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Amantadinium azide

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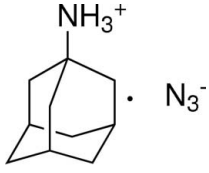
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.051; wR factor = 0.152; data-to-parameter ratio = 15.0.

The title compound, $C_{10}H_{18}N^+\cdot N_3^-$, was obtained by the reaction of adamantadine chloride and NaN_3. Within the azide anion, the N—N bond distances are 1.164 (2) and 1.153 (2) Å. Each protonated amantadine cation links with three azide anions *via* N—H···N hydrogen bonding, forming a supramolecular structure.

Related literature

For general background, see: Anga *et al.* (2002). For related structures, see: Bélanger-Gariépy *et al.* (1987); Singh *et al.* (2006); Yang *et al.* (2006).



Experimental

Crystal data

$C_{10}H_{18}N^+\cdot N_3^-$
M_r = 194.28
Monoclinic, $P2_1/n$
a = 8.524 (5) Å
b = 13.069 (7) Å
c = 10.013 (6) Å
β = 104.037 (7)°
V = 1082.1 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
0.56 × 0.46 × 0.44 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
5471 measured reflections
1907 independent reflections
1199 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.152
S = 1.08
1907 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.504 (2)	N3—N4	1.153 (2)
N2—N3	1.164 (2)		
N4—N3—N2	179.6 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N4^{i}	0.89	1.98	2.862 (3)	172
N1—H1B···N2	0.89	2.03	2.913 (3)	169
N1—H1C···N2^{ii}	0.89	2.01	2.863 (3)	160

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: XU2241).

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supplementary materials

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Comment

Energetic materials containing azide play an important role in the synthesis chemistry and explosive substances (Singh *et al.*, 2006). The use of azide contributes to high heats of formation, the release of large amounts of gases (e.g., N_2) as favored explosion products. Energetic salts often possess many advantages over conventional non-ionic energetic molecular compounds since the salts tend to exhibit lower vapor pressures than their non-ionic analogs. For related structure, urotropine, a model for energetic, nitrogen rich cage compounds, with the azide, has been reported (Anga *et al.*, 2002). There are two distinct hydrogen-bonded rings as a result of that the zide act as a bridge which are quite different from the other amantadinium (Bélanger-Gariépy *et al.*, 1987; Yang *et al.*, 2006).

In the structure of the title compound, (I), (Figure. 1), the azide ion is linear with an angle of 179.4 (3)° (N2—N3—N4). The bond lengths of N2—N3 and N3—N4 are 1.164 (3) Å and 1.153 (3) Å, respectively. Each azide ion is connected to three protonated amantadines *via* N—H···N hydrogen bonds (Table 2). The donor and acceptor sites form two distinct hydrogen-bonded $R_2^2(8)$ and $R_8^4(24)$rings. The $R_8^4(24)$ ring has the dimensions *ca.*10.5× 7.5 Å. Thus, N_3^- anions and $(C_{10}H_{18}N)^+$ cations generate the two-dimensional network *via* hydrogen bonding (Figure 2).

Experimental

A mixture of adamantadine chloride (185 mg, 1 mmol), NaN_3 (65 mg, 1 mmol), and H_2O (10 ml) was heated at 60°C. The resulting clear solution was cooled, and left for standing at room temperature over night. Crystals were collected (yield = 75.7 mg, 39% based on amantadine). Elemental Anal. Calcd (%) for $C_{10}H_{18}N_4C$, 61.82; H, 9.34; N, 28.84; found: C, 61.79; H, 9.42; N, 28.86.

Refinement

Ammonium H atoms were placed in calculated positions, with N—H = 0.89 A °, the torsion angle was refined to fit the electron density, and $U_{iso}(H) = 1.5U_{eq}(N)$. Other H atoms were placed in calculated positions with C—H = 0.97 or 0.98 A ° and refined in riding mode, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

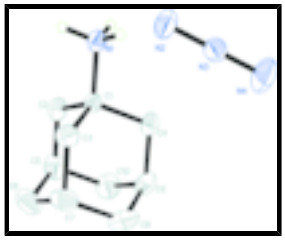

Fig. 1. The molecular structure of (I), shown with 30% probability displacement ellipsoids. H atoms on C atoms have been omitted for clarity.

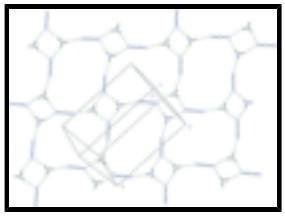

Fig. 2. The hydrogen-bonding network (dashed lines). All adamantanes are omitted for clarity.

