)	
H5	−0.1355	0.2286	−0.1371	0.112*	
N1	0.18164 (16)	0.15416 (9)	0.15211 (9)	0.0858 (4)	
H1A	0.1265	0.2016	0.1828	0.103*	
H1B	0.2665	0.1151	0.1846	0.103*	
N2	0.04055 (19)	0.11306 (11)	−0.15027 (9)	0.0948 (4)	
H2A	0.0108	0.1042	−0.2134	0.114*	0.50
N3	0.5000	−0.02503 (17)	0.7500	0.1009 (6)	
N4	0.5000	0.07152 (17)	0.7500	0.0760 (5)	
N5	0.5000	0.16656 (17)	0.7500	0.1062 (6)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0948 (10)	0.0867 (9)	0.0891 (8)	−0.0181 (8)	0.0353 (8)	−0.0118 (8)

C2	0.0744 (8)	0.0752 (8)	0.0879 (8)	−0.0108 (6)	0.0231 (6)	−0.0057 (6)
C3	0.0681 (7)	0.0682 (7)	0.0762 (9)	−0.0163 (6)	0.0206 (6)	−0.0037 (6)
C4	0.0765 (8)	0.0799 (8)	0.0834 (9)	−0.0071 (6)	0.0205 (7)	−0.0009 (7)
C5	0.0938 (10)	0.1000 (11)	0.0854 (11)	−0.0100 (8)	0.0132 (8)	0.0090 (8)
N1	0.0900 (8)	0.0873 (8)	0.0803 (8)	0.0016 (5)	0.0144 (6)	−0.0086 (6)
N2	0.1105 (10)	0.1050 (9)	0.0714 (8)	−0.0251 (7)	0.0223 (7)	−0.0032 (7)
N3	0.0964 (13)	0.0868 (12)	0.1185 (15)	0.000	0.0141 (10)	0.000
N4	0.0626 (9)	0.0993 (13)	0.0671 (9)	0.000	0.0139 (6)	0.000
N5	0.1123 (14)	0.0918 (14)	0.1242 (16)	0.000	0.0488 (12)	0.000

Geometric parameters (Å, º)

C1—N2	1.333 (2)	C4—H4	0.9300
C1—C2	1.356 (2)	C5—N2	1.341 (2)
C1—H1	0.9300	C5—H5	0.9300
C2—C3	1.3985 (18)	N1—H1A	0.8600
C2—H2	0.9300	N1—H1B	0.8600
C3—N1	1.3437 (17)	N2—H2A	0.8600
C3—C4	1.395 (2)	N3—N4	1.182 (3)
C4—C5	1.357 (2)	N4—N5	1.164 (2)
N2—C1—C2	123.06 (14)	C3—C4—H4	120.2
N2—C1—H1	118.5	N2—C5—C4	122.83 (15)
C2—C1—H1	118.5	N2—C5—H5	118.6
C1—C2—C3	119.58 (14)	C4—C5—H5	118.6
C1—C2—H2	120.2	C3—N1—H1A	120.0
C3—C2—H2	120.2	C3—N1—H1B	120.0
N1—C3—C4	121.68 (12)	H1A—N1—H1B	120.0
N1—C3—C2	121.36 (13)	C1—N2—C5	117.93 (13)
C4—C3—C2	116.97 (13)	C1—N2—H2A	121.0
C5—C4—C3	119.63 (14)	C5—N2—H2A	121.0
C5—C4—H4	120.2	N5—N4—N3	180.000 (1)
N2—C1—C2—C3	−0.5 (2)	C2—C3—C4—C5	0.10 (18)
C1—C2—C3—N1	−179.81 (11)	C3—C4—C5—N2	0.0 (2)
C1—C2—C3—C4	0.11 (17)	C2—C1—N2—C5	0.6 (2)
N1—C3—C4—C5	−179.98 (11)	C4—C5—N2—C1	−0.3 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···N5^{i}	0.86	2.15	3.008 (2)	174
N1—H1*B*···N3^{ii}	0.86	2.14	2.9942 (18)	172
N2—H2*A*···N2^{iii}	0.86	1.84	2.689 (3)	169

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