Amantadinium azide

Crystal data

$C_{10}H_{18}N^{+}\cdot N_3^{-}$	F_{000} = 424
M_r = 194.28	D_x = 1.193 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 1511 reflections
a = 8.524 (5) Å	θ = 2.6–24.2º
b = 13.069 (7) Å	μ = 0.08 mm^{-1}
c = 10.013 (6) Å	T = 298 (2) K
β = 104.037 (7)º	Block, colorless
V = 1082.1 (10) Å^3	0.56 × 0.46 × 0.44 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	1199 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.039
Monochromator: graphite	θ_{max} = 25.0º
T = 298(2) K	θ_{min} = 2.6º
φ and ω scans	h = −8→10
Absorption correction: none	k = −15→13
5471 measured reflections	l = −10→11
1907 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0698P)^2 + 0.1298P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.051	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.152	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
S = 1.08	$\Delta\rho_{min}$ = −0.29 e Å^{-3}
1907 reflections	Extinction correction: none
127 parameters	
Primary atom site location: structure-invariant direct methods	

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

Special details

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}
N1	0.00540 (17)	0.11217 (11)	0.36198 (15)	0.0407 (5)
H1A	−0.0562	0.1668	0.3343	0.061*
H1B	0.0411	0.1134	0.4532	0.061*
H1C	−0.0525	0.0557	0.3369	0.061*
N2	0.1359 (2)	0.08765 (14)	0.65720 (18)	0.0567 (5)
N3	0.23433 (19)	0.14675 (13)	0.70858 (16)	0.0434 (4)
N4	0.3317 (3)	0.20559 (17)	0.7586 (2)	0.0858 (8)
C1	0.1470 (2)	0.11312 (13)	0.29695 (17)	0.0345 (5)
C2	0.0849 (2)	0.11057 (15)	0.14103 (19)	0.0473 (5)
H2A	0.0173	0.1698	0.1102	0.057*
H2B	0.0204	0.0495	0.1136	0.057*
C3	0.2509 (2)	0.01921 (15)	0.3462 (2)	0.0452 (5)
H3A	0.1876	−0.0424	0.3199	0.054*
H3B	0.2881	0.0204	0.4458	0.054*
C4	0.2454 (2)	0.21027 (14)	0.3403 (2)	0.0442 (5)
H4A	0.2829	0.2126	0.4398	0.053*
H4B	0.1785	0.2700	0.3104	0.053*
C5	0.2299 (3)	0.11080 (18)	0.0758 (2)	0.0601 (6)
H5	0.1914	0.1089	−0.0247	0.072*
C6	0.3348 (3)	0.0170 (2)	0.1248 (3)	0.0736 (8)
H6A	0.4260	0.0163	0.0827	0.088*
H6B	0.2722	−0.0447	0.0972	0.088*
C7	0.3960 (3)	0.01944 (17)	0.2814 (3)	0.0610 (7)
H7	0.4636	−0.0407	0.3125	0.073*
C8	0.4945 (3)	0.11661 (19)	0.3237 (3)	0.0687 (7)
H8A	0.5874	0.1167	0.2838	0.082*
H8B	0.5336	0.1186	0.4231	0.082*
C9	0.3904 (2)	0.21073 (17)	0.2748 (2)	0.0557 (6)
H9	0.4546	0.2728	0.3022	0.067*
C10	0.3299 (3)	0.20765 (18)	0.1186 (2)	0.0655 (7)

H10A	0.4211	0.2084	0.0766	0.079*
H10B	0.2640	0.2675	0.0871	0.079*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0393 (9)	0.0349 (9)	0.0448 (9)	0.0009 (6)	0.0042 (7)	0.0006 (7)
N2	0.0614 (11)	0.0511 (11)	0.0546 (11)	−0.0223 (9)	0.0085 (9)	−0.0074 (9)
N3	0.0480 (10)	0.0412 (10)	0.0440 (10)	−0.0065 (9)	0.0168 (8)	−0.0040 (8)
N4	0.0813 (15)	0.0775 (16)	0.0968 (17)	−0.0436 (13)	0.0179 (13)	−0.0271 (13)
C1	0.0340 (10)	0.0296 (10)	0.0381 (10)	−0.0005 (7)	0.0054 (8)	0.0016 (7)
C2	0.0551 (13)	0.0419 (12)	0.0397 (12)	−0.0055 (9)	0.0015 (10)	0.0000 (9)
C3	0.0451 (11)	0.0358 (12)	0.0542 (13)	0.0048 (8)	0.0113 (9)	0.0079 (9)
C4	0.0486 (12)	0.0335 (11)	0.0452 (11)	−0.0045 (8)	0.0013 (9)	0.0002 (8)
C5	0.0807 (17)	0.0619 (16)	0.0400 (12)	−0.0101 (12)	0.0189 (12)	0.0004 (10)
C6	0.0909 (18)	0.0626 (17)	0.0836 (19)	0.0021 (14)	0.0526 (15)	−0.0082 (13)
C7	0.0546 (14)	0.0499 (14)	0.0842 (18)	0.0174 (11)	0.0279 (12)	0.0143 (11)
C8	0.0370 (12)	0.0841 (19)	0.0850 (18)	0.0008 (11)	0.0150 (12)	0.0160 (14)
C9	0.0525 (13)	0.0494 (14)	0.0634 (15)	−0.0185 (10)	0.0108 (11)	0.0034 (10)
C10	0.0807 (16)	0.0589 (16)	0.0623 (15)	−0.0095 (12)	0.0283 (13)	0.0139 (11)

Geometric parameters (Å, °)

N1—C1	1.504 (2)	C4—H4B	0.9700
N1—H1A	0.8900	C5—C6	1.527 (3)
N1—H1B	0.8900	C5—C10	1.528 (3)
N1—H1C	0.8900	C5—H5	0.9800
N2—N3	1.164 (2)	C6—C7	1.528 (3)
N3—N4	1.153 (2)	C6—H6A	0.9700
C1—C2	1.522 (3)	C6—H6B	0.9700
C1—C3	1.524 (2)	C7—C8	1.525 (3)
C1—C4	1.526 (2)	C7—H7	0.9800
C2—C5	1.531 (3)	C8—C9	1.526 (3)
C2—H2A	0.9700	C8—H8A	0.9700
C2—H2B	0.9700	C8—H8B	0.9700
C3—C7	1.530 (3)	C9—C10	1.524 (3)
C3—H3A	0.9700	C9—H9	0.9800
C3—H3B	0.9700	C10—H10A	0.9700
C4—C9	1.533 (3)	C10—H10B	0.9700
C4—H4A	0.9700		
C1—N1—H1A	109.5	C6—C5—H5	109.5
C1—N1—H1B	109.5	C10—C5—H5	109.5
H1A—N1—H1B	109.5	C2—C5—H5	109.5
C1—N1—H1C	109.5	C5—C6—C7	109.89 (18)
H1A—N1—H1C	109.5	C5—C6—H6A	109.7
H1B—N1—H1C	109.5	C7—C6—H6A	109.7
N4—N3—N2	179.6 (2)	C5—C6—H6B	109.7
N1—C1—C2	109.15 (14)	C7—C6—H6B	109.7

N1—C1—C3	108.52 (14)	H6A—C6—H6B	108.2
C2—C1—C3	110.15 (15)	C8—C7—C6	109.30 (19)
N1—C1—C4	109.12 (14)	C8—C7—C3	109.33 (19)
C2—C1—C4	109.90 (15)	C6—C7—C3	109.04 (18)
C3—C1—C4	109.98 (14)	C8—C7—H7	109.7
C1—C2—C5	108.75 (16)	C6—C7—H7	109.7
C1—C2—H2A	109.9	C3—C7—H7	109.7
C5—C2—H2A	109.9	C7—C8—C9	110.10 (17)
C1—C2—H2B	109.9	C7—C8—H8A	109.6
C5—C2—H2B	109.9	C9—C8—H8A	109.6
H2A—C2—H2B	108.3	C7—C8—H8B	109.6
C1—C3—C7	109.09 (15)	C9—C8—H8B	109.6
C1—C3—H3A	109.9	H8A—C8—H8B	108.2
C7—C3—H3A	109.9	C10—C9—C8	109.5 (2)
C1—C3—H3B	109.9	C10—C9—C4	109.35 (17)
C7—C3—H3B	109.9	C8—C9—C4	109.02 (17)
H3A—C3—H3B	108.3	C10—C9—H9	109.7
C1—C4—C9	109.05 (16)	C8—C9—H9	109.7
C1—C4—H4A	109.9	C4—C9—H9	109.7
C9—C4—H4A	109.9	C9—C10—C5	109.67 (17)
C1—C4—H4B	109.9	C9—C10—H10A	109.7
C9—C4—H4B	109.9	C5—C10—H10A	109.7
H4A—C4—H4B	108.3	C9—C10—H10B	109.7
C6—C5—C10	109.4 (2)	C5—C10—H10B	109.7
C6—C5—C2	109.40 (18)	H10A—C10—H10B	108.2
C10—C5—C2	109.57 (18)		

N1—C1—C2—C5	−179.61 (15)	C5—C6—C7—C3	60.0 (2)
C3—C1—C2—C5	−60.6 (2)	C1—C3—C7—C8	59.6 (2)
C4—C1—C2—C5	60.8 (2)	C1—C3—C7—C6	−59.8 (2)
N1—C1—C3—C7	−179.74 (15)	C6—C7—C8—C9	59.3 (2)
C2—C1—C3—C7	60.8 (2)	C3—C7—C8—C9	−60.0 (2)
C4—C1—C3—C7	−60.4 (2)	C7—C8—C9—C10	−59.5 (2)
N1—C1—C4—C9	179.57 (14)	C7—C8—C9—C4	60.1 (2)
C2—C1—C4—C9	−60.78 (19)	C1—C4—C9—C10	59.8 (2)
C3—C1—C4—C9	60.6 (2)	C1—C4—C9—C8	−59.8 (2)
C1—C2—C5—C6	59.8 (2)	C8—C9—C10—C5	59.6 (2)
C1—C2—C5—C10	−60.1 (2)	C4—C9—C10—C5	−59.8 (2)
C10—C5—C6—C7	59.8 (2)	C6—C5—C10—C9	−59.8 (2)
C2—C5—C6—C7	−60.2 (2)	C2—C5—C10—C9	60.1 (2)
C5—C6—C7—C8	−59.4 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···N4^{i}	0.89	1.98	2.862 (3)	172
N1—H1B···N2	0.89	2.03	2.913 (3)	169
N1—H1C···N2^{ii}	0.89	2.01	2.863 (3)	160

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

Fig. 1

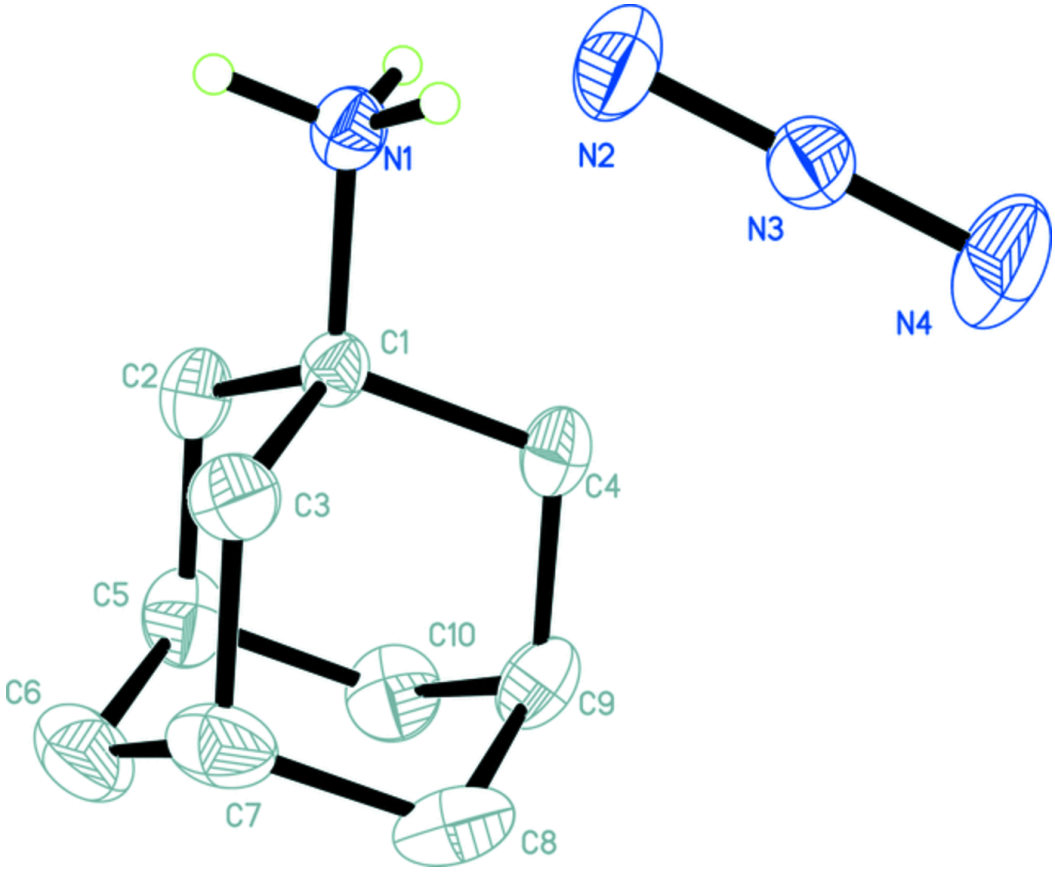

Fig. 2